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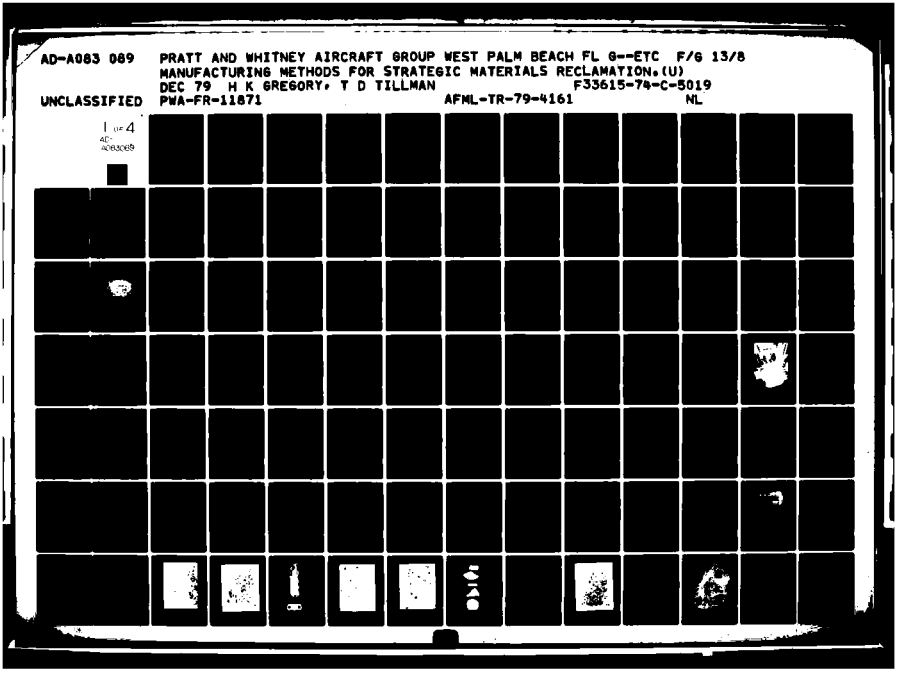

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LEVEL II

(2)

MANUFACTURING METHODS FOR STRATEGIC MATERIALS RECLAMATION

United Technologies Corporation
Pratt & Whitney Aircraft Group
Government Products Division
West Palm Beach, Florida

14 December 1979

Technical Report AFML-TR-79-4161
Final Report for Period 15 March 1974 - 31 May 1979

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This final report was submitted by Pratt & Whitney Aircraft Group, Division of United Technologies Corporation, Government Products Division, West Palm Beach, Florida under Contract F33615-74-C-5019, Manufacturing Technology Program 162-4, "Manufacturing Methods for Strategic Materials Reclamation". Mr. Kenneth L. Love, AFML/LTM was the project manager.

This technical report has been reviewed and is approved for publication.

K.L. Love

Kenneth L. Love
Project Manager

For The Commander

H.A. Johnson

H.A. Johnson
Chief, Metals Branch
Manufacturing Technology Division

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This document details the work done on a three-phase program to establish manufacturing methods for reclamation of strategic materials. The work was divided into two categories: (1) scrap management, and (2) scrap reclamation technologies. Two material, Ti-6Al-4V and Waspaloy, were investigated for the possibility of reclamation to aerospace quality.

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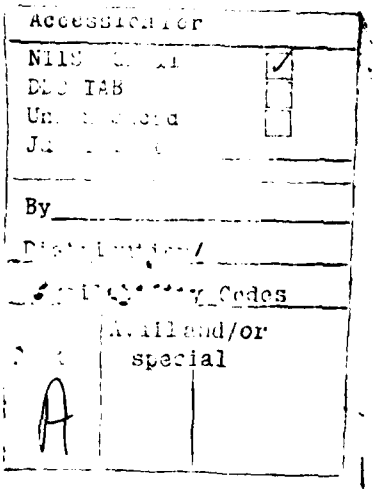
Due to the location and duration of this contract, the following individuals have also served and contributed to the report as Program Managers:

J. R. Doyle
J. E. Flynn
P. R. Holiday.



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SECTION I

INTRODUCTION

Raw material cost represents a significant portion of overall gas turbine engine component cost, particularly in those components requiring relatively scarce and expensive metals such as nickel-base superalloys and titanium. The high quality requirements of the aerospace industry have frequently resulted in inefficient use of raw materials. More than 7 of every 10 lb of aerospace raw material becomes scrap during component fabrication operations. Failure to effectively recycle this scrap back to the aerospace industry represents a large cost burden. Aerospace materials are bought for dollars-a-pound as raw material and sold for cents-a-pound as scrap, predominantly to nonaerospace users. In addition to this cost burden, the dependence on imported materials, in an emerging era of shortages, is of growing concern. These problems can be relieved to some extent by effective scrap recycling.

The primary material producers, such as melters, have established effective in-house scrap handling systems based on internal control of revert material quality; however, much of the scrap generated by secondary processors is sold to dealers who apply processing methods to upgrade the scrap for available, frequently nonaerospace, markets. Titanium turnings with potential tungsten carbide tool bit contamination, and mixed alloy lots of nickel-base alloys or titanium alloys, are typical examples of scrap forms having a well established nonaerospace market, but only limited available reclamation alternatives for upgrading to aerospace quality standards. A strong incentive exists for establishing reclamation methods for these materials.

This three-phase program that was conducted by P&WA under AFML contract sought to establish scrap reclamation under two general approaches: (1) scrap management, and (2) scrap reclamation technology. Scrap management concepts include the handling of scrap in a manner to optimize its reclamation potential, and identification of methods to increase scrap utilization without compromising required material quality. Scrap reclamation technologies include establishment of separation methods, melting methods, etc., to upgrade scrap quality.

The program plan is summarized in Section II.

SECTION II

PROGRAM PLAN SUMMARY

A three-phase program was conducted to establish production capability for upgrading titanium alloy and nickel-base superalloy scrap. The program consisted of parallel scrap management and reclamation technology efforts. A program flow chart is presented in Figure 1 to identify program tasks and subcontract efforts.

The program, as shown in Figure 1, represents a broad approach to the scrap reclamation problem, involving both titanium and nickel-base alloys in various forms. Basically, the plan comprises four major lines of effort: (1) scrap management; (2) evaluation of systems for separation of titanium and/or nickel-base superalloy chips; (3) evaluation of nonconsumable melting processes for titanium chips; and (4) evaluation of a chemical process to improve the purity of nickel-base alloy grindings, and sludges. Detailed experimental investigation was required for evaluation of scrap reclamation methodology. The program plan presents an overview of the investigation; details of experiments and evaluations as the experiments were conducted. The following paragraphs detail the work content of the program by phase.

A. PHASE I

The objective of Phase I was to investigate and select the most promising method for the management and reclamation of scrap titanium alloys and nickel-base superalloys.

1. Management System

The cornerstone of a successful scrap reclamation approach must be an overall scrap management system which carefully considers available technologies, process economics and quality control requirements. The management system should provide control of scrap from its point of generation to its reintroduction back into the raw material supply system. Accurate information on the character and availability of scrap is a prerequisite to the establishment of such a system. In consideration of this, a Phase I scrap management effort consisting of the following elements was planned.

1. Seminar — A scrap reclamation seminar was held at the inception of the program to provide a forum for information exchange and discussion of scrap reclamation concepts by a broad spectrum of the aerospace industry. A brief description of the seminar is contained in Section IV of this report. A seminar summary report was distributed.

2. Survey — A survey was conducted to determine the character, quality, disposition and domestic availability of titanium and nickel-base superalloy scrap generated by the aerospace industry. Suisman and Blumenthal played a major role in conducting this survey. The membership of Suisman and Blumenthal in national scrap dealers associations, and their in-depth knowledge of a broad spectrum of the industry enabled efficient accumulation of information. P&WA assisted in determining specific types of survey information, as required. It is believed that this was the first such broad survey of scrap. The survey also included a forecast of any significant changes in the character or quantities of scrap which may affect the establishment of reclamation technologies. For example, the change in scrap character to be anticipated with the expanding usage of Borazon tool bits was assessed.

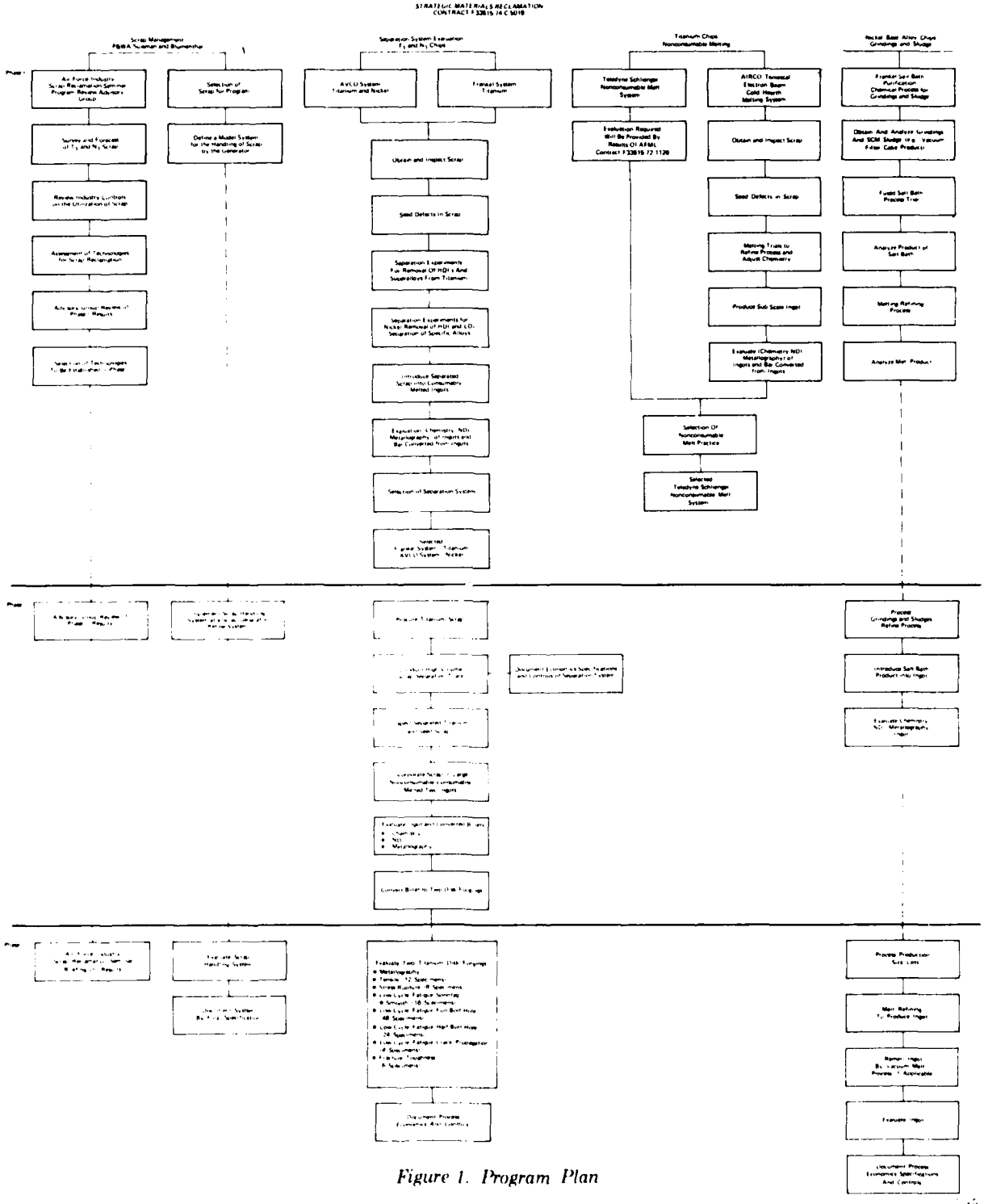

Figure 1. Program Plan

3. Review of Industry Controls — It is recognized that a significant restraint to the increased utilization of scrap is the controls imposed either by raw material suppliers on themselves, or by the users on raw material suppliers. The sensitive and frequently proprietary nature of these controls precludes a specific detailed accounting of industry scrap controls. P&WA, however, sought general information on industry controls, and reviewed general policies which have a major influence on the utilization of scrap. Specific controls which preclude or limit the use of scrap were defined and reviewed, where possible.

4. Review of Available Technologies — P&WA, in reviewing and selecting technologies for investigation in this program, identified several technologies which, due to insufficient current development, uncertain economic potential, or prohibitive funding requirements to establish the process, were considered inappropriate for inclusion in the program. For example, an Electrostag Melting System (ESR) has potential for reclamation of various forms of contaminated nickel-base alloy scrap; however, it was not sufficiently developed to be included in this program. An up-to-date assessment of rapidly emerging scrap reclamation processes, in addition to an assessment of the technologies to be investigated in Phase I, was conducted prior to selection of technologies to be investigated in Phases II and III.

5. Definition and Implementation of a Model Scrap Handling System — An initial survey of the scrap reclamation problem indicates that a significant amount of aerospace scrap is downgraded beyond potential reclamation for aerospace uses by inadequate scrap handling procedures. Some aerospace vendors receive little or no compensation for the value of scrap removed from their plants by secondary metal processors. These problems highlight the need for an educational program in the aerospace industry to maintain the maximum potential for reclamation of scrap. P&WA subcontracted to Suisman and Blumenthal the tasks of defining a model system for the handling of scrap by the generator. Suisman and Blumenthal, who provide both plant pick-up service and conduct subsequent processing of scrap, were qualified to carry out this task. The model system was documented by a specification or set of guidelines and an educational brochure (Appendix A).

2. Technologies Investigated

Parallel to the establishment of a management system in Phase I, scrap reclamation technologies were investigated. Technologies investigated were in some cases alternatives, and in these cases a single technology was selected for continuation and scale-up in Phase II, as reflected in the program flow chart (Figure 1). The Phase I scrap reclamation technologies that were investigated are as follows:

1. Two density separation methods, i.e., the AVCO ferrofluid method and the Frankel fluidized bed method, were investigated. The AVCO method was applied to both titanium and nickel while the Frankel method was applied to titanium only. The AVCO ferrofluid process accomplishes separation by introducing scrap into a fluid in which a variable apparent fluid density may be induced magnetically. Separation is achieved by the controlled sinking or floating of known density scrap constituents. The Frankel system utilizes an air fluidized bed to achieve an apparent density medium for separation of scrap by rising or sinking. The ability of these processes to cost-effectively

separate contaminants from scrap was assessed by a series of experiments utilizing scrap with known contamination. The Frankel fluidized bed separation method was later selected for Phase II scale-up.

2. Two nonconsumable melt processes were investigated: the Teledyne-Schlienger rotating-electrode system, and the AIRCO-Temescal electron-beam cold-hearth melting system. These systems each have the potential to remove high-density contaminants by entrapment with a melt-skull, and each provides a capability for introduction of large quantities of scrap into the melt. The electron-beam cold-hearth system is currently being applied successfully to the reclamation of Ti-5Al-2½Sn scrap to a commercial product, not requiring chemistry control. The capability of this process to produce controlled chemistry, aerospace quality ingot from Ti-6Al-4V scrap input material was assessed in Phase I. Evaluation of the Teledyne nonconsumable melt process did not require any contract effort during Phase I and was based on the results of AFML Contract F33615-72-1126. The Teledyne nonconsumable melt system was selected for Phase II scale-up.

3. A molten salt bath process developed by Frankel Company was investigated for purification of nickel-base superalloy grindings and sludges. This process was evaluated for its potential to reclaim segregated grindings for subsequent vacuum melting. The highest potential for recovery of sludges by this process would be a pure multi-element master alloy. The molten salt purification process has been demonstrated to be efficient on a small scale but has not been evaluated under full-scale industrial conditions. Grindings and sludges which pass through the molten salt purification process come out as a dry substance containing only metallic and inorganic particles, particularly grinding compounds like carborundum or aluminum oxide particles. These inorganic particles can be separated from the metallic particles by gravity separations similar to the ones utilized in the ore industry. The thus purified metal particles are sufficiently pure to be remelted in furnaces. In cases of certain metallic sludges where the metallic content (mainly nickel content) is in oxide form, these oxides can be reduced to metallic form in regular smelting furnaces.

4. Evaluation of reclaimed material during Phase I included extensive chemical analysis of processed scrap and sub-scale ingots, NDI of converted barstock from ingots, and selective mechanical properties determination.

B. PHASE II

The objectives of Phase II were to establish the scrap reclamation processes for titanium and nickel-base superalloys that were shown in Phase I to be most promising; to evaluate the process variables and accomplish the necessary refinements; and to conduct preliminary testing of the product after the reclamation cycle was established.

Evaluation of the scrap management systems, established in Phase I and reclamation technologies selected during Phase I, were continued in Phase II. The model scrap handling system defined in Phase I was implemented and refined at the plant of a selected generator of titanium and nickel-base alloy scrap during Phase II. The practicality of the system was determined and suitable refinements incorporated.

The scrap reclamation technology effort in Phase II was as follows:

1. The Frankel density separation method and Teledyne's nonconsumable melting method, selected during Phase I, were established and evaluated on a production scale basis and were applied sequentially for Ti-6Al-4V chips to establish a complete titanium reclamation system.

2. Two 5000 lb lots of titanium chips were processed by the density separation method.

3. Two 5000 lb 26-in. dia titanium ingots were produced utilizing scrap processed by the density separation method.

4. Evaluation of reclaimed material, during Phase II, included chemical analyses, NDI of converted barstock and billet, and selective mechanical properties determination.

5. The TF33 second-stage fan disks (Ti-6Al-4V) were forged from converted billets. These forgings were evaluated in Phase III.

6. The molten salt purification process was subject to further investigation. Two 800 lb lots of grindings and/or sludge were processed, melted to ingots, and evaluated, primarily by chemical analysis.

C. PHASE III

The objective of Phase III was to verify and establish the reliability, reproducibility, and economy of the reclamation process. Evaluation of the scrap management system, refined during Phase II, and the reclamation technologies established during Phase II, was also continued. In addition, the scrap handling system established during Phase II was evaluated by comparing the amounts of scrap recyclable from the selected generator back into the aerospace material supply system before and after implementation of the system.

The scrap reclamation technology effort in Phase III was as follows:

1. A 6000 lb lot of Ni grinding sludge was refined and a cost analysis of processing was performed.

2. Mechanical properties of the Ti-6Al-4V forgings produced during Phase II were determined and compared to P&WA specifications.

SECTION III

MATERIAL SELECTION

The primary materials utilized for investigation of reclamation methods were a titanium alloy, Ti-6Al-4V and a nickel-base superalloy, Waspaloy. A limited effort was conducted on other titanium alloys (Ti-8Al-1Mo-1V and Ti-6Al-2Sn-4Zr-2Mo) and superalloys (Inconel 718, Incoloy 901, and A-286) to determine compatibility with the separation reclamation processes. Ti-6Al-4V scrap was selected because of the preponderence of this alloy in the scrap market. Waspaloy was selected as a representative of the widely used precipitation hardenable nickel-base superalloys, and because of the high degree of quality required of the alloy in its primary utilization in critical rotating hardware.

Scrap utilized in this program was obtained from a common source in order to eliminate the scrap as a variable while evaluating alternative reclamation methods in Phase I. Frankel Company, a leader in the scrap processing industry, provided scrap to all subcontractors. Scrap to be utilized for the majority of Phase I experiments was obtained by Frankel from Suisman & Blumenthal under their plant service contract with P&WA. Frankel provided a limited amount of scrap from their own inventory to meet special small lot program requirements. Particular attention was given to the scrap selection to ensure that this material was representative of the bulk aerospace scrap supply in terms of form, chemistry and life cycle. The Ti-6Al-4V scrap form selected for the majority of the program experiments was a mix of flat-to-curled crushed turnings in the medium bulk density range (i.e., 15-30 lb/cu ft). A sample of the Ti-6Al-4V scrap selected, as obtained by Frankel from Suisman and Blumenthal for processing, is shown in Figure 2. The selected Waspaloy scrap was obtained by Frankel in a noncrushed form.

Analyses of Ti-6Al-4V and Waspaloy scrap, before Frankel processing are listed in Table 1. Applicable specification chemical analyses are included for comparison. The Waspaloy scrap deviates from specification because of bismuth content, a typical superalloy scrap contamination problem. The Ti-6Al-4V scrap deviates from specification because the total percentage of nonspecified elements exceeds 0.4%, again a typical problem. The Aqua Regia soluble measurements provide a specific indication of contaminants in the Ti-6Al-4V scrap, as most contaminants are soluble while the titanium alloy is unsoluble in this acid medium. The ratio of Aqua Regia soluble Ni to Cr indicates more superalloy contamination than stainless steel contamination for this lot. Bulk densities are also listed; however, it was not possible to measure the bulk density of Waspaloy before processing because the scrap was not in a crushed condition.

The scrap material utilized in Phase I was processed by Frankel Company and was distributed to applicable subcontractors. The processing steps are listed in Table 2.

The Ni grinding sludge refined and evaluated in Phase III was obtained from the Vac-Air Alloys Corp., and represents nonsegregated Ni-base superalloy grinding sludge.

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Figure 2. Titanium (Ti-6Al-4V) Chips Supplied by Suisman and Blumenthal, Inc. Prior to Processing by Frankel Co. Processed Chips Were Utilized in Phase I Reclamation Technology Development

TABLE 1
SPECIFICATION VS PREPROCESS ANALYSES (TI-6AL-4V AND WASPALOY)

	Ti-6Al-4V		*Waspaloy*	
	AMS 4928		*PWA 1007*	
	Spec	*Preprocess*	*Spec*	*Preprocess*
Density (lb/ft³)		25 lb/ft³		—
Oil and Moisture		19.6		2.0
Magnetics		0.4		0.1
Free Al		<0.05		
Ni			R***	R
Co			12.0 to 15.0	13.5
Fe	0.3 Max		2.0 Max	1.30
Ta				<0.1
Mo		-	3.5 to 5.0	4.00
Cr			18.0 to 21.0	19.3
Cb		<0.05		<0.1
Zr		0.05	0.02 to 0.12	0.05
Ti	R***	R	2.75 to 3.25	3.09
V	3.50 to 4.50	3.85		<0.1
Mn		—	0.75 Max	0.07
Cu			0.10 Max	0.03
W				<0.1
Al	5.5 to 6.75	—	1.2 to 1.6	—
Si			0.75 Max	—
C	0.10 Max	—	0.02 to 0.10	—
S			0.02 Max	—
Pb		<0.05	10.0 ppm Max	—
Bi			0.5 ppm Max	—
B			0.003 to 0.010	—
Sn		0.5		—
O_2	0.20 Max	—		
H_2	125 ppm Max	—		
N_2	500 ppm Max	—		
Mg		—		
Fe(ARS)**		0.1		
Ni(ARS)		0.3		
Ci(ARS)		0.1		
Other	0.4 Max	0.6		

* Titanium scrap was received in crushed condition with a bulk density of 16.1: recrushing of this scrap by Frankel resulted in a bulk density of 25.

**ARS - Aqua Regia Soluble.

***R Remainder

— To be determined at post-process analyses

TABLE 2
SCRAP PROCESSING STEPS

Ti-6Al-4V	*Waspaloy*
Double-crush in ring-type mechanical crusher	Same
Continuous sample (100 lb per lot) diverted by crusher	Same
Bulk density determination	Same
Procedure several ¼ lb arc melted buttons	Produce 17 lb VIM ingot
Preprocessing chemistry determination	Same
Hot detergent alkaline wash	Vapor degrease
Screen elimination of 10-mesh or lower material	Same
	Permanent magnet
Medium intensity electromagnet	Same
	High intensity (12,077 gauss) electromagnet
Melt ingot from drill auger chip sample	Same
Post-processing chemistry determination	Same

SECTION IV

STRATEGIC MATERIALS RECLAMATION SEMINAR

The Strategic Materials Reclamation seminar was held at the Holiday Inn, Hartford, Connecticut, 14-15 May 1974. The seminar was hosted by P&WA in partial fulfillment of the Strategic Materials Reclamation contract. The objective of this seminar was to provide an overview of the current titanium and superalloy scrap situation and to identify new concepts for increased utilization of scrap materials for aerospace components.

This seminar represented one of a continuing series of seminars seeking to further define the general conclusions of the Air Force/Industry Manufacturing Cost Study Seminar held at Sagamore, New York. The inefficient utilization of aerospace raw material and the failure to effectively recycle scrap back to the aerospace industry were cited by the Sagamore Conference as a significant manufacturing cost factor. This seminar provided the opportunity to explore in greater depth potential cost savings through the reclamation of aerospace scrap and to define specific areas for future AFML funding consideration.

Seminar participants included representatives of the Air Force, Navy, Bureau of Mines and Aerospace industry, including scrap dealer/processors, melters, forgers, casting producers and gas turbine engine manufacturers. Forty-two individuals representing 25 organizations attended. The two-day agenda included one session of keynote presentations, two sessions of panel workshops, and a summary/recommendations session. Participants were divided into a titanium panel and a superalloy panel by prior arrangement for the workshop sessions. Panel chairmen guided the workshop discussions utilizing a discussion outline, collectively established by participants prior to the seminar, and reference material submitted by participants on topics assigned prior to the seminar.

The seminar achieved its objective and provided useful input for current and future work to be conducted in this technology area under Air Force sponsorship. As a follow-up to this meeting, participants were invited to discuss their individual comments or recommendations with AFML.

A seminar summary report was prepared and distributed.

SECTION V

MATERIAL PROCUREMENT

Approximately 10,000 lb of medium bulk density Ti-6Al-4V, and 1,300 lb of Waspaloy chips, obtained from Suisman and Blumenthal, were processed by the Frankel Co. for Phase I reclamation experiments. The Ti-6Al-4V chip lot was divided as follows: AVCO received 2,700 lb of fully-processed chips, 1,100 lb of processed but unscreened chips and 600 lb of unprocessed chips; AIRCO received 4,000 lb of fully processed chips, and Frankel retained 1,500 lb of processed chips. The Waspaloy chips were all allotted to AVCO. Frankel additionally processed chips from their own inventory in order to fulfill their own requirements, and those of AVCO, for small specialty lots, e.g., high- and low-bulk density Ti-6Al-4V, contaminant-materials and various superalloys. AVCO scrap requirements are listed in Table 3. Required chips were shipped to the subcontractors in mid-July 1974.

TABLE 3
AVCO SCRAP REQUIREMENTS FOR PHASE I OF STRATEGIC MATERIALS RECLAMATION PROGRAM

Lot Number	*Alloy*	*Source*	*Bulk Density Range (lb/cu ft)*	*Size Range (in.)*	*Oil/Water Content*	*Quantity Req (lb)*
01	Ti-6Al-4V	Frankel*	20 to 30	3 to $\frac{1}{8}$	Neg	2,700
02	Ti-6Al-4V	Frankel	20 to 30	3	***	500
03	Ti-6Al-4V	Frankel	20 to 30	3	Neg	1,100
04	Ti-6Al-4V	Frankel	10 to 20	3 to $\frac{1}{8}$	Neg	1,200
05	Ti-6Al-4V	Frankel	30 to 40	3 to $\frac{1}{8}$	Neg	1,300
06	Ti-8Al-1Mo-1V	S&B**	25 to 30	3 to $\frac{1}{8}$	Neg	200
07	Ti-6Al-2Sn-4Zr-2Mo	Frankel	25 to 30	3 to $\frac{1}{8}$	Neg	200
08	Ti-5Al-2.5Sn	S&B	25 to 30	3 to $\frac{1}{8}$	Neg	100
09	Waspaloy	Frankel	Medium	3 to $\frac{1}{8}$	Neg	1,300
10	IN 901	Frankel	Medium	$\frac{1}{4}$ to $\frac{1}{8}$	Neg	10
11	IN 718	Frankel	Medium	$\frac{1}{4}$ to $\frac{1}{8}$	Neg	10
12	L 605	Frankel	Medium	$\frac{1}{4}$ to $\frac{1}{8}$	Neg	10
13	A 286	Frankel	Medium	$\frac{1}{4}$ to $\frac{1}{8}$	Neg	10
14	304 Stainless Steel	S&B	Medium	3 to $\frac{1}{8}$	Neg	200

*Chips purchased from Suisman and Blumenthal Inc., by Frankel Co.
**Suisman and Blumenthal, Inc.
***Not controlled. As-received.

SECTION VI

MATERIAL PROCESSING AND ANALYSIS

A. TITANIUM

1. Processing

Uncrushed titanium turnings were unloaded from the receiving carrier (truck or railroad car) and transported directly to a ring-type crusher, which was similar to a hammermill except the crushing was done with rings rather than hammers. During operation, a stream of water was run through the crusher to prevent ignition of the titanium turnings. A conveyor transported the crushed turnings, referred to as chips, from under the crusher into drums. The drums contained a sampling tube which captured a chip sample, referred to as the crusher sample. This chips were held in the drums until the crusher sample (approximately 100 lb) was evaluated for chemistry.

After the crusher sample evaluation, the chips were subjected to further processing. The chips were cleaned with a hot alkaline detergent in a rotary tumble washer. After 15 to 30 min, depending upon the condition and type of chips, the washer dumped the chips into a hopper, from which a conveyor transported the chips into a centrifuge. On the conveyor, the chips were spray-rinsed to remove all traces of detergent. The centrifuge removed most of the water and moisture, and the chips were then conveyed into a rotary hot-air dryer. Dryer air temperature was controlled to a maximum of 275°F to preclude chip ignition. The dryer system included high-temperature-limit switches and audible and visible warning devices. The chips were removed from the dryer through a pneumatically-operated air lock. The exhausted air was passed through a cyclone separator to isolate dust entrapped in the air stream. The dust, a serious fire hazard, was removed from the cyclone through a rotary valve. After the cyclone, a scrubber removed the last traces of particulate matter from the air stream, thereby meeting stringent anti-pollution requirements. The titanium chips were conveyed from the dryer to a rotary-magnet separator, which separated the tramp iron, chrome steel, tool steel and larger tungsten carbide particles. The chips were then passed onto a double-deck oscillating screen, which simultaneously removed the oversize particles and the fines. The output was then conveyed into shipping containers (drums or boxes), depending upon customer specifications. A final sample was obtained by drilling each shipping container with an auger. If this sample met specification, the chip lot was approved for shipment.

2. Analysis

The titanium crusher sample and the final sample were each quartered down to one 500g and three 100g portions. In the case of the crusher sample, an additional 200g sample was obtained for an oil and moisture and magnetic determination. The three 100g portions were melted into three buttons in an inert atmosphere arc melting button furnace (crusher samples were degreased and the magnetics removed prior to melting). The melting was performed in the furnace under an argon atmosphere with a nonconsumable carbon electrode.

The face of each button was machined flat and the Mo, Cb, Zr, V, Mn and Fe content were determined on the X-ray spectrometer. The X-ray spectrometer was calibrated with the best available standards (NBS Standards were used where available, such as 6Al-4V, etc.). Aluminum was determined by Atomic Absorption.

The 500g portion of the chips was leached overnight with Aqua Regia to selectively dissolve most of the common nontitanium alloy contaminants, e.g., stainless steel, nickel-base alloys, aluminum alloys. After filtering off the titanium chips with glass wool, the solution was analyzed for Ni, Cr, Fe and Al. The aluminum determined in the Aqua Regia solution was reported as free Al. The nickel, chrome and iron determined in the Aqua Regia solution was reported as A. R. soluble Ni, Cr, Fe. The iron determined on the X-ray spectrometer was reported as total Fe, due to the fact that it represented both the iron contained in the titanium as an alloy ingredient and the iron contained in tramp contamination like stainless steel.

On the final sample, an additional 50g portion was washed with ether to extract all residual oil from the chips. An appropriate aliquot was transferred into a carbon determination crucible, and the ether evaporated at low temperature, leaving the residual oil in the crucible. After adding carbon free iron and tungsten accelerator, the carbon was determined. This percentage was the carbon from the oil left on the chips after cleaning, and was indicative of the cleanliness of the chips.

Table 4 presents the post-processing titanium lot chemical analyses, and for comparison, the previously-reported preprocessing crusher sample analyses, and specification limits. As shown in the table, the chemistry of post-process scrap does not differ significantly from that of preprocessed scrap. It should be noted that since magnetics were removed from the preprocess sample prior to chemical analysis, a significant reduction of contaminant elements would not be expected in the post-processing analysis. The medium bulk density Ti-6Al-4V overall scrap chemistry, however, remains deviant from specification because the cumulative percentage of contaminant elements exceeded the allowable limit.

Proper sampling methods are particularly important to the determination of scrap chemistries because of the potential for lack of homogeneity in scrap. Initial "grab samples" of the medium bulk density scrap, as analyzed by Suisman & Blumenthal and by Frankel, differed somewhat from the reported crusher sample analysis. The grab sample data is not reported because established sample technique was not applied. The chemistry of processed Frankel scrap, as checked by one subcontractor, also differed from the reported analysis, particularly in contaminant element level.

B. NICKEL ALLOY

1. Processing

Uncrushed nickel alloy turnings were unloaded from the receiving carrier (truck or railroad car) and transported directly to the crusher. The turnings were crushed in a ring-type crusher, as described previously. The crushed turnings, i.e., chips, were transported from the crusher by conveyor through a chute into drums. The main chute had a small side-chute which cut out a continuous sample from the chips. This sample (approx. 100 lb) was used for the evaluation of the incoming shipment (crusher sample). The chip lot was held in the drums until the crusher sample was evaluated. This sampling method differed somewhat from the previously described titanium sampling, i.e., the use of a side chute was impractical because of the heavy moisture content of wet-crushed Ti chips.

After the evaluation of the nickel chips sample, the chips were cleaned in a vapor degreaser. The chips were fed into a pan suspended from the top of the unit, and then exposed to trichlorethylene vapors while traveling up and out of the unit on a vibrating spiral conveyor. At the upper portion of the conveyor, above the vapor condensers, the chips were dried by steam heating the tracks of the spiral conveyor. Vapor degreasing was used for cleaning the superalloy chips because the technique has demonstrated acceptability; the newer alkaline wash method has only been adequately demonstrated for titanium.

TABLE 4
FRANKEL ANALYSIS OF TITANIUM LOT SAMPLES

	6Al-4V Chips (Medium) Crusher Sample	*6Al-4V Chips (Medium) Proc Sample*	*6Al-4V Chips (Heavy) Proc Sample*	*6Al-4V Chips (Light) Proc Sample*	*AMS 4928 Spec*
Oil and Moisture	19.6*	**	**	**	
Carbon Content of Free Oil		0.06%	0.01%	0.014%	
Magnetics	0.4%	**	**	**	
Bulk Density lb/ft³	25	23.2	36.3	15.6	
Al		6.55%	6.70%	6.50%	5.50 to 6.75%
Total Fe		0.38%	0.38%	0.57%	0.3% Max
AR					
Soluble					
Ni	0.3%	0.30%	0.25%	0.10%	
Cr	0.1%	0.09%	0.08%	< 0.05%	
Fe	0.1%	0.11%	< 0.05%	0.07%	
Free Al	< 0.05%	< 0.05%	< 0.05%	0.21%	
Pb	< 0.05%	< 0.05%	< 0.05%	0.18%	
Mo		0.15%	0.22%	< 0.05%	
Cb	< 0.05%	< 0.05%	< 0.05%	< 0.05%	
Zr	0.05%	0.07%	< 0.05%	0.10%	
V	3.85%	3.70%	3.49%	3.80%	3.50 to 4.50%
Mn	< 0.05%	< 0.05%	< 0.05%	< 0.05%	
Sn	0.05%	0.09%	< 0.05%	0.05%	
N_2		450 ppm	330 ppm	600 ppm	500 ppm Max
C					0.10% Max
O_2					0.2% Max
H_2					125 ppm Max
Total of Nonspecification Elements	0.60%	0.70%	0.55%	0.38%	0.4% Max

* High value reflects wet processing of chips. Subsequent comparison of dry chip weight to as-received weight enables determination of as-received moisture content

** Not measured — values typically below measurable limits after processing

From the degreaser, the superalloy chips dropped onto an oscillating screen which removed the dust and fines. The larger the mesh-size of the screen, the better the removal of the fines. Conversely, the losses were also proportionately larger, since the fines which were removed from the chips were applicable for refinery purposes only, rather than for vacuum melt applications, and as such were of considerable lower value. For the purpose of this program a 10-mesh screen was used.

From the screen, the chips moved onto a belt conveyor, passed under a standard cross-belt magnet, then under a custom-made high-intensity electromagnet. The cross-belt magnet removed tramp iron, tool steel, chrome steel and other highly magnetic contaminants. The high-intensity electromagnet had an adjustable field up to 12,000 gauss and was capable of removing extremely weak magnetic substances, like 302 stainless steel, Inco 901, or other substances which are normally considered as "nonmagnetic." The chips passed from the conveyor directly into the shipping containers.

A final sample was taken by drilling each container with an auger. Shipment of the material was made if the sample evaluation indicated conformance to the applicable specifications.

2. Analysis

The nickel alloy crusher sample, or the final sample, was quartered down to a 17 lb lot, and this amount was then melted into an ingot. In the case of the crusher sample, it was melted after

degreasing, and after removal of the magnetics from the sample in the laboratory. The melting was performed in a vacuum induction furnace at a pressure of 10 to 20μ. The melt was poured into a tapered ingot mold, which is approximately 3 1/2 in. in dia. The ingots were cooled under vacuum. After removing the ingot from the mold, a 1/2-in. slice was cut from the lower third of the ingot with an abrasive saw.

The ingot slice was analyzed on the X-ray spectrometer for Ni, Co, Fe, Ta, W, Mo, Cb, Cr, Zr, Ti, V, Mn and Cu. The X-ray spectrometer was calibrated with the best available standards for each alloy (NBS Standards are used where available, such as Waspaloy, Inco 718, Inco 901, Hastelloy X, etc.). An optical emission spectrograph was used for the determination of silicon, and for a general survey of any extraneous contamination. Atomic Absorption was used for aluminum determination. Carbon and sulphur were determined with a LECO CS-44 unit, after appropriate calibration. Trace elements were determined by flameless Atomic Absorption Spectrophotometry, using the procedure established by Welcher, Kreige and Marks of P&WA.[1] Lead and bismuth were determined on all samples. Ag, Sn, Sb, Te, Tl, Se were determined by the same procedure, as required. An additional unmelted portion of the chips was used to determine oil and moisture on the crusher samples, and bulk density if required. On the final sample, an additional unmelted portion of the chips was subjected to a visual inspection in the laboratory for general cleanliness, foreign metal contamination, etc. The ingot slices were used as a reserve sample and preserved for a period of not less than one year. The analytical records will be preserved for not less than ten years.

Table 5 presents the pre- and post-processing chemical analyses of the Waspaloy scrap supplied for the programs. The Waspaloy scrap deviates from specification because of excessive Bismuth content following processing.

TABLE 5
FRANKEL ANALYSIS OF WASPALOY LOT SAMPLES

	Crusher Sample	*Processed Sample*	*PWA 1007 Specification*
O&M	2.0%		
Magnetics	0.1%		
C		0.068%	0.02 to 0.10%
S		0.003%	0.02% Max
Al		1.25%	1.2 to 1.6%
B		0.004%	0.003 to 0.010%
Bi		5.5 ppm	0.5 ppm Max
Cb	<0.1%	<0.1%	
Co	13.5%	13.7%	12.0 to 15.0%
Cr	19.3%	19.35%	18.0 to 21.0%
Cu	0.03%	<0.05%	0.10% Max
Fe	1.30%	1.07%	2.0% Max
Mn	0.07%	0.06%	0.75% Max
Mo	4.00%	3.95%	3.5 to 5.0%
Ni	R*	R	R
Pb		<1 ppm	10.0 ppm Max
Si		<0.05%	0.75% Max
Sn		<0.05%	
Ta	<0.1%	<0.05%	
Ti	3.09%	3.00%	2.75 to 3.25%
V	<0.1%	<0.1%	
W	<0.1%	<0.1%	
Zr	<0.05%	<0.07%	0.02 to 0.12%
Bulk Density		84.2 lb/ft³	

*R Remainder

[1] G. G. Welcher, O. H. Kreige, and J. Y. Marks, Analytical Chemistry, Volume 46, page 1227, (1974).

SECTION VII

MATERIAL RECLAMATION TECHNOLOGY

A. SCRAP TECHNOLOGIES REVIEW

1. Background

P&WA reviewed the state of the art of separation processes available at the initiation of this program, and identified several applicable technologies. Some technologies were not chosen for program evaluation due to insufficient development status, proprietary status, uncertain economic potential, or prohibitive funding requirements to establish the process. Two separation processes based on the density separation principle, i.e., the ferrofluid process and the fluidized bed process, were selected for evaluation of separation capability in this program. This review was intended to update the available technologies for scrap reclamation and was based upon a literature review, personal discussions and related information as available.

Technologies have been classified into three basic separation concepts: physical/mechanical, pyrometallurgical/melting and chemical; and are described in the following portions of this review.

2. Physical/Mechanical Separation Processes

a. Conventional Manual and Visual Separations

Conventional separation procedures followed by most scrap dealers originate with visual conformity of material within the shipping container. If uniformity exists, spot checking with a hand magnet will spot gross magnetic contamination within the container. Inexpensive thermoelectric testers are utilized to identify pieces with known alloy standards and also to validate entire container alloy designation.

The color of aerospace metals is of some value as a means of identity. Copper, brass, aluminum and other nonferrous contaminants are color distinguishable. Much aerospace intermixture of Ti and Ni alloys is visually separable by competent scrap salvage department personnel by color and texture. Likewise, visual separation is often possible by mill or plant markings, although many instances of mislabeling have been noted.

b. Screening (Mechanical and Magnetic) Separations

Scrap turnings are usually reduced to ¼- to 1-in. length segments by crushing in a ring or jaw crusher. This crushing is likely to loosen entrapped contaminants such as tool bit particles.

(1) Mechanical Screening

Vibratory, mechanical screening is a standard procedure to separate various particle sizes after the crushing operation. This is beneficial in removal of dust, fines, grindings, etc., which generally are contaminated with interstitials and also in the removal of carbide chip contaminants. The latter, being brittle, tend to be reduced to fines during the crushing operation.

(2) Magnetic Screening

Magnetic screening is a standard procedure to segregate carbide chips as well as other magnetic (ferrous) materials. Both permanent and electromagnet separation varies in its effectiveness with magnetic response of the particular contaminant and its size. Consequently, current magnetic separators are not very effective in removing small and weak magnetic fragments.

Recent developments of a High-Gradient Magnetic Separator (HGMS) broaden the usefulness of magnetic separators for scrap reclamation. The HGMS units claim that even very small and weak magnetic particles (e.g., paramagnetic particles) can be separated. This device utilizes a high magnetic field coupled with high magnetic gradients. The process consists of a canister packed with a matrix of ferromagnetic filaments placed in a highly magnetic field. The matrix filaments divide the overall magnetic field and high magnetic gradients are created along the filament edges. The magnetic contaminants are then trapped by these filaments. These mechanical and magnetic screening techniques are limited to particle size and magnetic response and considered partially successful at best.

It has been estimated that up to 95% scrap segregation could be attained upon efficient application of the combined standard procedures of the aforementioned paragraphs (a) and (b) if the scrap generator and processor properly controlled alloy lineage. In reality, this level of control is not presently applied.

c. Size Reduction Separations

(1) Cryogenic Crushing

The Bureau of Mines has developed a cryogenic technique to separate many nonferrous alloys from others by cryogenic cooling to below the ductile/brittle transition temperature of some malleable alloys. Subsequent crushing during this cryogenic step readily pulverizes the alloy that has undergone transition, while other alloys remain malleable and resist crushing. Alloy separation is effected upon subsequent screening. Separations have been attained with various combinations of Cu, Al, Zn, steel and nonmetallics. The intention of this work is to effect reasonable levels of separation; however, the likelihood of complete separation has not been established.

(2) Hydrided Ti Crushing

A considerably different method to effect Ti separations consists of hydriding plus crushing plus dehydriding of Ti alloy chips. The hydriding readily embrittles the Ti without influencing contaminant stainless steel and WC tool bit fragments. Crushing thereby reduces the brittle Ti and WC to powder; these are readily separable by magnet. Continued development in this area was terminated due to incomplete (80-96% effective) WC separation and unacceptable oxygen contamination of the Ti powder.

Recent modifications to this hydride/screening/dehydride process include additional steps to reduce the contaminant oxygen level such as anodic dissolution and degassing. These steps have reportedly yielded a reclamation product of acceptable chemical and mechanical properties when care is taken to assure input scrap alloy lineage.

d. Radiographic Separation

High density contaminants in scrap (e.g., WC tool bit fragments) are distinguishable by radiographic or fluoroscopic screening. Radiography offers greater sensitivity; the advantage of the permanent film record is questionable here. Although an apparently costly procedure, it is

reported that one titanium melter uses radiographic examination of purchased turnings to search out carbide inclusions on a 100% inspection basis after such turnings have gone through several steps of mechanical screening and magnetic separation. The turnings are spread in a thin layer on trays, or formed into briquets, and radiographed. The film is examined for any inclusions, which then can be removed manually by comparing the film with the tray or briquet.

e. Fluoroscopic Separation

Fluoroscopy can likewise be utilized to inspect turnings. This inspection is considerably more cost-effective than radiography, but particle resolution is markedly reduced. Small fragments of high density and foreign alloys are less detectable. Many melters have fluoroscopes to inspect incoming sponge for other types of inclusions, but they have not used fluoroscopes effectively to screen turnings other than, in some cases, their own internally generated turnings for which the history is well known.

f. Density Separations

Sink-float separation of an alloy from a contaminant can be accomplished if the density of the alloy differs from that of the contaminant and also if the density of the separation fluid is between that of both the alloy and contaminant. Separation efficiency is increased as the magnitude of the difference in density between alloy-fluid-contaminant increases.

There are ample density differences in alloys of concern for scrap recovery. For instance, titanium and its alloys have densities of 4.4 to 4.7 gm/cm^3, while aluminum has a density of about 2.7 gm/cm^3, steels, stainless steels, and superalloys have a density in the range of 7.7 to 9.0 gm/cm^3, and tungsten carbide tool bits have a density in excess of 15.0 gm/cm^3. Thus, the density differences provide a convenient basis for the purification of titanium or for the separation of contaminant titanium or aluminum from scrap superalloys.

Three density-based separation processes were considered for scrap recovery: conventional dense-media processes, dry fluidized bed processes and the ferrofluid process.

(1) Conventional Dense-Media Processes

These processes rely on the choice of various fluid media that have densities between the alloy and contaminants of concern. Conventional dense-media processes, using a water-based slurry of magnetite or ferrosilicon, have an upper density limit of about 3.3 gm/cm^3. Thus, they would be capable of making a sink-float separation of aluminum (2.7 gm/cm^3) from titanium (4.4 to 4.7 gm/cm^3). Other media, such as acetylene tetrabromide (2.9 gm/cm^3), methylene iodide (3.32 gm/cm^3) and thallium salt solution (3.5 to 4.9 gm/cm^3) could be utilized for separation of some titanium alloys; however, each of these fluids are expensive, hazardous and toxic. Another handicap is that all of these fluids are not capable of separating the denser contaminants such as steels, superalloys and carbide tool chips from titanium.

Heavy media success has been reported in upgrading the metallic content of shredded auto scrap rejects using conventional sink-float procedures and finely ground magnetite, ferrosilicon, and galena as media. However, there does not appear to be active industrial usage of the conventional dense-media process at this time.

(2) Dry Fluidized Bed Process

A variation of the dense-media process is the dry fluidized bed process, wherein the separation medium is a bed of dry granules or powder of some heavy material such as lead or iron. The bed is "fluidized" by an air stream so that the medium has an apparent density of something less than that of the contaminating materials. A mixture of materials of different density can be made to flow through the fluidized bed and effect a sink-float separation if the apparent density of the medium is intermediate to that of the materials to be separated.

The Frankel Company fluidized bed process, using a fluidized bed of titanium or nickel alloy chips supported in air media was included in this program of evaluation of scrap reclamation technologies. This process is being used industrially in high-volume applications to effect scrap reclamation.

Reasonable success has been reported in studies on the recovery of nonferrous metals (Al, Cu, Zn) from shredded auto scrap rejects by use of the fluidized air bed process. Also, the "Dryflo" system, manufactured in England, is being marketed in this country for the separation of plastic insulation materials from finely-granulated copper or aluminum electrical wire. Details of this process were unavailable.

(3) Ferrofluid Process

The ferrofluid metal separation process is based upon an ability to vary the density of a magnetic fluid. This process is applicable to nonferrous metal separations. The magnetic fluids, known as ferrofluids, are stable colloidal dispersions of a ferromagnetic material in a carrier liquid, and they behave as magnetic liquids. There has been considerable research in this area and industrial usage should be forthcoming.

High separation rates have been attained in the separation of crushed, nonferrous auto scrap. Also, this process is being applied to studies on scrap recovery and ore separation. One area includes the recovery of nonferrous metals from incinerator residues.

The ferrofluid process, as conducted by AVCO, was included in this program of evaluation of scrap reclamation technologies. The AVCO process uses a pool of ferrofluid suspended between the poles of an electromagnet. An input conveyor delivers the incoming mixed metals to the pool. Output conveyors take away the separated "float" and "sink" fractions, respectively. A unique characteristic of the system is that the effective density of the ferrofluid pool may be varied at will from less than 1 gm/cm^3 to well over 20 gm/cm^3 by simply changing the current to the electromagnet. Separation of objects differing in density by as little as 10% has been demonstrated and routine separations of Al, Ti, superalloys, and WC have been established.

Lastly, there are indications that separation of superalloys with similar densities but different magnetic properties may be feasible. The weak magnetic properties may influence the true density and effect a separation. Initial experiments have been inconclusive and further work is necessary here.

3. Direct Consolidation Processes

Considerable effort has been expended in developing direct consolidation processes for Ti alloy scrap reclamation. These efforts usually rely on adequate cleaning procedures prior to consolidation to reduce interstitial levels that are inherent with Ti chips. Consolidation of scrap Ti chips is then conducted similar to the consolidation of virgin Ti powder. In theory, Ti alloys "scrub" their surfaces clean by elevated temperature (below beta transus) dissolution of

interstitials thereby giving fully diffusion-bonded billets. Chip compaction has been reported by hot isostatic compaction, hot extrusion, and hot vacuum pressing. Direct consolidation eliminates melting requirements and yields extrusions or billets available for subsequent forging operations.

An interesting variation of the direct consolidation process has been developed at the P&WA/Florida R&D Center for the reclamation of titanium alloy scrap. Machine turnings are cleaned by conventional perchlorethylene solvent degreasing and magnetic contaminants are removed by a high-density magnetic separator. The turnings are then hydrided and pulverized to a fine particle size. Coarse particles (i.e., — major contaminants such as stainless steel, superalloys, etc.) do not hydride and then pulverize; they are retained on a 60 mesh (200 micron) screen size and discarded. Minor contaminants are then removed by anodic dissolution of the hydrided particle fines. Anodic dissolution is effectively a reverse-plating process whereupon soluble contaminants are removed from the insoluble Ti alloy. The clean Ti fines are then rewashed and processed through a semicontinuous vacuum hot press facility that has been modified to incorporate a preheated, vacuum dehydriding section. The final step in this facility is vacuum hot compaction of the clean, separated Ti particles into a billet. Conventional reduction and forging operations, applied to this billet, have produced aerospace-quality components. Process scale-up is required to define production capabilities and resolve the true cost effectiveness.

Two technical problems exist. The effectiveness of preconsolidation cleaning in consistently maintaining specification interstitial levels must be established. Also, direct consolidation is not a separation process; alloy separation of scrap chips must be assured to avoid off-grade compositions in the final product. In addition, the economics of direct consolidation processes must be ascertained.

4. Chemical Separation Processes

In theory, the main advantage of chemical recovery processes is that they can be utilized to handle any segregated or unsegregated scrap. Furthermore, they produce essentially elemental raw material equivalents.

In reality, several studies have been applied to the recovery of Ti and superalloy scrap by chemical processes with varying degrees of success, mostly seen in the superalloy field. It is believed that superalloy scrap, after suitable pretreatment, is being utilized in Canada and Europe as feed material in existing hydrometallurgical operations for nickel and cobalt recovery. These processes are viable only because the nickel and cobalt reclaimed from scrap represents a small fraction of the total metal production of these operations. Accordingly, use of such processes solely for the reclamation of superalloy scrap in the U. S. is currently economically unfeasible.

a. Modified Sponge Process for Ti Scrap

A potential chemical extraction process for recovery of Ti alloy scrap chips is a modification of the Kroll sponge process. Scrap is mixed with $TiCl_4$ in a first-stage molten salt reactor to yield a Ti/Cl/molten salt product. This product is reduced with Mg in a second-stage reactor to yield Ti sponge after an appropriate distillation step. To our knowledge, this process has not been reduced to practice.

b. Exothermic Disintegration/Chemical Extraction Process for Martensitic Stainless Steel Scrap

A number of processes are known for the separation of nickel, cobalt and copper values from ferrous alloys. In the treatment of ferro-nickel, for example, the alloy is ground to a fine powder

and leached in aqueous oxygenated ammonium sulphate or sulphuric acid for extraction of nonferrous values.

These known processes are generally unsatisfactory for extraction of contained nickel, cobalt and copper values from iron-chromium scrap alloys. Such material is extremely tough and nonfriable and can only be ground to a leachable size with great difficulty. The problems encountered in grinding such material are greater still if the material contains abrasives such as alumina or other alloying metals such as manganese, molybdenum, and tungsten. Furthermore, even after grinding such material to a fine state, the material is usually resistant to leaching. Severe leaching conditions and prolonged times are required to extract nickel and other desired values such as cobalt and copper from the material.

A Canadian process has been reported to be useful in the recovery of Ni, Co, and Cu from Fe-Cr scrap turnings and chips. The process involves contacting the alloy with an alkali metal sulphate or an alkali metal sulphite additive and heating the alloy and additive under controlled reducing conditions to initiate and continue an exothermic reaction. The reaction product is then quenched to cause disintegration thereof to a finely divided state amenable to a leaching treatment for extraction of nickel, cobalt and copper values. There is insufficient information to establish if this process has been reduced to practice.

c. *Solvent Extraction Processes for Superalloy Grindings*

A laboratory chemical extraction process has been disclosed to recover Ni, Co, Mo, and Cr from superalloy grindings. These metals comprise the greater part of waste produced during melting, casting, and machining of nickel- and cobalt-based superalloys. Much of this scrap is best utilized by remelting; however, when the scrap is contaminated, consists of grindings, or is of obsolete alloys, chemical processing can reportedly convert the contained metals to marketable forms.

The process developed included the following operations: Contaminated scrap was cleaned to remove trash, cutting oils, and nonmetallic grinding wheel debris. Cleaned scrap then was dissolved in hot, chlorinated, diluted hydrochloric acid. Tungsten and traces of silica were removed from the leach liquor by carbon absorption followed by three successive solvent extraction operations to separate and recover molybdenum, iron, cobalt, and manganese. Finally, chromium was separated from nickel contained in the partly purified solution by selective precipitation.

Massive alloy shapes were not used in this study because the corrosion rate of such material was relatively slow. Turnings, borings, and grindings dissolved rapidly and therefore appeared most suitable for chemical recovery.

This process has not attained commercial practice and has not received an economic evaluation. Nevertheless, there would appear to be interest in this process in that it can feasibly handle all forms of superalloy scrap. An area of potential concern is reduced solubility of some superalloys in HCl.

An apparently similar solvent extraction process has been reported in England. Process scrap is dissolved in hot aqueous HCl and then the metals of interest are purified and isolated by a series of precipitation, leaching and solvent extraction stages. A desirable feature of the process involves the use of HCl for the dissolution and MgO for the main precipitation stages. These reagents are obtained by a high temperature hydrolysis of the $MgCl_2$ formed in the end solution, thereby reducing pollution problems.

Difficulties in HCl dissolution of some superalloys is again of concern. No evidence of reduction to practice has been obtained on this process.

5. Pyrometallurgical (Melting) Processes

In the recovery of scrap there are generally two objectives of pyrometallurgical processes: physical separation and elemental refinement.

Upon bulk melting of scrap, insoluble inclusions of higher melting points and higher densities will settle to the bottom of the melt; lighter compounds i.e., -- slag and oxides, tend to rise to the surface of the melt. This physical separation phenomenon can be utilized in nonconsumable melting processes to separate contaminants by selective decanting of the melt.

Elemental refinement can be realized in melting processes by the removal of volatile contaminants via vaporization; vacuum melting processes greatly enhance this separation.

Pyrometallurgical processes cannot be used to separate nickel, cobalt, iron and copper. In addition, only under exceptional circumstances can molybdenum and tungsten be separated from the above elements and current pyrometallurgical processes have only limited capabilities in removing some trace elements such as tin. This means that if pyrometallurgical processes are to be used, the furnace charge must have an average composition that is fairly close to some commercial alloy grade. Fortunately, a large amount of superalloy waste is presently segregated by grade, and it is technically feasible to segregate an even larger fraction of these wastes. Pyrometallurgical processing is relatively inexpensive and well established.

Several pyrometallurgical processes are being evaluated and/or are in present industrial usage to recover scrap. While usually discussed with reference to a particular system (titanium or superalloy), they are not necessarily limited to these materials. A brief review of these processes follows.

a. Induction and Arc Air Melting

Air melting can be achieved by induction heating with the use of a suitable flux, or in a conventional air arc furnace. These methods are used on a limited basis for melting less critical alloys.

Miscellaneous machining chips can be melted in an air furance. A typical charge may include both air and vacuum grade superalloys. The melt can be analyzed, oxidizable elements oxidized (if necessary), and the bath composition adjusted to produce a useful composition. The quality of the resulting product as well as the profitability depend upon the average composition of the charge and current market conditions.

A large variety of products can be produced. The range runs from compositions suitable for use in vacuum induction melting to remelt stock suitable for use in stainless and specialty steels. In most cases, some of the valuable elements in the original alloy are lost. Columbium would be lost from Inco 718 alloy during air melting while cobalt would be a useless but tolerable contaminant in non-nuclear grade stainless steels. Consequently, air melting is an economical approach for consolidating superalloy chips, but its use is severely limited for many reclamation cycles.

b. Vacuum Induction Melting

Vacuum induction melting, performed in a ceramic crucible, is widely used to prepare superalloy master melts and castings.

The recycling of segregated superalloy solids is in limited industrial practice; likewise, the recycling of superalloy chips is of less industrial popularity because of the alloy segregation problem. Grindings and dust are not normally recovered because an untenable level of slag is produced in addition to the alloy segregation problem.

The vacuum induction process is not well suited to refining because a prohibitively long time is required to effect surface vaporization of principal contaminants. Carbon deoxidation is readily achieved; excessive superheat can cause crucible interaction and subsequent contamination.

c. Vacuum Consumable Arc Melting

Vacuum consumable arc melting followed by remelting is a traditional process for production of Ti. An arc is established between a consumable electrode and a water-cooled, copper mold. The process exposes a large quantity of the melt to the vacuum environment and produces a sound ingot with minor levels of segregation.

However, as applied to scrap recovery, the process is limited by "green strength" compaction requirements of the consumable electrode. Scrap additions exceeding about 25% of the electrode-total reduce the electrode integrity; electrode fracture during melting thereupon causes an aborted melt. Also, as mentioned earlier, consumable melting does not offer the decanting capability for separation of heavy and/or light contaminants. Insofar as scrap recovery is concerned, the process is more suited to final processing of materials that were upgraded by another process. Despite these process limitations, this process has found reasonable acceptance by several industrial scrap melters for specific applications.

d. Nonconsumable Melting

Five nonconsumable melting processes, or modifications thereof, offer potential advantages with respect to scrap reclamation. The status of these processes, applied to scrap, ranges from laboratory demonstration to trial heats to industrial production (depending upon the process).

(1) Nonconsumable Vacuum Rotating Arc Melting

The process is based upon the concept of sinking dense, contaminant particles i.e., WC, into a harmless skull area. Earlier work utilized conventional nonconsumable melting equipment. The rotating electrode was applied to this area and extensive additional work was thereupon performed. This latter process, known as the nonconsumable rotating electrode melting process, has been applied as a vacuum arc melting process and offers an improved system specifically designed for Ti or superalloy scrap reclamation. The process features a Schlienger-designed furnace with a rotating water-cooled copper nonconsumable electrode, a water-cooled copper melting crucible casting mold and a feeding system capable of introducing a variety of unconsolidated raw material forms into the melting crucible.

The process has demonstrated the capability of processing large amounts of reasonably segregated scrap (in various forms) to produce high quality cast remelt ingots. In addition, the process is capable of removing heavy inclusions (e.g., tungsten carbide chips) through entrapment in the titanium skull.

The process has several advantages over consumable arc melting processes. Higher pool temperatures can be sustained for controlled periods, enabling partial solution of low density inclusions as well as complete melting and alloying. In addition, the process is amenable to

processing materials in a manner which prevents extreme pool temperatures in high vacuums with consequent loss of volatile alloying elements.

A reduced melt volume and absence of hot topping are potential disadvantages which can result in compositional variations if the feedstock varies appreciably. For these reasons, nonconsumable arc melting is conventionally followed by a consumable electrode remelt.

A variation of the rotating electrode process, the Durarc process, features a stationary electrode with arc rotation induced magnetically. A further variation combines a rotating electrode with a magnetically inducted arc oscillator.

(2) Nonconsumable Plasma Arc Melting

Nonconsumable plasma arc melting utilizes a gas stabilized arc which stabilizes and improves the control of energy concentration within the melt. The electrode melt rate and heating of the liquid metal bath can be theoretically controlled. High heat transfer rates and high gas velocities are attainable. Plasma arc furnaces offer the option of operating under high temperature and high pressure gas environments.

In practice, utilization of the plasma arc can be varied considerably due to the arc control achievable. Impingement upon the surface of a molten pool into which particulates are added, or impingement of the arc onto an electrode feed rod which drip melts, are being evaluated. One important limitation of the plasma arc process is the existence of a moderate pressure gas environment resulting from the required arc stabilizing gas.

An extensive evaluation of the performance of plasma arc furnaces, including an assessment of their capability for superalloy scrap recycling, has been reported. Small heats of Inco 718 were plasma induction melted using nondegreased scrap. Chemical analysis indicated partial refining occurred with complete removal of Sn. Preliminary studies using the Schlienger nontransferred plasma arc process encountered heat transfer problems in that only a shallow molten pool could be maintained. It would appear that nonconsumable plasma arc melting is a viable process for superalloy or Ti scrap reclamation, but additional development effort is required.

(3) Nonconsumable Electron Beam Melting

Active interest exists in the reclamation of scrap by the nonconsumable electron beam melting process and several process variations have surfaced. Although all efforts appear to have been directed toward Ti scrap reclamation, it would appear that the process would also be amenable to superalloy scrap.

Extensive work has been performed using a Leybold-Heraeus electron beam skull furnace. Segregated, crushed, degreased and dried titanium scrap is fed into a furnace and non-consumably melted dense contaminant particles, i.e., WC, etc., sink harmlessly to the bottom of the skull, while metal at the top of the pool is caused to overflow by directing the electron beam across the pouring spouts to pour the metal in small slugs or drops. These drops are solidified on a rotating drum, cooled on a conveyor, and collected in a container as pellets. The pellets are then blended, analyzed, and melted in a conventional consumable electrode furnace. The pellets can be added to sponge at the compacting press or side fed to the consumable or nonconsumable electrode furnace during the melt.

The principal advantage of this process is the conversion of titanium scrap into a very usable form of revert material. Completion of the scrap reclamation cycle requires conversion of this revert material into a nonconsumably or consumably melted ingot.

This concept of sinking of dense contaminant particles into a harmless skull area was utilized in an electron beam nonconsumable skull melting process that has the additional advantage of converting Ti scrap chips directly into ingot, as compared to the aforementioned process that produces revert pellets. In this process, segregated, crushed, degreased, and dried Ti scrap is continuously fed into a nonconsumable skull melt. Dense contaminants sink to the water-cooled skull bottom, while molten metal is poured from the skull lip into an ingot-retracting water-cooled mold. Electron beams are focused upon both the skull and mold surfaces to maintain fluidity and solidification requisites. This process, utilized by AIRCO, was selected and evaluated during the Phase I portion of this contract.

This evaluation showed that complete separation of seeded WC contaminant was attained; the resulting cast Ti ingot had a desirable homogeneous macrostructure (equiaxed and relatively fine grain). The high vacuum level necessary to maintain electron beam was beneficial in removing volatile contaminants; however, areas of ingot nonhomogeneity also occurred (i.e., partial Al vaporization). A revised feed system is necessary to correct this problem of uniform co-addition of Ti sponge, scrap and master alloy to the electron beam furnace. A further advantage of this process is that the end product is potentially a single-melt ingot of a quality comparable to that of consumable arc melting.

(4) Electroslag Melting

A process conceptually exists for reclamation of superalloy scrap processed in a modified ESR furnace. The process is basically a consumable electrode arc melting process with the important distinction that the melting takes place within a molten flux. During its passage through the flux, the molten metal is scavenged of various impurities as well as protected from oxidation. The mold is basically divided into two compartments. Scrap, in the form of grindings or turnings, is fed into the skull melting side where it is melted, and, subsequently, the molten alloy flows over the dam into the ingot-forming portion of the unit where the ingot is continually withdrawn from the mold. The potential advantages of the process over conventional techniques result from reduced labor and material cost (combined with such factors as charge make-up and refining reactions), ability to process most scrap forms, and the elimination of at least one conventional induction melting operation.

(5) Inductoslag Melting

A cold-mold induction-melting process, reportedly capable of handling 100% Ti scrap, has recently been developed. The basic equipment required to induction melt reactive metals, a split water-cooled crucible, was developed much earlier. The current research effort incorporates slag melting with improvements in induction melting crucible design and designated as "inductoslag melting."

The process features a water-cooled copper crucible with a surrounding induction coil. Longitudinal slits in the copper crucible limit inductive coupling to the crucible and preclude excessive heating of the crucible. In addition to molten and solidified metal, the crucible contains a small amount of a slag such as calcium fluoride that freezes in the crucible slits, thereby insulating them from each other while forming a thin thermal barrier between the ingot and the crucible. Some slag floats on top of the molten pool, but the cover is only partial and most of the liquid metal surface is exposed to the ambient gas which is usually an inert gas at about one-third atmospheric pressure. Properties of annealed unalloyed titanium plate produced from inductoslag melted sponge compare favorably with arc melted material.

Potential slag-induced (CaF) contamination imparted to reactive metals is of concern; application of the process to superalloy scrap may be preferable. Also, the process has not been attempted on any commercial scale to our knowledge.

e. Comparison of Melting Process Parameters

The basic parametric features of several pertinent melting processes as applied to titanium scrap reclamation have been catalogued. Interpretations and estimates have been applied where deemed necessary.

Melting processes catalogued are vacuum consumable arc (Section 5c), nonconsumable vacuum rotating arc (Section 5d (1)), nonconsumable plasma arc (Section 5d (2)), and two versions of nonconsumable electron beam (Section 5d (3)).

6. Scrap Technologies Recommendations

Several of the aforementioned technologies for scrap reclamation have attained industrial practice either in their original state or combined modifications thereof. Many other technologies would appear to have technical merit and could readily attain industrial acceptance with sustained developmental support. Two Ti processes should receive consideration for future development support based upon this literature review of available data. These processes are direct consolidation and nonconsumable electron beam melting. Direct consolidation could appear to be a viable method for Ti scrap reclamation if consistent control is maintained of the Ti alloy scrap lineage, thereby avoiding the uncorrectable situation of mixed Ti alloys and varying chemical configurations. Nonconsumable electron beam remelting had demonstrated the feasibility of Ti scrap reclamation. Feed control problems must be resolved here to assure consistent feed of the scrap/sponge/master alloy to ensure consistent chemical homogeneity of the ingot product.

The selection of the two density separation technologies (dry fluidized bed, Section 2f (2), and ferrofluid, Section 2f (3)) utilized in Phase I of this contract still appears valid for scale-up studies. This review of all other aforementioned technologies indicated that technology redirection was not advisable.

B. DENSITY SEPARATION PROCESSES

Two density separation methods, i.e., the AVCO ferrofluid method and the Frankel fluidized bed method, were investigated. The AVCO method was applied to both titanium and nickel while the Frankel method was applied to titanium only. The AVCO ferrofluid process accomplishes separation by introducing scrap into a fluid in which a variable apparent fluid density may be induced magnetically. Separation is achieved by the controlled sinking or floating of known density scrap constituents. The Frankel system utilizes an air fluidized bed to achieve an apparent density medium for separation of scrap by rising or sinking. The ability of these processes to cost-effectively separate contaminants from scrap was assessed by a series of experiments utilizing scrap with known contamination.

1. AVOC Ferrofluid Method

a. System Technique

The AVCO Corporation developed a process to separate nonmagnetic solids according to density, by a sink-float mechanism, in a "ferrofluid" situated within a controlled magnetic field. Ferrofluids are stable colloidal dispersions of superparamagnetic particles that retain their liquid

properties in a magnetic field. These colloidal dispersions form a unique class of magnetic fluids. In the presence of a suitable magnetic field, ferrofluids can float nonferrous metals which are more dense than the ferrofluid and would therefore sink in the absence of the magnetic field. As a result, ferrofluids are said to have an "apparent density" which is controllable by a magnetic field. The apparent density of a ferrofluid can be varied from about 1 gm/cm^3 to well over 20 gm/cm^3 by adjusting the current applied to a surrounding electromagnet.

Any two nonmagnetic or weakly-magnetic metals differing in density by about 10% may be separated by adjusting the apparent density of the ferrofluid pool to a value intermediate between that of the two metals. Upon introducing the mixed metals, the less dense material floats and the more dense material sinks. Mixtures of three or more metals may be separated by multiple passes through the separator, with the ferrofluid density being adjusted each time to either float or sink only one of the metals present. The ferrofluid separation method, applied to aerospace scrap metal, provides for the separation of wanted, from unwanted fractions of the scrap mixture on the basis of their different densities. As shown in Table 6, most contaminating materials differ in density from that of the desired titanium or superalloy by more than 10%.

TABLE 6
DENSITY OF AEROSPACE METALS AND TYPICAL CONTAMINANTS

Material	*Density gm/cm^3*
Aluminum	2.7
Titanium and Titanium Alloys	4.4 to 4.7
Superalloys	7.9 to 8.8
Copper Alloys	8.3 to 8.9
Lead Alloys	10.0 to 11.3
Tungsten Carbide	About 15

Process principles are described in the following paragraphs.

A ferrofluid placed in a nonhomogeneous magnetic field experiences a net magnetic force which tends to drive it, like all magnetizable objects, towards regions of highest magnetic field intensity. The magnetic body force, F_M, per unit volume of fluid, is proportional to the induced magnetic dipole moment, M, and to the applied field gradient ∇H:

$$F_M = M \nabla H \tag{1}$$

When a nonmagnetic object is immersed in a ferrofluid in the presence of a magnetic field gradient, as shown in Figure 3, there is a magnetic force, F_M, on the object which tends to expel it to a region of minimum field. This magnetic body force is equal to, but opposite in sign to F_M defined above. If the magnetic field gradient is parallel to the direction of gravity, the magnetic body force can be used to cancel the gravitational body force, F_g, on a nonmagnetic object immersed in a ferrofluid. Consequently, an object of high density can float in a ferrofluid of low density when F_M is greater than F_g. F_g is given by Archimedes:

$$F_g = (\rho_s - \rho_f)g \tag{2}$$

This effect is demonstrated when a copper ball with a density, ρ_s, of 8.90 gm/cm^3, floats on a ferrofluid with a density, ρ_f, of 1.14 gm/cm^3, placed in a gradient field of 1500 oe/cm, established by the tapered poles of a permanent magnet. The fluid magnetization, $4\pi M$, is 200 gauss. In this case, the product, $M \nabla H$ (24,000 dynes/cm^3) is high enough to float even the densest metal, osmium, which has a density of 22.48 gm/cm^3.

When the object immersed in the ferrofluid is not totally nonmagnetic, the above treatment needs to be modified somewhat. If the magnetic dipole moment of this object is smaller than that of the ferrofluid, it is still forced from the region of high field to the region of low field. The magnitude of the force is smaller, however, than it would have been, had the object been completely nonmagnetic. If the magnitude of the object's magnetic dipole moment is greater than that of the ferrofluid, it will move to the region of high magnetic field and force the ferrofluid to the region of low field.

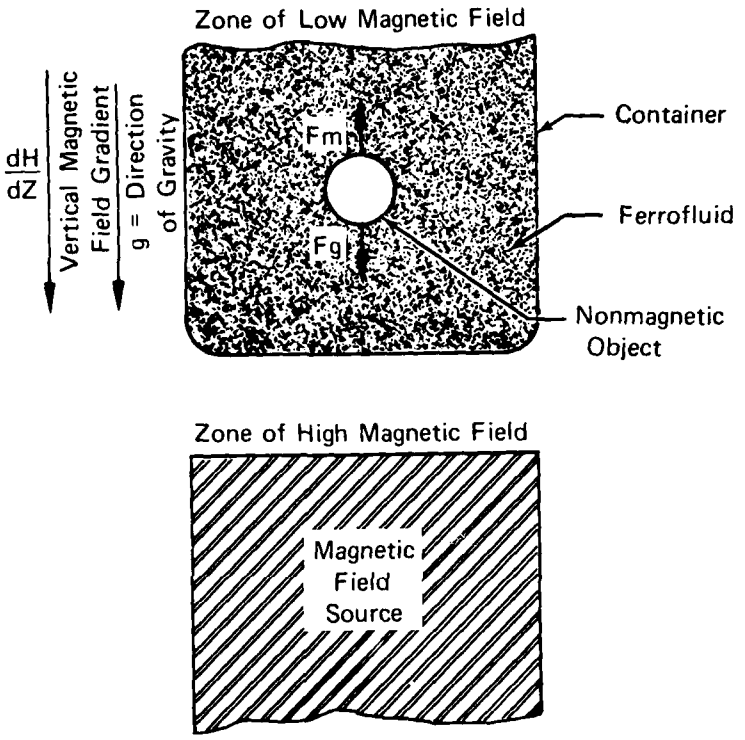

Figure 3. Forces on Nonmagnetic Body Immersed in Ferrofluid — AVCO

A pool of ferrofluid in the gap of a regulated electromagnet thus becomes a liquid whose apparent density can be continuously varied by controlling the current supply to a suitably designed field source. Thus, solid objects of different densities can be made to float or sink simply by varying the electrical current to the magnet. In this context, it is useful to define a ferrofluid's apparent density, ρ_a as:

$$\rho_a = \rho_f + \frac{\mathbf{M} \nabla \mathbf{H}}{\mathbf{g}} \tag{3}$$

From these equations, it can be seen that objects having a density lower than ρ_a will float in the ferrofluid, while those having a density greater than ρ_a will sink. By controlling the magnetic field and thus M and ∇H, ρ_a can be set to a value intermediate between the densities of the objects immersed in the pool, and their separation thus accomplished.

This separation technique is coupled to a means of removing the ferrofluid from the separated objects. For the common kerosene-based ferrofluids, this can be accomplished by washing the objects with a chlorinated hydrocarbon solvent or a hydrocarbon solvent, which has a lower boiling point than the kerosene. The ferrofluid is recoverable by boiling away the solvent. A schematic of such a process is shown in Figure 4.

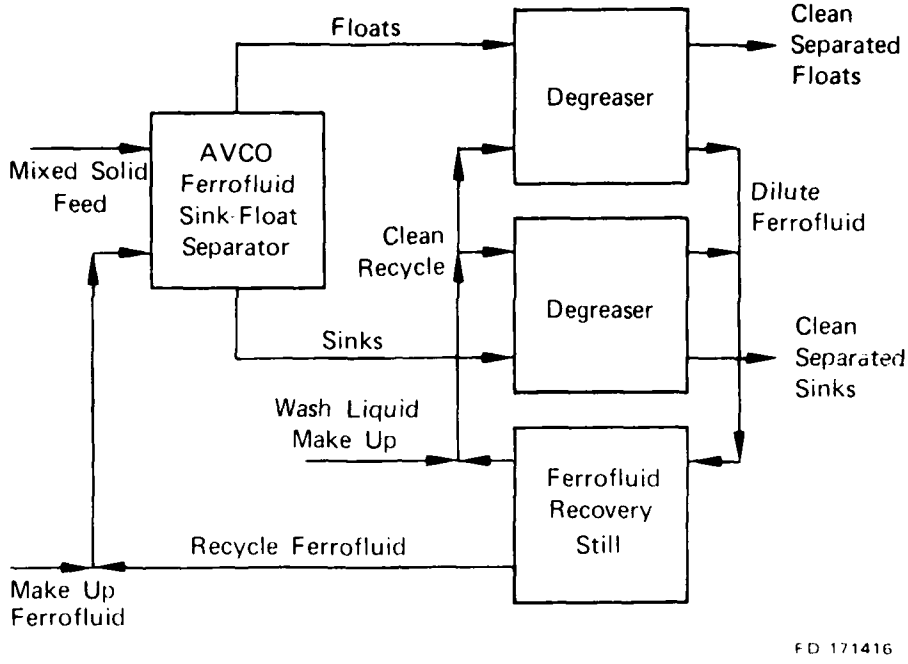

Figure 4. Schematic of AVCO Ferrofluid Recovery System

b. System Hardware

(1) Pilot Plant

AVCO designed and erected a pilot plant to purify nonferrous metal scrap by ferrofluid density separation. The key element of this facility is the ferrofluid scrap separation module. In addition to the separator and its power and control subsystems, the facility includes receiving and storage areas, auxiliary screening and magnetic equipment, product wash and ferrofluid recovery subsystems, as shown schematically in Figure 5.

This facility is capable of handling a wide variety of clean, particulate scrap approximately 1/8 to 3 in. in size, including crushed turning, cobbles and other irregular pieces. Mixtures which contain large quantities of magnetic contaminants can be handled in this facility by prepurification on a standard magnetic pulley separator.

(2) Separator Magnet

The AVCO sink-float separator consists of a magnet capable of generating a downward magnetic field gradient, and a materials handling system for containing the ferrofluid, introducing the mixed feed and removing the separated product.

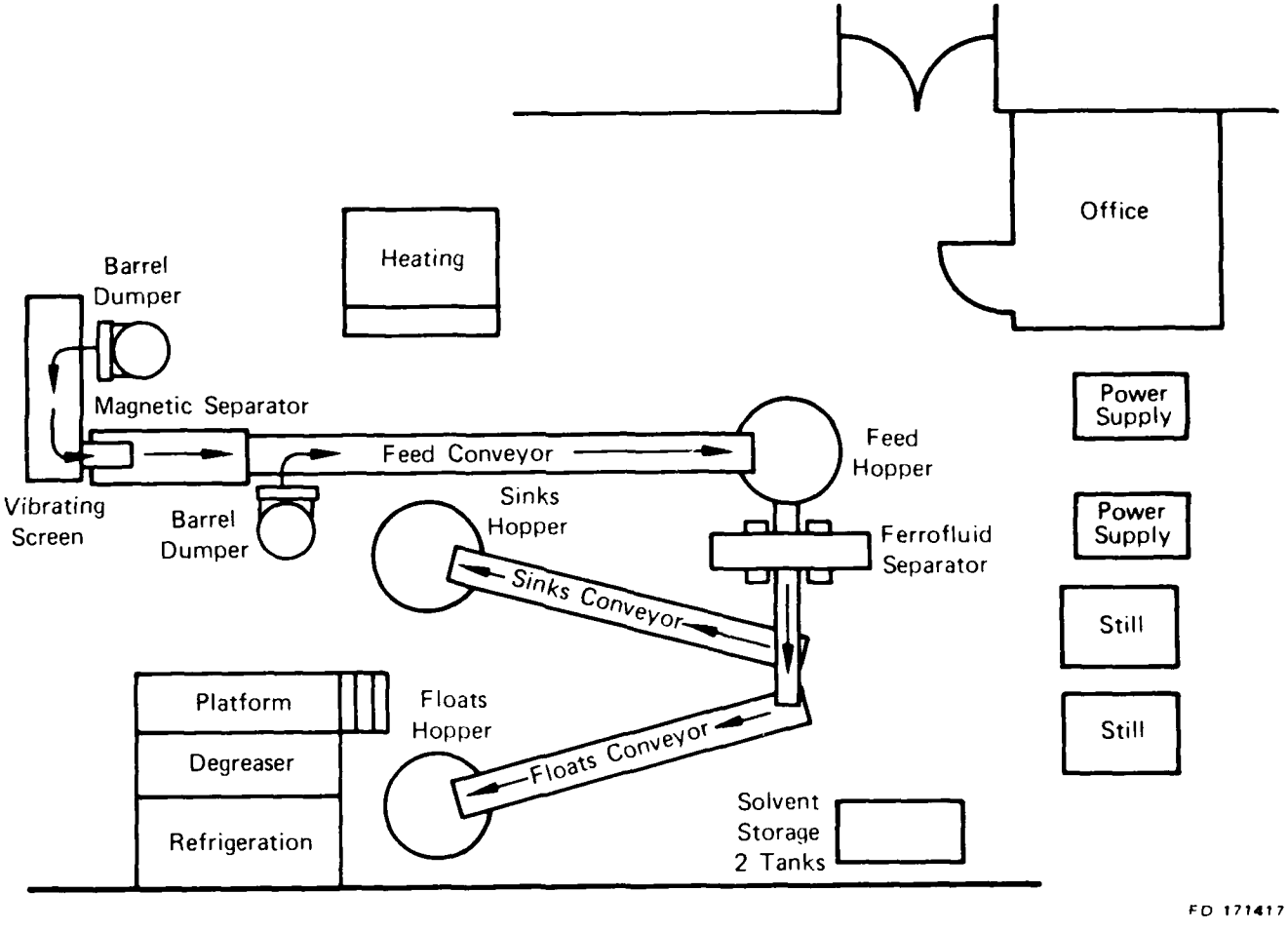

Figure 5. AVCO Ferrofluid Separation Pilot Plant

An iron yoke electromagnet with a modified rectangular hyperboloid pole configuration has been found to be a practical design for the field source of the separation modules built to date. This design configuration results in a constant gradient in the direction of gravity. The magnet is energized to the minimum magnetic field level for near saturation of the ferrofluid placed in the working volume. The magnetization of the ferrofluid will thus not change significantly over the height of the working volume, and as a result, the apparent density of the ferrofluid in the working volume is essentially constant. Apparent density of the ferrofluid is changed by increasing the gradient field, i.e., by increasing the current to the electromagnet.

The iron-yoke magnet design allows horizontal access to the pool of ferrofluid, and since the magnetic forces retain the ferrofluid in the gap of the magnet, conveyors or other means of introducing the feed or removing the separated products can be introduced directly into the ferrofluid pool without fluid leakage or sealing problems. AVCO has built a number of separator magnets of different scale based on the magnet configuration described above. The industrial-scale pilot plant separator magnet, to be utilized in the current program, is shown in Figure 6. This magnet generates a magnetic gradient constant to about 5% in a cube 8 in. on a side, located between the poles; at maximum power (63 Kw), a gradient of 250 oe/cm is generated. Under these conditions, a 500 gauss kerosene-base ferrofluid will have an apparent density of nearly 12 gm/cm^3. This density is sufficient to float all common industrial metals of interest. The DC power required to energize the electromagnet is provided by two standard 40 Kw welding generators.

(3) Separation Chamber

The separation chamber is a vessel that fits between the magnet pole pieces. The function of the chamber is to confine the ferrofluid pool and to provide means for introducing the mixed solids and for removing the separated products. For scrap metal separation, this design was adapted to the magnet constructed for the pilot plant by using suitable conveyors and increasing the scale by about a factor of 4. The conveyors were designed to carry scrap fragments up to about 2-3 in. in largest dimension at rates of over a ton an hour. A sketch of the prototype chamber is shown in Figure 7.

Since its original assembly several years ago, a number of modifications in the positioning and configuration of the feed and product removal conveyors were made to obtain reliable and jam-free operation of the separator. These modifications include: (a) extending the floats conveyor and the sinks conveyor beyond both edges of the magnet gap so that the ferrofluid pool is completely swept at its top and bottom, thereby eliminating dead zones where scrap can accumulate; (b) replacing the combed flights of the floats conveyor with solid elastomer flights in which scrap pieces cannot be entangled; (c) using undulating elastomer in the sinks conveyor to simplify its construction and prevent crevices where pieces of scrap could jam; and (d) replacing a horizontal feed conveyor with a slanted chute which guides the feed into the side of the ferrofluid pool. In this configuration, the particles in the feed fall into the pool under the influence of gravity with some mechanical assistance from a small paddle wheel whose axis is at the ferrofluid/air interface.

In addition to the feature directly related to scrap movement, the chamber also has an automatic system for controlling and recording the apparent density of the ferrofluid to maintain uniform processing conditions.

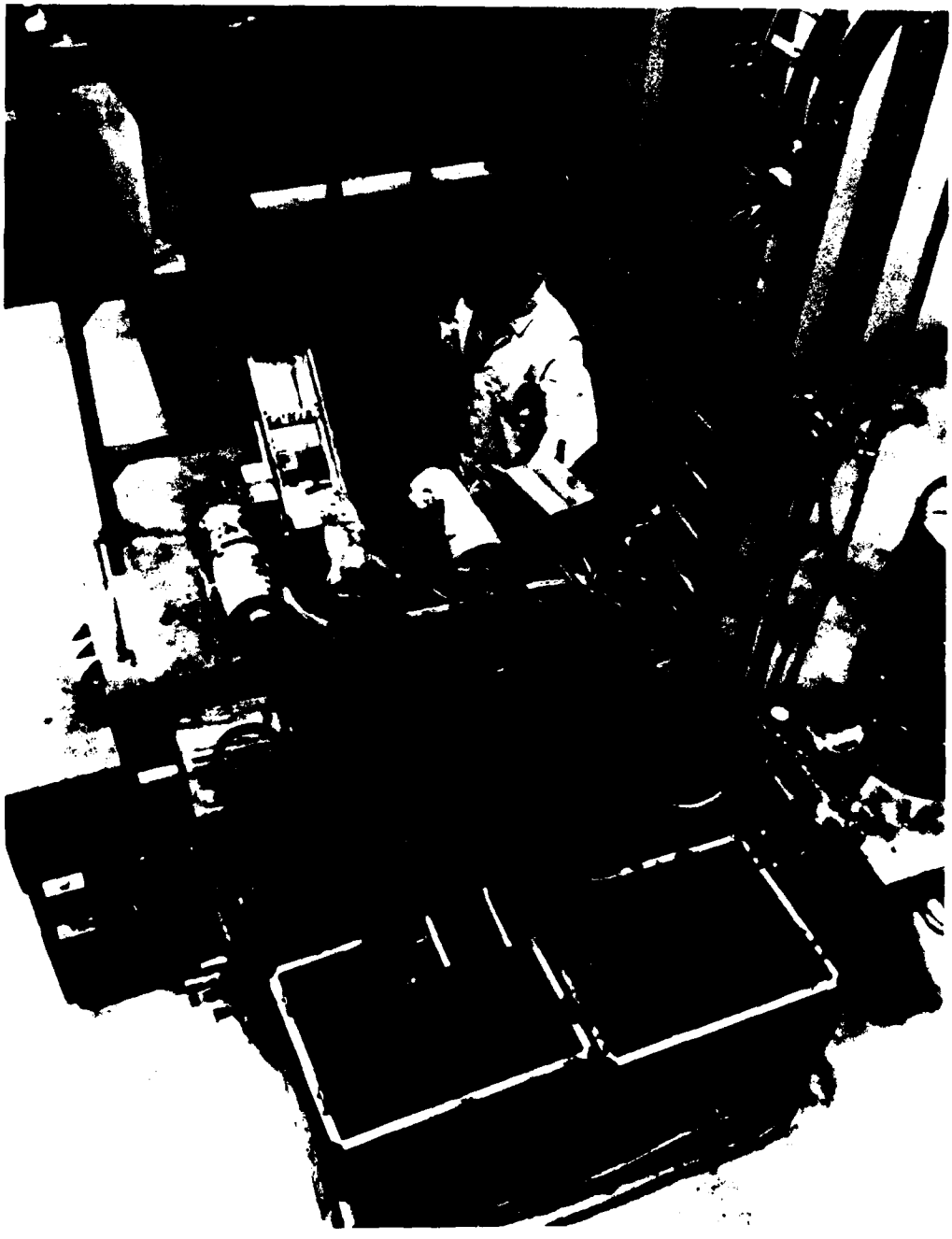

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Figure 6. AVCO Ferrofluid Nonferrous Scrap Separator

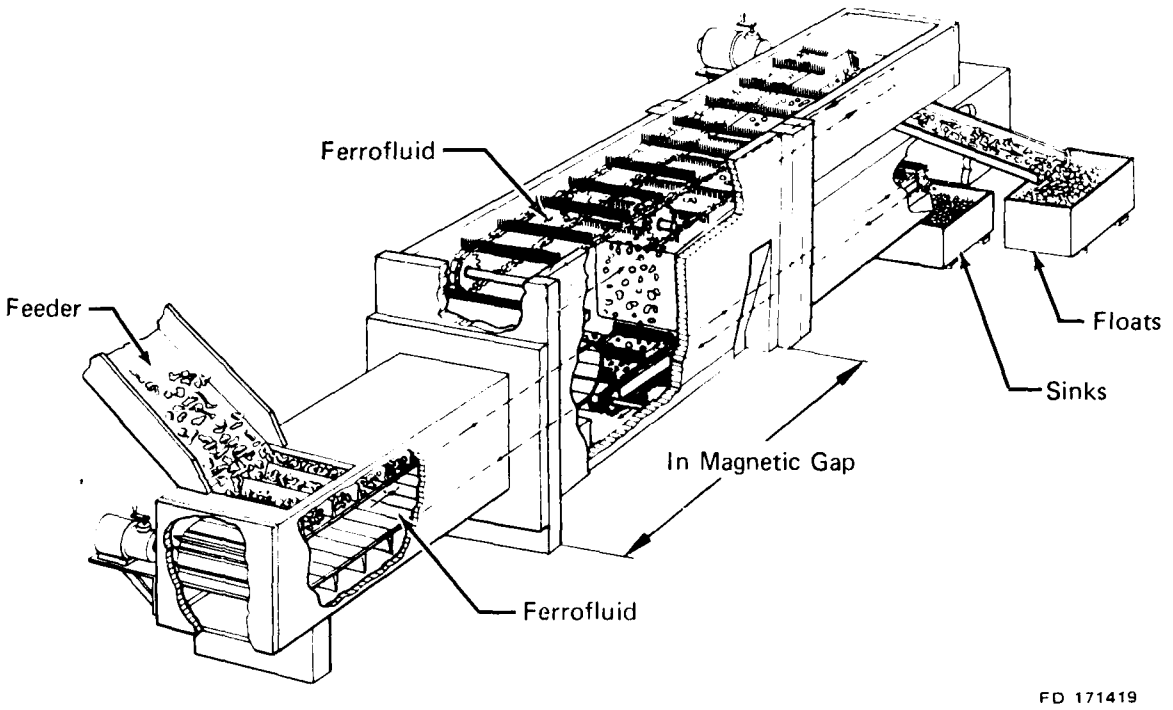


Figure 7. AVCO Ferrofluid Separation Process Material Handling System

(4) Ferrofluid Recovery

The recovery of ferrofluid that coats the metal pieces leaving the separator is accomplished in two steps: washing the solids with a suitable solvent that is miscible with the ferrofluid, and then boiling away the solvent to recover the ferrofluid. Volatile hydrocarbons and chlorocarbons have been found to be effective washing liquids. Because of the danger of flammability, hydrocarbons such as heptane have only been used in the laboratory. On a pilot-plant scale, trichloroethane is used because it presents no fire hazard, and has proven to be an effective wash liquid.

In the scrap separation pilot plant, ferrofluid is removed from the scrap leaving the separator by trichloroethane in a batch cascade degreaser. The degreaser consists of three chambers. Clean solvent is introduced in the third chamber. This chamber overflows into a second chamber which in turn overflows into the first chamber, with the overflow of this chamber going to a storage tank. The first two chambers are heated to boiling while the third chamber is operated cold. A basket that contains approximately one cubic foot of scrap is dipped sequentially in each of the three pools and placed in the vapor space over the first pool. In this manner, the scrap receives a four-stage wash, the fourth wash being obtained by condensing vapors on the cold scrap leaving the third chamber. The hot scrap is then air dried to remove residual solvent. The ferrofluid-solvent mixture leaving the degreaser accumulates in a storage tank until approximately 200 gal are obtained, and is recycled.

The washing solvent is pumped from storage into the cold tank of the degreaser. Ferrofluid-laden solvent is withdrawn from the boiling tank and sent to a holding tank. When a sufficient quantity (150 gal) of ferrofluid-solvent mixture is obtained, it is pumped to a batch still. Heat is applied to the still to attain a liquid temperature of 250°F, assuring volatilization of the solvent.

The liquid is then allowed to cool, withdrawn from the bottom of the still, and returned to ferrofluid storage. In the event that a minimum liquid level is attained prior to achieving the 250°F temperature, the product remaining in the still is mixed with the next lot of diluted ferrofluid. By operating in this manner, it is possible to obtain a ferrofluid of constant composition and properties.

c. *Program Plan Details (Phase I)*

(1) Titanium Program

The principal contaminants precluding the use of titanium scrap in critical applications are tungsten carbide tool fragments. A secondary, although still significant, class of contaminants are nonmagnetic or weakly-magnetic steels and nickel-base alloys. Aluminum is the only important metal less dense than titanium that frequently occurs as a contaminant. This is not a serious problem because the principal titanium alloys contain significant quantities of aluminum. Therefore, aluminum contamination, unless very large, can be corrected by dilution with titanium sponge and other alloying ingredients. The emphasis in this program was accordingly on the removal of the tungsten carbide, superalloy, stainless steel, and other contaminants more dense than titanium itself.

In order to develop and convincingly demonstrate the purification of titanium turnings, it was necessary to consider the variation in scrap characteristics likely to be encountered in practice, and to demonstrate the flexibility of the purification system to handle these scrap types.

From the viewpoint of sink-float separation, the most important variable scrap characteristics are tabulated below:

Characteristics	*Degree of Control in Scrap Pretreatment*
Size Range	Fair
Bulk Density	Poor
Oil and Water Content	Good
Contaminant Content	Poor

Prior to purification by sink-float separation, turnings must be crushed in a hammer-mill, cleaned, and dried. The size range desired is from 3 in. to about 1/8 in. These specifications are typically met in the usual chips available from scrap dealers. Very small chips can accumulate in the separator and harm purification efficiency thus reducing processing rates; it is therefore desirable that the lower limit be as large as possible, as long as this does not result in the rejection by screening of too large a proportion of the turnings. The proper balance between removal of small turnings and product loss is therefore an important decision.

For a fixed size range, the bulk density of turnings depends strongly on their thickness. This variable is controlled totally by the machining process, and crushed turnings may range in bulk density from about 10 to 40 lb/ft^3. It is expected that high bulk density chips should be processable at a higher rate than low bulk density chips. Accordingly, it was necessary to study the purification of various types.

As noted previously, typical chips available from scrap dealers are oil- and water-free. This would be a specification placed on chips to be purified by sink-float separation. The presence of oil and water in turnings can have two types of harmful effects on the sink-float process, one potentially much more serious than the other. The less serious effect is the dilution of the ferrofluid by the oil (water not being soluble in ferrofluid would be floated to the surface and have no harmful effect). During the actual purification run, this dilution effect would be compensated

for automatically by the ferrofluid density control system. Should the dilution be so severe that compensation becomes impractical, the density of the ferrofluid would fall, and some titanium would start to sink rather than float. This is a fail-safe situation for titanium since it does not lead to contamination of the purified titanium. If the presence of oil in the turnings is large and persistent, it would lead to serious dilution of the ferrofluid supply and require make-up by concentrated ferrofluid and intentional purge of some of the diluted ferrofluid. Both of these contingencies, loss of titanium and loss of ferrofluid, although economically harmful, do not threaten the quality of the purification and thus the basic integrity of the process.

The more important harmful effect of the presence of oil and water in turnings is to make them "sticky," particularly if the oil and water are emulsified. The stickiness may result in the adhesion of small dense impurities to large titanium turnings. The resulting composite may be close enough to the density of titanium to float in the ferrofluid and thus to contaminate the product. Because of the potential seriousness of this effect, the purification of scrap contaminated by large quantities of oil and water was examined in the course of this program.

The most harmful class of contaminants requiring removal from titanium turnings are, of course, tungsten carbide tool fragments. Because of the potential seriousness of dense inclusions in critical aircraft parts, the removal of these contaminants, above a certain size, must be complete. As a working goal, this size was 1/64 in. When fragments of this size are removed completely, it can be assumed that considerably smaller ones will also be removed with high efficiency.

Because minor contamination by nonmagnetic or weakly magnetic steel is a less drastic problem than contamination by tungsten carbide fragments, the removal of the former class of contaminants need not be complete. A residual level of 0.1% or so is tolerable.

The contamination level in the sample of titanium turnings to be purified in certain experiments was artificially raised by the addition of tool bits, stainless steel, and superalloy turnings. Used tools were crushed to form chip fragments ranging in size from about 5 mesh (0.157 in.) to less than 80 mesh (0.007 in.). The stainless steel and the superalloy turnings had about the same size range as the titanium turnings themselves. The lower limit of the size range of the tool chips was chosen to provide the required severe test of the separation process, and the upper limit to provide information on the effect of size on separation efficiency. The size range of the turnings was typical of the size range of superalloy and stainless steel scrap that could contaminate the titanium turnings. The level of contamination for tool chips was 0.3%, a level higher than is likely to be encountered. The level of stainless steel and superalloy contamination was 5%, a high, although reasonably typical level for heavily contaminated scrap. No variation in these contamination levels was planned, because the removal efficiency of contaminants by sink-float separation, in this low concentration range, was not a function of their concentration. Consequently, the removal efficiencies found at these levels was confidently used at other contamination levels in the low range to predict the residual contamination after purification.

Based on the above considerations, a matrix of scrap-related variables, as listed below, was explored in Phase I of the program.

	Level	
Variable	*Low*	*High*
Smallest Size of Turning	As-Received	+5 Mesh
Bulk Density	10 — 20 lb/ft^3	30 — 40 lb/ft^3
Oil Content	Negligible	5%
Water Content	Negligible	5%
Tool Chip Content	As-Received	0.3% Added
Stainless Steel and Superalloy Content	As-Received	5.0% Added

The choice of operating variables presents less difficulty than the choice of scrap-related variables. The two important operating variables are the apparent density of the ferrofluid pool and the processing rate. An apparent density only slightly greater than the density of titanium, i.e., 4.5 gm/cm^3, and a low processing rate are both conducive to high purification efficiencies. Too low a density may, however, result in an unacceptable loss of titanium to the sinks fraction and too low a processing rate has obvious penalties. Based on practical conditions tentatively identified in experiments conducted prior to the initiation of this program, an apparent density range of 4.7 to 5.3 gm/cm^3 and a processing rate of 400 to 700 lb/hr was studied.

The experiments were carried out in groups of three, as shown in Figure 8. In the former sequence, a sample of the feed turnings was X-rayed to determine the normal contamination content of tungsten-carbide tool chips. After separation, samples of the sinks and floats were X-rayed again to measure the efficiency of the separation process. The other two-thirds of the sample were contaminated with tool bits and superalloy turnings, and a sample X-rayed. One-half of this lot was purified once and samples of the products X-rayed; the other half was purified twice prior to X-raying the products. Separated fractions were submitted to P&WA for chemical analysis of selected samples. Selected samples were melted into ingots, converted to barstock, and subjected to chemical, NDI, microstructural and mechanical properties determination.

This experimental sequence provided a great deal of important data about the efficiency of the process. The sample with the normal contamination level provided data on the concentration of contaminants naturally occurring in titanium turnings, as well as the efficiency of the separation process. The sample with a large, controlled impurity content provided more accurate information on the efficiency of the separation process. The third sample was purified twice; the floats from the first purification were repurified (without ferrofluid removed between the two purifications). This provided data on the value of dual purification as a means of attaining extremely high purities. On a commercial scale, this two-step purification process option can be carried out at very little increase in cost relative to a one-step purification.

Throughout this portion of the program, a careful record was maintained of ferrofluid properties as well as the apparent density of the ferrofluid in the separator during the separations; however, it was not possible to determine accurately any tendency for ferrofluid degradation in this phase of the work because the quantity of titanium to be processed was limited. Any long-term tendency for degradation was indicated in the Phase II runs.

Table 7 indicates the principal experiments that were carried out to explore the effects of the major variables previously identified. The scheduled experiments were carried out in the range of operating parameters that appeared favorable based on work that AVCO had already carried out. Based on an analysis of the results of these experiments, limited additional runs were made at optimum process conditions.

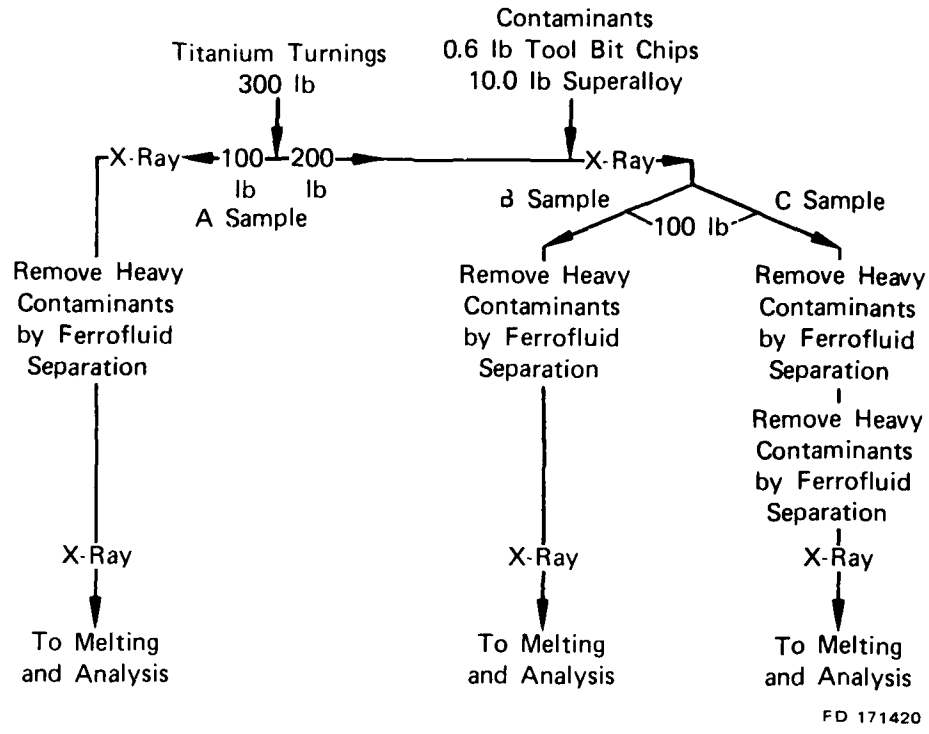


Figure 8. Basic Proposed Test Sequence — Titanium Scrap Separation — AVCO

TABLE 7
AVCO FERROFLUID SEPARATION SYSTEM PROPOSED TEST VARIABLES

TITANIUM PURIFICATION — SERIES A

OBJECTIVE

Determine effects of apparent density and processing rate on separation efficiency.

SCRAP PROPERTIES

1. Alloy — Ti-6Al-4V
2. Bulk Density — 25 to 30 lb/ft³
3. Oil and Water — Negligible
4. Crushed Scrap Screened to Remove — -1/8 in. Material

EXPERIMENTS

Apparent Density of Ferrofluid (g/cm³)	Processing Rate (lb/hr) 400	700	1000
4.7	x(a)(b)	x(b)	?(b)
5.0	x(a)(b)	x(b)	?(b)
5.3	x(b)	x(b)	?(b)

x — Scheduled Experiments
? — Tentative Experiments
a — Separation of Uncontaminated Sample i.e., "blank"
b — Separation of Contaminated Sample

SCRAP REQUIREMENTS

Titanium	2,400 lb
Waspaloy	40 lb
Stainless Steel	40 lb

TITANIUM PURIFICATION — SERIES B

OBJECTIVE

Determine effect of not removing 1/8 in. material prior to separation.

SCRAP PROPERTIES

1. Alloy — Ti-6Al-4V
2. Bulk Density — 25 to 30 lb/ft³
3. Oil and Water — Negligible
4. Crushed Scrap Not Screened to Remove — -1/8 in. Material

EXPERIMENTS

Apparent Density of Ferrofluid (g/cm³)	Processing Rate (lb/hr) 400	700	1000
4.7	x(b)	x(b)	
5.0	x(b)	x(b)	?(b)
5.3			

x — Scheduled Experiments
? — Tentative Experiments
a — Separation of Uncontaminated Sample
b — Separation of Contaminated Sample

SCRAP REQUIREMENTS

Titanium	1,000 lb
Waspaloy	25 lb
Stainless Steel	25 lb

TABLE 7
AVCO FERROFLUID SEPARATION SYSTEM PROPOSED TEST VARIABLES (CONTINUED)

TITANIUM PURIFICATION — SERIES C

OBJECTIVE

Determine effect of high bulk density Ti on separation efficiency.

SCRAP PROPERTIES

1. Alloy — Ti-6Al-4V
2. Bulk Density — 35 to 40 lb/ft³
3. Oil and Water — Negligible
4. Crushed Scrap Screened to Remove — ¼ in. Material

EXPERIMENTS

Apparent Density of Ferrofluid (g/cm³)	Processing Rate (lb/hr) 400	700	1000
4.7	x(a)(b)	?(b)	
5.0	?(b)	x(a)(b)	
5.3	x(b)	?(b)	

x — Scheduled Experiments
? — Tentative Experiments
a — Separation of Uncontaminated Sample
b — Separation of Contaminated Sample

SCRAP REQUIREMENTS

Titanium	1,200 lb
Waspaloy	25 lb
Stainless Steel	25 lb

TITANIUM PURIFICATION — SERIES D

OBJECTIVE

Determine effect of low bulk density Ti on separation efficiency.

SCRAP PROPERTIES

1. Alloy — Ti-6Al-4V
2. Bulk Density — 10 to 20 lb/ft³
3. Oil and Water — Negligible
4. Crushed Scrap Screened to Remove — ¼ in. Material

EXPERIMENTS

Apparent Density of Ferrofluid (g/cm³)	Processing Rate (lb/hr) 200	400	700
4.7	x(a)(b)	?(b)	
5.0	?(b)	x(b)	
5.3	x(b)	?(b)	

x — Scheduled Experiments
? — Tentative Experiments
a — Separation of Uncontaminated Sample
b — Separation of Contaminated Sample

SCRAP REQUIREMENTS

Titanium	1,000 lb
Waspaloy	25 lb
Stainless Steel	25 lb

TITANIUM PURIFICATION — SERIES E

OBJECTIVE

Determine effect of oil and water presence on separation efficiency.

SCRAP PROPERTIES

1. Alloy — Ti-6Al-4V
2. Bulk Density — 25 to 30 lb/ft³
3. Scrap Not to Be Cleaned or Screened After Crushing

EXPERIMENTAL CONDITIONS

Best Two Conditions as Determined from Series A

SCRAP REQUIREMENTS

Titanium	500 lb
Waspaloy	10 lb
Stainless Steel	10 lb

(2) Nickel Alloy Program

The recycling of superalloy scrap presents a more complex problem of purification than titanium. Contaminating elements, both less and more dense than the superalloy, have several origins, ranging from simple contamination by other scrap metals such as titanium, to adhering potting compounds containing bismuth. Tungsten carbide bits are not a significant problem because the long holding times in superalloy melting furnaces ensure the dissolution of these small fragments. However, interalloy contamination of superalloy turnings is a serious problem because of the large number of superalloys in common use. In this program, the major emphasis was placed on the removal of titanium contaminants from superalloy scrap. Emphasis was also placed on the removal of lead, which is a very harmful contaminant and typical of the contaminants denser than the superalloys.

The separation of superalloys from each other appears to be an exceedingly difficult problem because of the large number of alloys in use and their very similar densities. Therefore, in addition to the basic purification work, an exploratory study was conducted on the feasibility of employing the weak magnetic properties of superalloys as a basis for separating four superalloys from each other. The light impurities were Ti-5Al-2.5Sn turnings and the dense-lead shot.

As in the titanium work, a three-experiment sequence was used. The three-experiment sequence is shown in Figure 9. The reasons for the use of this sequence were the same as those given for titanium. The first sample provides data on the naturally occurring contaminants, while the second and third samples provided accurate data on purification efficiency for the one-step and two-step purification process.

Analyses included bulk density measurements on the various contaminant fractions, whether sinks or floats, as a means of obtaining a semiquantitative measure of contaminant content and Waspaloy loss. An X-ray was not planned because this method would only yield reliable information on lead content, and it was more economical to obtain the lead content as part of the elemental analysis of the melted samples.

The same ferrofluid properties were measured as in the titanium portion of the program.

The operating conditions under which the separation was carried out are listed in Table 8. These conditions were based on preliminary tests on Waspaloy purification previously carried out by AVCO.

As has been pointed out in previous sections, the physical density differences among most nickel-base superalloys are too small to serve as a basis for their reliable separation by sink-float separation. The differences in the weak magnetic properties of superalloys may, however, augment these density differences sufficiently for the separation of many alloys to become feasible. This is due to the fact that the apparent density of a magnetizeable object in a ferrofluid separator is larger than the object's physical density. A small exploratory program was therefore proposed to investigate the feasibility of this approach. The alloys chosen for this study and the relevant physical properties of these alloys are listed in Table 9.

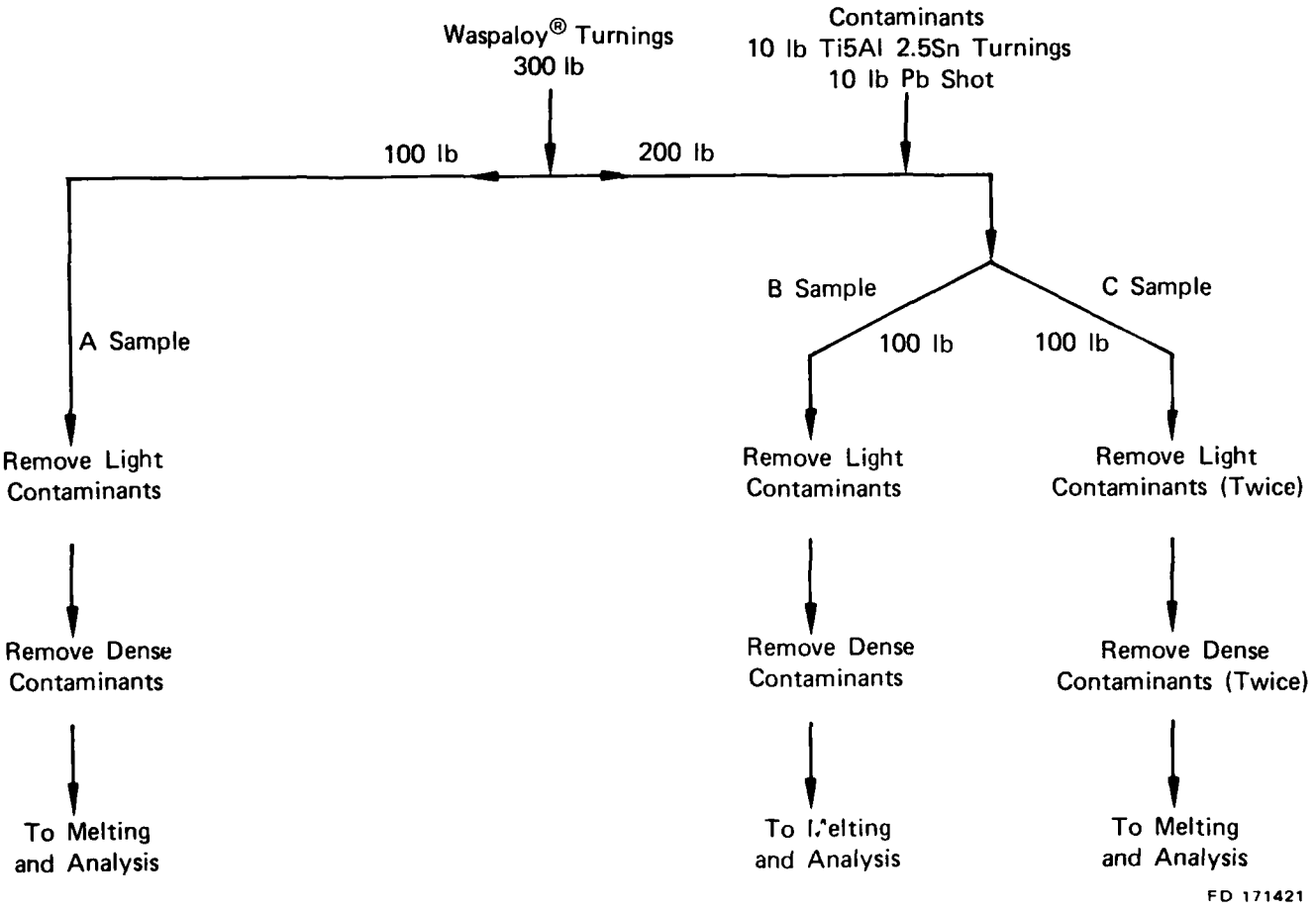


Figure 9. Basic Proposed Experimental Test Sequence — Waspaloy Scrap Purification — AVCO

TABLE 8
AVCO FERROFLUID SEPARATION SYSTEM
PROPOSED TEST VARIABLES WASPALOY
PURIFICATION EXPERIMENTS

OBJECTIVE

Determine effects of apparent density and processing rate on separation efficiency.

SCRAP PROPERTIES

1. Crushed, degreased and dry turnings.
2. Size Range 3 to [illegible] in

EXPERIMENTS

Apparent Density of Ferrofluid

Removal of Light Contaminants	Removal of Dense Contaminants	Processing Rate (lb/hr) 600	900	1200
8.0	8.5	x(a)(b)	x(b)	?(b)
7.7	8.5	x(b)	x(b)	
7.7	8.8			?(b)

x Scheduled Experiments
? Tentative Experiments
a Separation of Uncontaminated Sample
b Separation of Contaminated Sample

TABLE 9
PHYSICAL PROPERTIES OF
SELECTED SUPERALLOYS*

Alloy	*Density (gm/cm^3)*	*Magnetic Properties (Permeability - μ)*
A286	*7.92*	*1.01 to 1.05 at 200 oersted*
IN901	*8.23*	*1.008 at 200 oersted*
IN718	*8.21*	*1.001 at 200 oersted*
Waspaloy	*8.20*	*1.012 at 200 oersted*

* *Taken from Aerospace Structural Metals Handbook, Volume 4, AFML-TR-68-115 1972 Publication*

d. Separation Test Program (Titanium)

(1) Test Procedure

With few exceptions, a common procedure was used during all the AVCO experimental test runs. For blank (uncontaminated) runs, chips were dumped directly from the shipping drums onto a conveyor going to the feed hopper for the ferrofluid separator. For runs carried out with a salted (contaminated) feed, the contaminants (stainless steel, Waspaloy and tungsten carbide tool bit fragments) were added to the conveyor at the same time as the chips. This was expected to result in a reasonably uniform feed mixture to the ferrofluid separator.

The feed rate to the ferrofluid separator was controlled by the output of a vibratory feeder at the bottom of the feed hopper. At the start of each series, the output of the vibratory feeder was calibrated with the type of scrap to be processed by measuring the weight of the scrap collected in a barrel within a given period of time at different settings of the vibratory feeder. At any given setting, the reproducibility of the output (feed rate) of the vibratory feeder varied by ± 10%.

The scrap leaving the vibratory feeder fell into the inclined chute leading to the ferrofluid density separation vessel, and was then forced to the side of the ferrofluid pool by a rotating paddle wheel. The three rubber blades of the rotor, turning at 100 rpm, distributed the scrap in the pool as quickly as it came down the chute, thereby preventing a mechanical jam and possible interruption of separator operation.

Low density fractions of the feed were skimmed from the top ferrofluid pool, swept into a chute, and then collected in a screen-lined basket. The high density fraction of the feed was removed from the bottom of the ferrofluid by a moving rubber belt conveyor (linear velocity from 35 to 70 ft/min) and dumped into a second basket alongside the magnet. This was possible because the ferrofluid in the gap is retained by the magnetic field. Separate baskets were used to collect sinks and floats to eliminate cross contamination from run to run.

AVCO Ferrofluid No. 1224 was used as the working medium for the titanium separation tests. The magnetization curve for this kerosene-based ferrofluid is presented in Figure 10. It had a nominal magnetization of 290 gauss at 3500 oe. The magnetization curve was obtained on a daily basis during operation as a quality control measure. There was less than a 3% variation in the measured magnetization of the ferrofluid. This ferrofluid has a true density of 1.160 gm/cm^3 and a viscosity of 3.5 cp at 30°C.

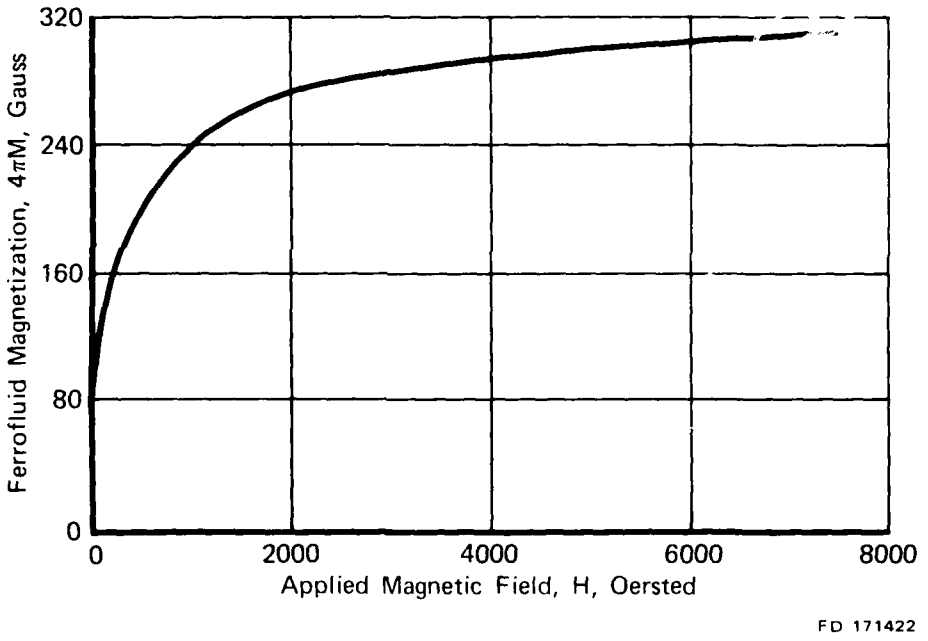


Figure 10. Magnetization Curve of AVCO Ferrofluid 1224 Used in Titanium Purification Tests

The apparent density of the ferrofluid pool was adjusted by controlling the current to the electromagnet. In these tests, the DC current ranged from 300 to 420 amps corresponding to a variation of 150 to 200 oe/cm to the vertical component of the magnetic field gradient.

Operating density was established by varying the current to the electromagnet until the desired reading on the density control elements was attained. This density controller measured the pressure of the ferrofluid against a sensor at the bottom of the pool. Since the liquid level in

the pool was kept constant, this pressure measurement of the equivalent head of ferrofluid was proportional to the apparent density of the ferrofluid. A calibration curve (Figure 11) was obtained by establishing the pressure level at which reference objects of known density were neutrally buoyant in the ferrofluid. This curve is independent of the properties of the ferrofluid outside the magnetic field. An operating density, controllable to 0.5 in. H_2O (equivalent to a density variation of 0.05 gm/cm^3), can be maintained even if the properties of the ferrofluid vary slightly.

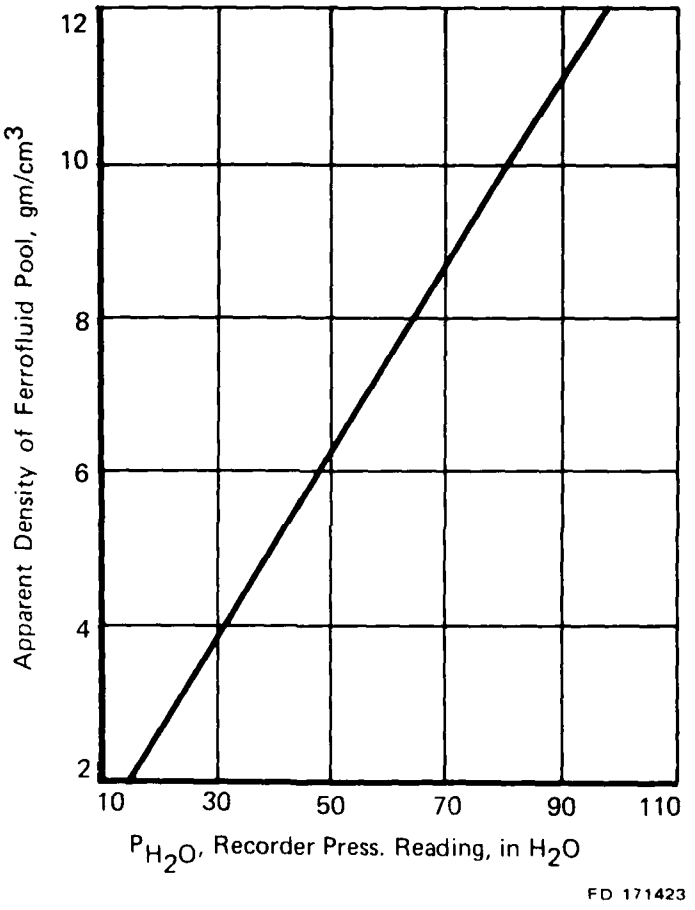


Figure 11. Correlation Between Pressure Readout and Apparent Ferrofluid Density for AVCO Pilot Separator

The separated fractions leaving the separator were collected in open-mesh baskets lined with 50-mesh screening. The baskets were then countercurrently washed with trichloroethane in a batch degreaser. After cleaning, the fractions were dumped in plastic-lined, tared containers, i.e., either pails or drums depending on the amount of material and weight. When there was sufficient material to use a drum, a fraction of the contents of each basket was placed in a tared pail to provide a representative sample for further analysis. The baskets were then cleaned for reuse in a subsequent run.

(2) Test Results (Titanium)

AVCO completed a series of experimental tests with titanium chip samples to establish the effects of system variables on the efficiency of the ferrofluid separation process. The original test plans and the test sequence were presented in Table 7 and Figure 8, respectively. The actual test program, summarized in Table 10, conformed to the previous plans, with minor deviations in Series A through D. Series E, originally intended to test the effects of a high oil and water content, was cancelled for two reasons: (a) AVCO judged the ferrofluid system to be tolerant of high levels of oil and water content; and (b) adequate, efficient chip processing procedures have been established to control oil and water content at acceptable levels. Table 11 consolidates all data obtained during the AVCO Test Series A-D.

(a) Discussion of Results

The data obtained on the distribution of feed material into sinks and float fractions provides a significant indication of the efficiency of the ferrofluid density separation; however, a comprehensive evaluation requires analytical data on the composition of the separated fractions, because the sinks will inevitably contain clean chips and the floats may contain contaminants, dependent on process conditions.

The effect of separation conditions on the samples of Lot 01 (Table 3) are summarized in Figures 12 through 14. In each figure, the weight-percent sinks are plotted as a function of feed rate of turnings at a given operating density. As shown in these figures, the weight-fraction sinks for the first pass are generally higher than the weight-fraction sinks obtained when the floats are reprocessed. In all cases, the weight-fraction sinks for the first pass are higher than the level of contaminants added. This suggests that the bulk of the contaminants may be removed in the first pass. The fact that the blank sample and Pass II sinks are at similar levels also tends to support the hypothesis. This was substantiated by subsequent X-ray analysis of the samples.

Figure 12 shows that at an operating density of 5.3 gm/cm^3, there was little effect of feed rate over the range of 350 to 800 lb/hr on the weight-fraction sinks; however, increasing the feed rate of chips to ~ 1200 lb/hr resulted in a significant increase in weight-percent sinks (more than 20% of the total), both for Pass I and Pass II, thereby indicating a significant amount of noncontaminated titanium in the sinks.

In general, it appears that at lower operating densities (5.0 gm/cm^3 and 4.7 gm/cm^3) which approach the density of titanium, there is a noticeable effect of feed rate on the weight-percent sinks (Figures 13 and 14). As the feed increases, the weight-percent sinks increase, indicating misclassified titanium. The sharp rise in the sinks level can be explained in terms of particle interaction. With a small driving force tending to float the titanium, which results in a long residence time, and at high volumetric feed rate, the concentration of particles in the ferrofluid pool in the separator increases to the point that the particles interact which leads to hindered settling. In those runs where 20% sinks were observed, it was calculated that the volumetric titanium particle concentration was approximately 0.25%. In arriving at this estimate, it was assumed that the chips were inertially controlled and were only present in the upper half of the working volume of the separator. The effective concentration of the chips is much higher than this value because the hydrodynamic interaction volume of a flat chip is a function of the cube of its largest dimension. Since a typical chip appears to be approximately 1/2-in. by 1/2-in. by 1/32-in. thick, the hydrodynamic volume of a chip is about 16 times larger than its actual volume, resulting in an effective chip concentration of 4% by volume in these cases. Since there is less than one chip length separating any two chips in the fluid, hindered settling can occur.

TABLE 10
AVCO FERROFLUID SEPARATION OF TITANIUM ALLOY CHIPS — TEST DATA

Apparent Ferrofluid Density (g/cm³)		*Series A Lot 01 Medium Density Screened*	*Series B Lot 03 Medium Density No Screening of Fines*	*Series C Lot 05 High Density Screened*	*Series D Lot 04 Low Density Screened*
			200 lb/hr Nominal Processing Rate		
4.7		–	–	–	
5.0		–	–	–	C130*
					CR202
5.3		–	–	–	
			400 lb/hr Nominal Processing Rate		
4.7	**	B400	–	–	B384
		C439	C410	–	C345
		CR400	CR492	–	CR395
5.0		B425	–	–	
		B455	–	–	
		C410	C440	C430	C365
		CR446	CR385	CR620	CR390
5.3		B494	–		
		C365	C475		C400
		CR410	CR612	–	CR365
			700 lb/hr Nominal Processing Rate		
4.7			–	B725	
		C735	C550	C715	
		CR550	CR650	CR785	
5.0			–	B660	
		C659	C760	C715	
		CR600	CR695	CR930	
5.3		–	–	–	
		$C_1$530	–	C955	C695
		C_{1R}585		CR935	CR665
		$C_2$785	–	–	
		C_{2R}800			
			1000 lb/hr Nominal Processing Rate		
4.7		–	–	–	
5.0		C890	–	–	
		CR795	–	–	
5.3		C1200	–	C1090	
		CR1140		CR1100	

*Actual Processing Rate

**B — Blank (Noncontaminated) 100 lb run
C — Contaminated (artificially) 200 lb run
CR — Contaminated (artificially) Rerun 100 lb floats from C
C_1,C_{1R},C_2,C_{2R} — Duplicate runs

TABLE 11
AVCO EXPERIMENTAL DENSITY SEPARATION TEST DATA TITANIUM SERIES A-D

Feed Material	*Run No.*	*Contaminants*	*Feed Rate (lb/hr)*	*Operating Pressure (in. H_2O)*	*Operating Density (gm/cm^3)*	*Weight Total Product Stream (lb)*	*Weight Floats (lb)*	*Weight Sinks (lb)*	*Weight Percent Floats*	*Weight Percent Sinks*
Series A										
Lot 01	2	Blank	400	37[1]	4.7	109.0	83.0	26.0	76.1	23.9
Medium Density	1	Blank	455	40	5.0	129.6	116.1	13.5	89.6	10.4
Ti-6Al-4V Screened	53	Blank	425	40	5.0	95.9	94.7	1.2	98.7	1.3
	52	Blank	494	42	5.3	115.2	113.7	1.5	98.7	1.3
	9	Contaminated	439	37[1]	4.7	252.6	228.0	24.6	90.3	9.7
	10	Contaminated	735	37[1]	4.7	234.0	184.0	50.0	78.6	21.4
	6	Contaminated	410	40	5.0	241.6	227.0	14.6	94.0	6.0
	7	Contaminated	659	40	5.0	211.8	194.0	17.8	91.6	8.4
	8	Contaminated	890	40	5.0	217.0	163.0	54.0	75.1	24.9
	3	Contaminated	365	42	5.3	208	197.0	11.0	94.7	5.3
	4	Contaminated	530	42	5.3	169.6	160.0	9.6	94.3	5.7
	5	Contaminated	785	42	5.3	180	170.0	10.0	94.4	5.6
	54	Contaminated	1200	42	5.3	235.9	174.9	61.0	74.1	25.9
Half of Run 9 Floats	17	Contaminated	400	37	4.7	101.3	95.6	5.7	94.4	5.6
Half of Run 10 Floats	18	Contaminated	550	37	4.7	89.4	85.7	3.7	95.8	4.2
Half of Run 6 Floats	14	Contaminated	446	40	5.0	113.4	110.7	2.7	97.6	2.4
Half of Run 7 Floats	15	Contaminated	600	40	5.0	82.2	80.0	2.2	97.4	2.6
Half of Run 8 Floats	16	Contaminated	785	40	5.0	66.9	55.5	11.4	83.0	17.0
Half of Run 3 Floats	11	Contaminated	410	42	5.3	91.5	89.6	1.9	97.9	2.1
Half of Run 4 Floats	12	Contaminated	585	42	5.3	83.8	82.6	1.2	98.6	1.4
Half of Run 5 Floats	13	Contaminated	800	42	5.3	76.6	75.7	0.9	98.8	1.2
Half of Run 54 Floats	55	Contaminated	1140	42	5.3	80.3	65.1	15.2	81.1	18.9
Series B	20	Contaminated	410	37	4.7	235.5	213.3	22.2	90.5	9.5
Lot 03	21	Contaminated	550	37	4.7	171.2	123.5	47.7	72.2	27.8
Medium Density	23	Contaminated	440	40	5.0	179.9	169.2	10.7	94.0	6.0
Ti-6Al-4V	22	Contaminated	760	40	5.0	248.0	197.0	51.0	79.4	20.6
Not Screened	19	Contaminated	475	42	5.3	273.7	252.0	21.7	92.1	7.9
Half of Run 20 Floats	25	Contaminated	492	37	4.7	89.4	82.0	7.4	92.2	7.8
Half of Run 21 Floats	26	Contaminated	650	37	4.7	48.7	37.6	11.1	77.2	22.8
Half of Run 23 Floats	28	Contaminated	885	40	5.0	59.7	58.7	1.0	98.3	1.7
Half of Run 22 Floats	27	Contaminated	695	40	5.0	76.4	73.4	3.0	96.1	3.9
Half of Run 19 Floats	24	Contaminated	615	42	5.3	108.8	106.2	2.6	97.6	2.4
Series C										
Lot 05	30	Blank	725	37	4.7	98.0	94.8	3.2	97.0	3.0
High Density	29	Blank	600	40	5.0	54.3	52.2	1.0	98.1	1.9
Ti-6Al-4V										
	34	Contaminated	715	37	4.7	214.4	194.0	20.4	90.5	9.5
	35	Contaminated	430	40	5.0	122.0	109.2	12.8	89.6	10.4
	33	Contaminated	745	40	5.0	214.9	198.7	16.2	92.5	7.5
	31	Contaminated	955	42	5.3	99.0	95.0	4.0	95.9	4.1
	32	Contaminated	1090	42	5.3	214.4	214.0	10.4	95.4	4.6
Half Run of 34 Floats	39	Contaminated	785	37	4.7	72.6	66.4	6.2	91.5	8.5
Half Run of 35 Floats	40	Contaminated	620	40	5.0	44.4	42.8	1.6	96.4	3.6
Half of Run 33 Floats	38	Contaminated	690	40	5.0	78.1	76.5	1.6	98.0	2.0
Half of Run 31 Floats	36	Contaminated	935	42	5.3	120.6	119.2	1.4	98.8	1.2
Half of Run 32 Floats	37	Contaminated	1300	42	5.3	83.8	82.5	1.3	98.4	1.6
Series D										
Lot 04	41	Blank	384	37	4.7	95.8	90.4	5.4	93.4	6.6
Low Density										
Ti-6Al-4V										

TABLE 11
AVCO EXPERIMENTAL DENSITY SEPARATION TEST DATA TITANIUM SERIES A-D (CONTINUED)

Feed Material	Run No.	Contaminants	Feed Rate (lb/hr)	Operating Pressure (in. H_2O)	Operating Density (gm/cm^3)	Weight Total Product Stream (lb)	Weight Floats (lb)	Weight Sinks (lb)	Weight Percent Floats	Weight Percent Sinks
	46	Contaminated	345	37[1]	4.7	187.9	146.9	41.0	78.2	21.8
	45	Contaminated	130	40	5.0	125.9	114.7	11.2	90.2	9.8
	44	Contaminated	365	40	5.0	198.5	184.5	14.0	92.9	7.1
	42	Contaminated	400	42	5.3	216.7	95.6	21.1	90.7	9.3
	43	Contaminated	695	42	5.3	211.8	132.8	79.0	62.6	37.4
Half of Run 46 Floats	51	Contaminated	395	37[1]	4.7	68.4	49.5	18.9	72.4	27.6
Half of Run 45 Floats	50	Contaminated	202	40	5.0	56.8	53.8	3.0	94.7	5.3
Half of Run 44 Floats	49	Contaminated	390	40	5.0	85.1	79.7	5.4	93.5	6.5
Half of Run 42 Floats	47	Contaminated	365	42	5.3	79.4	76.2	3.2	96.0	4.0
Half of Run 43 Floats	48	Contaminated	665	42	5.3	61.1	41.5	19.6	68.0	32.0

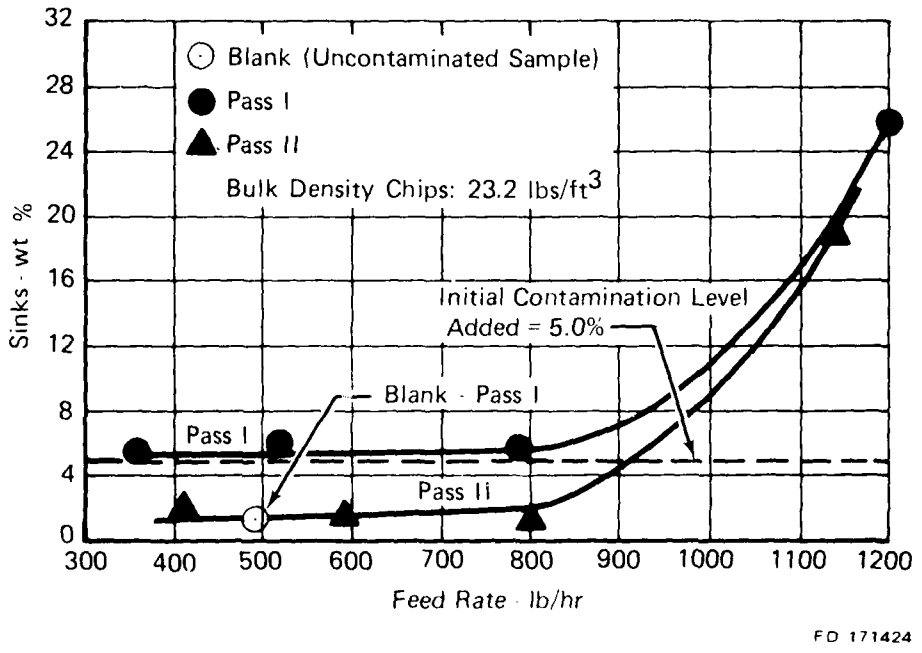


Figure 12. AVCO Ferrofluid Separation Test Series A. Weight-Percent Sinks vs Feed Rate of Contaminated Titanium Chips (Lot 01) at an Operating Density of 5.3 gm/cm^3 (P_{mag} 42 in. H_2O)

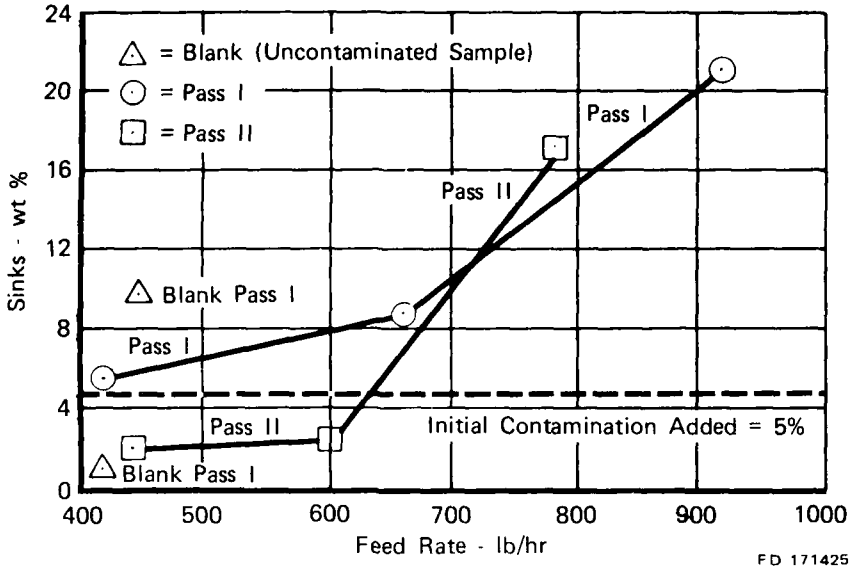


Figure 13. AVCO Ferrofluid Separation Test Series A. Weight-Percent Sinks vs Feed Rate of Contaminated Titanium Chips, Lot 01 of an Operating Density of 5.0 gm/cm³ (P_{mag} = 40-in. H_2O)

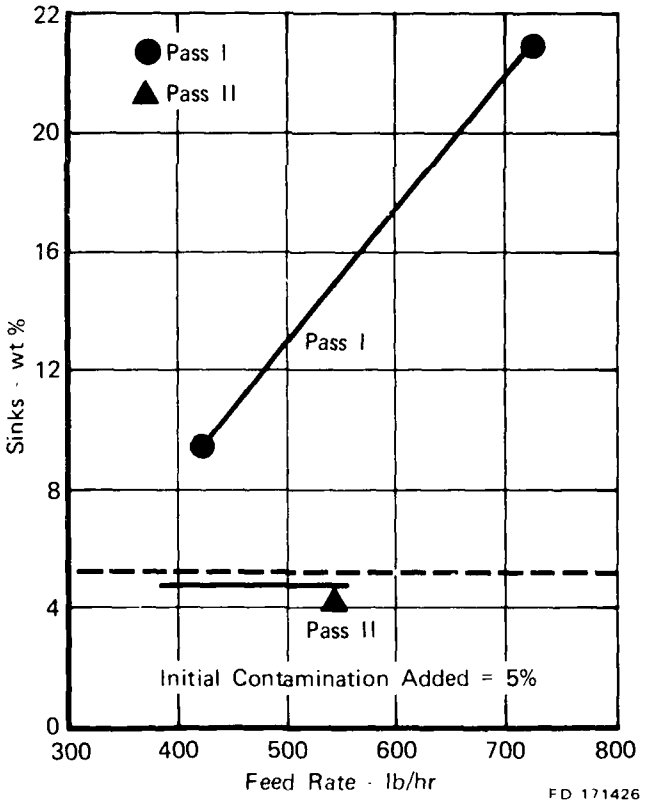


Figure 14. AVCO Ferrofluid Separation Test Series A. Sinks vs Feed Rate of Contaminated Titanium Chips, (Lot 01) at an Operating Density of 4.7 gm/cm³ (P_{mag} = 37 in. H_2O)

The threshold value of feed rate on a weight basis at which misclassification occurs, appears to be a function of the bulk density of the scrap being processed. Likewise, this threshold value decreases as the operating density approaches that of the titanium being processed, i.e., 4.5 gm/cm³. Similarly, for a given operating feed rate, sinks increase as the operating density approaches that of titanium.

Summary results for Lots 04 and 05 are presented in Figures 15 and 16. The results for Lot 03, which is similar to Lot 01 except that it was not screened to remove fines, are similar to those obtained for Lot 01. The chances of misclassification were anticipated to be higher without removal of the fines. X-ray results should establish the merits of removing fines. These results appear to be consistent with a model which assumes that there is little misclassification until the concentration of particles in the ferrofluid pool becomes so high as to result in particle interaction and hindered settling. The trajectory of a particle is then no longer a function of its density relative to that of the ferrofluid, but instead a function of the motion of the total particle population. If the feed rate is excessive, it is possible that incoming material misclassifies objects that would normally float or sink, e.g., a relatively heavy contaminant, prone to sink, may be carried up by a high concentration of light chips intent on floating, or light chips forced down by the concentration of incoming chips may be captured by the sinks conveyor.

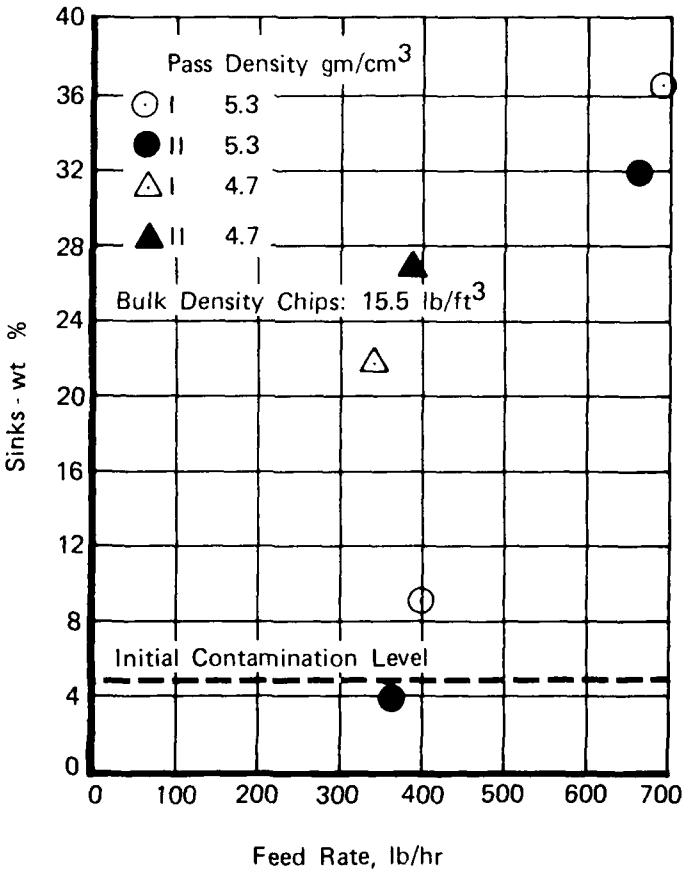


Figure 15. AVCO Ferrofluid Separation Test Series D. Weight-Percent Sinks vs Feed Rate of Contaminated Titanium Chips (Lot 04) at Various Feed Rates and Operating Densities

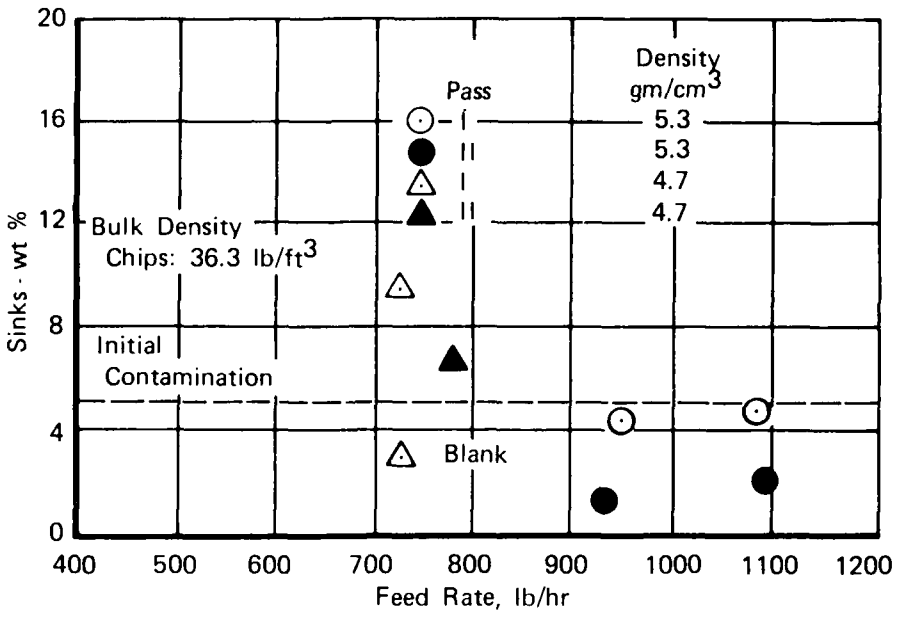


Figure 16. AVCO Ferrofluid Separation Test Series C. Weight-Percent Sinks vs Feed Rate of Contaminated Titanium Chips (Lot 05) at Various Feed Rates and Operating Densities

With proper conditions, where there is no gross interaction, and with steady-state operations characteristics of a production plant as compared to those of a pilot plant operation, it should be possible to completely eliminate all tungsten carbide inclusions larger than a minimum size with the ferrofluid process. However, there are indications that the ferrofluid process will not be able to produce a product totally free of inclusions below a minimum size. A small number of very fine inclusions will inevitably be present because of "hindered settling" and/or "rafting." A more detailed consideration of these phenomena follows:

(b) Hindered Settling

An estimate of the particle population in a ferrofluid levitation separator may be obtained by assuming that the rate-limiting step is the transfer of titanium scrap from its introduction in the middle of the ferrofluid separator to the top of the ferrofluid pool. The ferrofluid volume in which separation takes place is illustrated schematically in Figure 17. The feed is shown being introduced at the midplane, with the products being removed at the top and bottom. The volumetric transfer rate of scrap from the midplane to the end planes is given by:

$$Q = Ay\,\epsilon/t \tag{3}$$

where

A = horizontal cross-sectional area of separator,
ϵ = volume fraction of separator occupied by scrap,
t = transit time of scrap in the ferrofluid pool (hr^{-1}),
y = one-half the height of the ferrofluid pool.

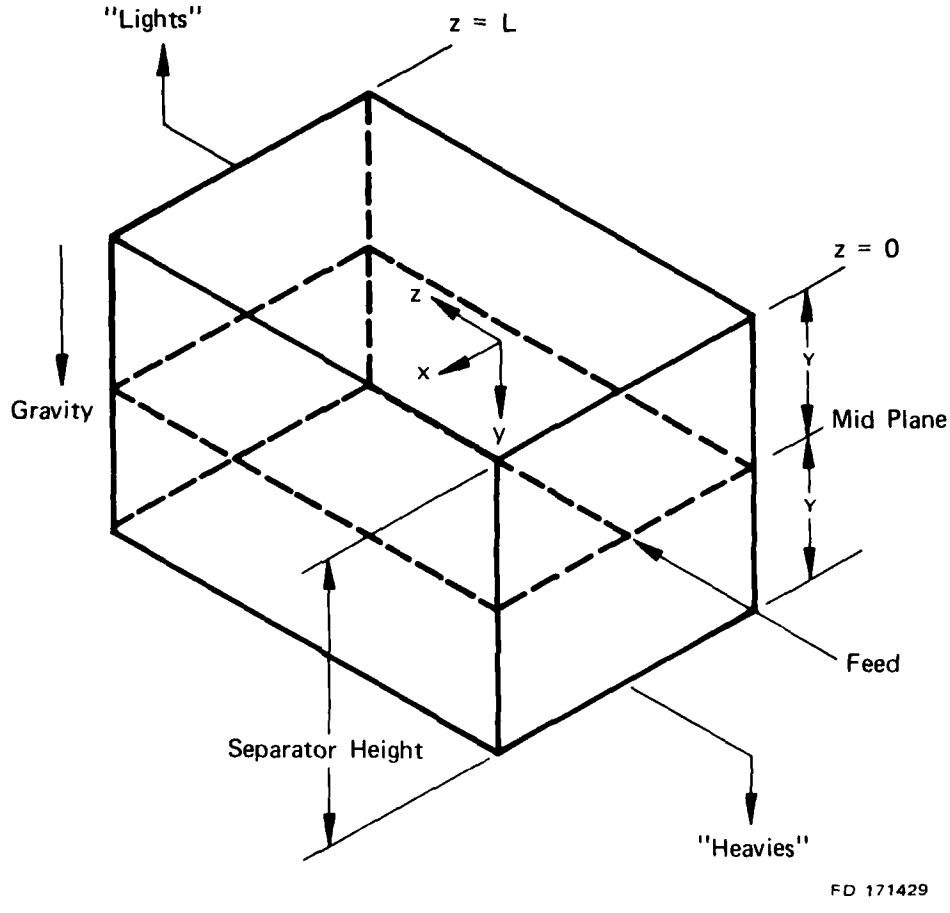


Figure 17. Schematic of AVCO Ferrofluid Pool

As derived in Appendix B, the transit time (t) of a titanium particle in a ferrofluid can be expressed by the following equation:

$$t = \frac{1}{3600} \sqrt{\frac{2y}{1 \frac{\rho_{ff}}{\rho_s} g}} \qquad (4)$$

in which,

g = acceleration of gravity,
ρ_{ff} = apparent density of the ferrofluid pool,
ρ_s = density of the solid to be sorted.

The factor 1/3600 is introduced to obtain a transit time in hr^{-1} instead of sec^{-1} usually obtained with a consistent set of units.

The product Ay is equal to half the Working Volume (V) of the ferrofluid pool. By transposing terms in Equation 3, the following explicit expression for ϵ is obtained as a function of process conditions:

$$\epsilon = \frac{2Qt}{V}. \tag{5}$$

The degree of misclassification is plotted as a function of ϵ for various process runs in Figure 18. In this figure, the values of the weight percent sinks obtained when titanium chips were processed a second time through the ferrofluid density separator are plotted against the product 2Qt/V or ϵ.

The value of t was calculated from Equation 3, using the experimental values of y = 10 cm, ρ_s, and ρ_{sf}. The value of V was taken as the working volume of the present separator which is equal to 8000 cm^3, or 0.296 ft^3. The volumetric feed rate used to obtain the value of ϵ presented in Figure 18 is based on the bulk volume of the titanium chips being fed to the system. The bulk density, rather than the true density of titanium, was used to take into account the structure of the chips and obtain a better estimate of the hydrodynamic interaction volume of a chip in a ferrofluid pool. The plot is limited to second run data in order to minimize the effect of the presence of foreign solids. It is assumed that most of the dense contaminants in the titanium in each case will have been removed in the first pass through the ferrofluid separator.

As noted in Figure 18, there appears to be little effect of chip concentrations on sorting accuracy for calculated values of $\epsilon < 0.02$. For higher values of ϵ, the weight percent sinks rises quickly and reaches dramatic values for values of $\epsilon \geq 0.03$. All of the various lots of processed titanium exhibited the same behavior even though the bulk density varied more than twofold.

These results indicate that at these higher values of ϵ, there is significant particle interaction. It is to be noted that a value of $\epsilon = 0.027$ corresponds to an average particle separation of two particle diameters. At these values, it is believed that particles are entering the separator at a faster rate than they would leave if they floated or sank of their own accord.

A design criterion for a ferrofluid separator is obtained from Figure 18. The value of ϵ should be significantly lower than 0.03.

Based on the data, it appears reasonable to use a value of $\epsilon \approx 0.015$, which is half the critical value found experimentally. By using this value of ϵ in Eq. 5, and using Eq. 4 to calculate transit time (t), it is possible to establish the maximum volumetric feed rate to the separator as a function of operating density. This volumetric feed rate can then be transformed into a mass feed rate by multiplying by the bulk density of scrap being processed. These data are presented in Table 12 for the four lots of scrap processed at apparent densities that range from 4.7 to 5.3 gm/cm^3. According to this table, it should be possible to process scrap at a volumetric feed rate of 14 to 23 ft^3/hr depending on the operating density, and in terms of mass values, from 220 to 830 lb/hr, depending on the bulk density of the scrap.

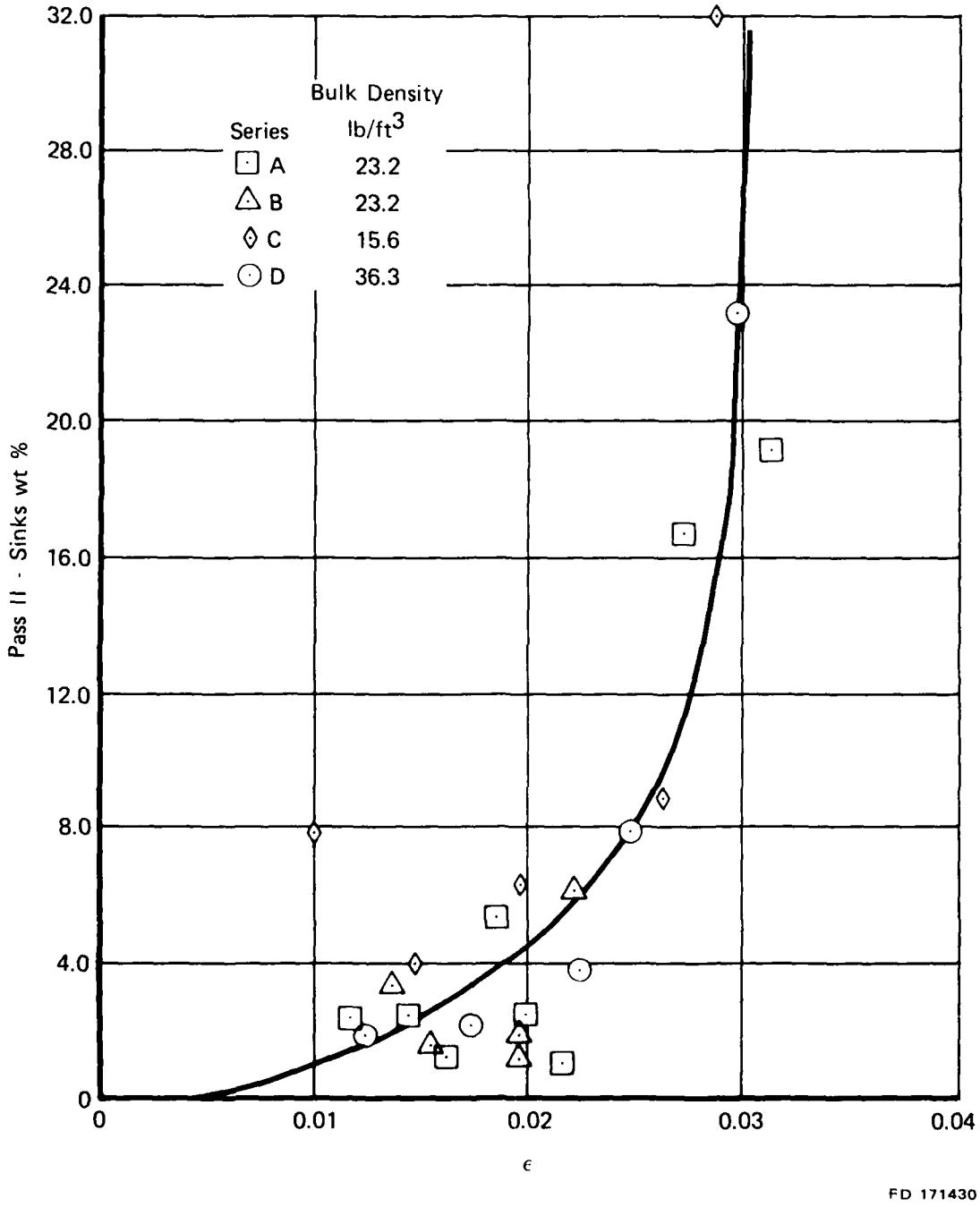


Figure 18. Weight Percent Titanium Chips in Sinks vs Dimensionless Feed Rate to AVCO Ferrofluid Density Separator

TABLE 12
DESIGN CAPACITY OF FERROFLUID SEPARATOR FOR TITANIUM CHIPS

Operating Density, ρ_{af}, gm/cm³	4.7	5.0	5.3
Residence Time(t) of Titanium Chip (ρ_{ti} = 4.4 gm/cm³) at ρ_{af}, secs	0.56	0.41	0.35
Design Bulk Feed Rate of Scrap at ρ_{af} and ϵ = 0.015 ft³/hr	14.3	19.5	22.8
Equivalent Mass Feed Rates, lb/hr			
Lot 01 and 03 (ρ_{bulk} = 23.2 lb/ft³)	330	450	530
Lot 04 (ρ_{bulk} 15.6 lb/ft³)	220	300	360
Lot 05 (ρ_{bulk} 36.3 lb/ft³)	520	710	830

(c) Rafting

A small number of very fine inclusions will inevitably be present because of what can best be called rafting. A dense inclusion, which normally would sink if left to its own devices, may be prevented from doing so if it collides with a rising titanium chip. If the average density of the inclusion and the chip is greater than the density of the ferrofluid, the inclusion will sink and carry the chip with it. If the average density of the chip and the inclusion is less than the density of the ferrofluid, the inclusion will be forced up into the floats, e.g., it will be rafted. The following expression establishes the conditions for rafting:

$$V_L < V_{TL} \frac{\rho af - \rho ti}{\rho ai - \rho af} \qquad (6)$$

where

V_L = volume of the inclusion,
V_{TL} = volume of the titanium chip,
ρaf = apparent density of the ferrofluid,
ρai = apparent density of the inclusion,
ρti = density of the titanium chip.

The maximum size inclusion that can reasonably be expected to be rafted can be calculated by knowing the relevant densities and the maximum volume of the chips being processed. In the case of Lot 01, ten chips chosen at random were weighed on an analytical balance. These ranged in weight from 0.015 to 0.1531 gm with an average of 0.0556 gm. The volume of these chips is obtained by dividing by the density of the titanium alloy (ρ_{ti} = 4.43 gm/cm³). In this case, the volume of the largest chip is 0.035 cm³. By substituting these numerical values for the volume and density of the titanium chips into Equation 6, it is possible to plot (Figure 19) the volume of the largest inclusion that one would expect to find in the floats as a function of the apparent density of the ferrofluid and the apparent density of the dense inclusion. The diameters corresponding to equivalent spherical volumes are also presented in Figure 19. As can be seen in this figure, the maximum volume misclassified is significantly decreased by operating at a density close to the density of the scrap.

A tungsten carbide tool bit is a composite consisting of tungsten carbide particles cemented in a cobalt matrix. Because cobalt is highly magnetic, a tungsten carbide tool bit fragment will have an apparent density in a ferrofluid separator that will be much higher than its true density.

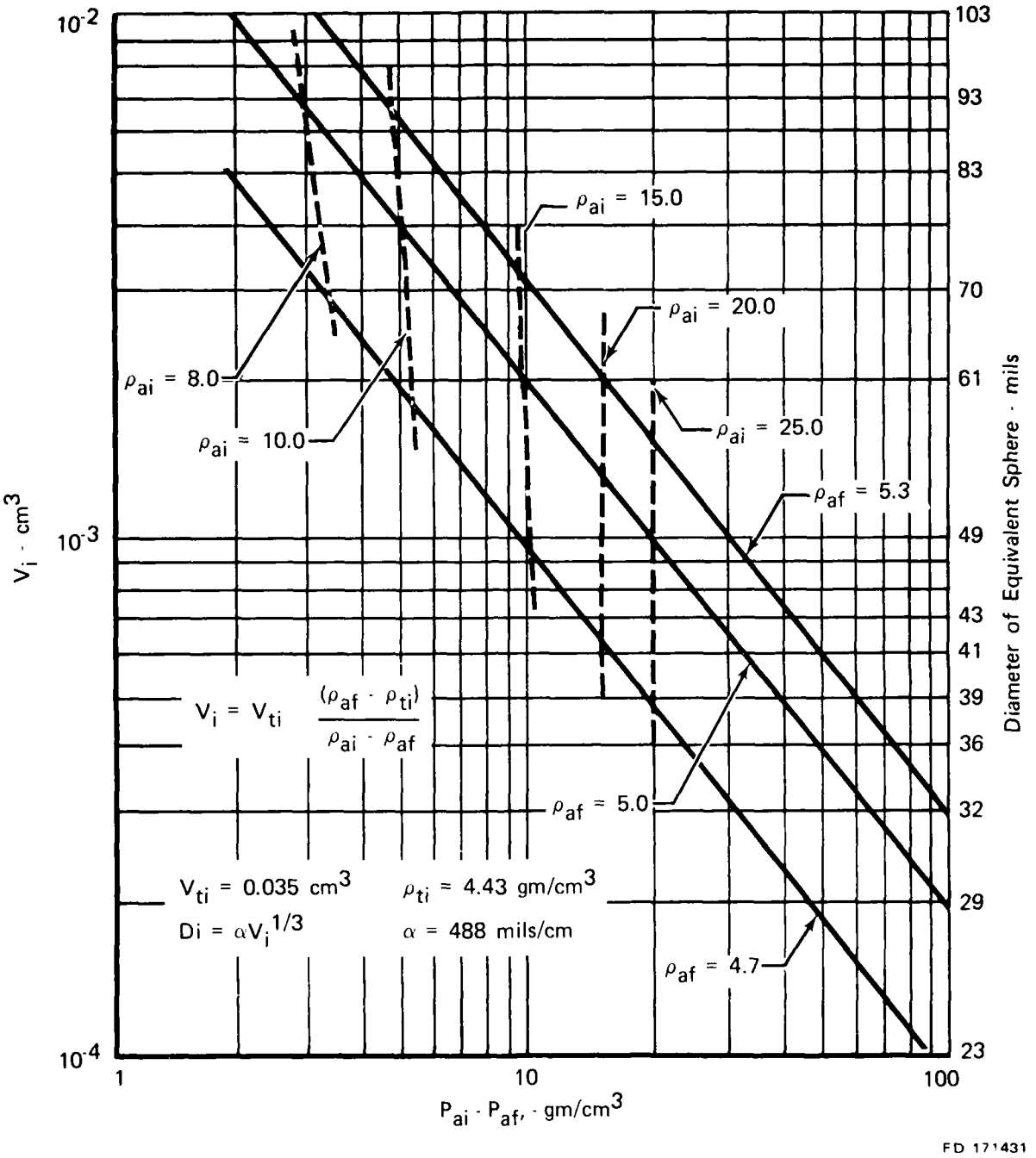


Figure 19. Expected Maximum Size of Misclassified Objects in Titanium Scrap Due to Rafting as a Function of the Apparent Densities of the Ferrofluid and Inclusion

The true density of tungsten carbide tool bit was found to be 15.0 gm/cm³. Since the density of the tungsten carbide is 15.7 gm/cm³, and the density of cobalt is 8.9 gm/cm³, this tool bit contains about 10.3% cobalt by volume. The apparent density of this object in a ferrofluid separator can be expressed by the following equation:

$$\rho ai = \rho i + \frac{(M_{Co}(V_{Co}) - M_f)}{4\pi g} \Gamma \qquad (7)$$

where

ρ_{ai} = apparent density of inclusion (tool bit), gm/cm³
ρ = true density of tool bit, gm/cm³
M_{Co} = Magnetization of cobalt, gauss
M_f = Magnetization of ferrofluid, gauss
g = acceleration of gravity
V_{Co} = volume fraction cobalt in tool bit
Γ = magnetic field gradient in separator.

In the experimental tests, a ferrofluid with a magnetization of 290 gauss was used. The magnetic field gradient was 175 oe/cm. The separator was operating at a density of 4.7 gm/cm³. The magnetization of cobalt is 24,000 gauss. By substituting these various numerical values in the above equation, the apparent density of a tungsten carbide tool bit is calculated to be about 47 gm/cm³. The apparent density of this object would be slightly higher at higher operating densities because of the higher applied gradient. From the above, the largest tungsten carbide tool bit fragment that would be rafted could be expected to decrease from 7.0×10^{-4} cm³ (D_i = 43 mils) when the ferrofluid separator was operated at an operating density of 5.3 gm/cm³, to 2.2×10^{-4} cm³ (D_i = 29 mils) when the ferrofluid separator was operated at a density of 4.7 gm/cm³.

(3) Conclusions (Test Series A-D)

The principal conclusion drawn from Test Series A-D is that it should be possible to obtain quantitative removal of macroscopic inclusions that are significantly denser than titanium. In particular, it should be possible to eliminate all tungsten carbide inclusions larger than 30 mils in diameter, as well as stainless steel and superalloy chips. The ferrofluid process will not be able to remove inclusions that have an apparent density similar to that of the titanium alloy being processed. A specific example are dense metal fragments that contain closed voids.

In order to obtain a minimum amount of misclassification in the ferrofluid separator, it is necessary to operate the separator so that there is a minimum difference between the densities of the ferrofluid (ρ_{ai}) and of the titanium alloy being processed. Furthermore, the concentration of chips, ϵ, in the separator should be small enough so that particle interaction is minimized. The operation of the system can be defined in terms of the parameters invesigated in this study. For Ti-6Al-4V alloy chips (ρ = 4.43 gm/cm³), operating the separator at an apparent density of 4.7 gm/cm³ and a maximum bulk volumetric feed rate of 14.3 ft³/hr, which results in a value of ϵ < 0.15, a quality product should be obtained in a single pass through the separator.

However, in the Test Series A-D, none of the titanium scrap was processed at chip concentration of ϵ < 0.015 and at an operating density of 4.7 gm/cm³. The runs for which ϵ < 0.015 were carried out at higher operating densities, and the feed rate for all the runs carried out at an operating density of 4.7 gm/cm³ was higher than 14.3 ft³/hr, so that ϵ > 0.15. In order to obtain operating data and samples of purified material under optimum processing conditions, additional runs were made.

(4) Supplemental Test Runs

Five supplementary runs were carried out with five different lots of titanium alloy turnings. Three runs were carried out utilizing material previously run (a remix of floats and sinks) from the three screened lots of Ti-6Al-4V of different bulk densities used in the Test Series A-D. The remaining two runs (100 lb nominal) were performed with chips of two other titanium alloys to demonstrate the general compatibility of the separation process. One run was carried out with Ti-6Al-2Sn-4Zr-2Mo turnings obtained from Frankel Co. This lot (07) had a bulk density of 35.3 lb/ft^3. The other run was carried out with Ti-5Al-2.5Sn chips. The bulk density of this material (AVCO Lot No. 08) was found to be 21.0 lb/ft^3. The 5-2.5 material was substituted for the originally planned Ti-8Al-1Mo-1V because of availability.

In Test Series A-D, the scrap was fed directly to the ferrofluid separator to eliminate the effects of other simple processing steps that would remove some of the contaminants. For these five supplementary tests, the scrap was processed under conditions that AVCO would use in a production operation. The contaminated scrap mixture was passed over a reciprocating screen, the oversize material discharged to a dry magnetic separator, and the undersize discarded. Initially, a 5-mesh reciprocating screen was used. After it was observed that significant fraction of the chips passed through the screen resulting in a significant loss of material in the process, this screen was replaced with a 10-mesh screen which retained most of the chips. A 5-mesh screen was used for Runs 56 through 58, and a 10-mesh screen was used for Runs 59 and 60.

The oversize material from the screen then passed over a magnetic pulley separator, which removed a significant fraction of strongly magnetic material from the charge stream. This dry separator prevented large pieces of very strongly magnetic material from entering the ferrofluid separator and interfering with its operation (removal of sinks). This preprocessing removes some, but far from all of the tungsten carbide tool bits present in the chips. The nonmagnetic stream from the separator was transported on a conveyor to the feed hopper for the ferrofluid density separator. The scrap was then processed through the separator under conditions so that the apparent difference in density between ferrofluid and the titanium alloy being processed was ~ 0.3 gm/cm^3 and that $\epsilon < 0.15$. For Run 57 in which $\rho_{Ti} \approx 4.46$ gm/cm^3, and Runs 58-60, in which $\rho_{Ti} = 4.43$ gm/cm^3, the operating density was 4.7-4.8 gm/cm^3. For Run 56 in which $\rho_{Ti} = 4.54$ gm/cm^3, the operating density was 4.8-4.9 gm/cm^3. In these runs, the values of ϵ ranged from 0.010 to 0.013.

In the supplementary test series, new degreasing baskets were used to receive the scrap. These baskets, which were lined with 30-mesh stainless screening, were much easier to clean and maintain than the baskets used in the previous runs. The coarser screen should not retain small tungsten carbide fragments as might have occurred before. With these baskets, recontamination of processed material should be minimal. As before, individual baskets containing separated scrap were processed in the batch degreaser to remove ferrofluid adhering to the scrap. The processed chips were then air dried and put in plastic-lined 55-gal drums.

(5) Results of Supplemental Tests

The material balances for each of the five supplemental test runs are presented in Table 13. The material balances for each of the runs in the demonstration test indicate the effectiveness of the ferrofluid density separation system to purify titanium alloy scrap. As shown in Table 13, the weight percent sinks observed experimentally correlated well with the concentration of dense contaminants originally added to the feed.

These results show that only a few percent of the titanium chips are misclassified; the majority of the titanium chips are recovered in the floats. The results were subjected to selective chemical analyses of the sinks fractions by P&WA, and radiographic inspection by AVCO.

TABLE 13
AVCO SEPARATION TEST DATA SUMMARY — SUPPLEMENTAL RUNS WITH TITANIUM ALLOY CHIPS

Run No.	*56*	*57*	*58*	*59*	*60*
Lot No.	07	08	04	05	01
Alloy Type	Ti-6Al-2Sn-4Zr-2Mo	Ti-4Al-2.5 Sn	Ti-6Al-4V	Ti-6Al-4V	Ti-6Al-4V
Density, gm/cm^3	4.54	4.46	4.43	4.43	4.43
Bulk Density, lb/ft^3	35.3	21.0	15.6	36.3	23.2
Weight % Contaminants Added at Avco, Wc Before Screening	4.5	4.9	4.5*	4.1*	8.6*
Feed Rate Scrap, lb/hr	375.0	287.0	215.0	455.0	245.0
Bulk Feed Rate Scrap, ft^3/hr	10.6	13.7	13.7	12.5	10.6
Operating Pressure Separator in H_2O	38 to 39	37 to 38	37 to 38	37 to 38	38.0
Operating Density FF gm/cm^3	4.8 to 4.9	4.7 to 4.8	4.7 to 4.8	4.7 to 4.8	4.7 to 4.8
$(\rho_{FF} - \rho_s)$ gm/cm^3	0.3	0.3	0.3	0.3	0.3
	0.011	0.012	0.013	0.012	0.010
Weight Scrap to System lb	118.0	121.1	88.9	106.0	98.2
Weight Removed by Screening and Ordinary Mag Sepn, lb	35.0**	48.5**	21.9**	3.0***	2.7***
Weight to Separator, lb	83.0	72.6	67.9	103.0	95.5
Weight Floats, lb	78.0	68.2	65.6	96.3	86.0
Weight Sinks, lb	4.5	4.0	2.3	7.0	8.6
Weight % Sinks, Ws	5.4	5.5	3.5	6.9	9.1
(Ws - Wc),	0.9	0.6	(−1.0)	2.8	0.5

*Estimated from remix proportions
**5 mesh screen
***10 mesh screen

(6) Separation Test Radiographic Program

After AVCO completed the X-ray calibration work, sample quantities (~10 lb) of Lots 01, 03, 04 and 05 were passed through the Ferrofluid Scrap Separator at an operating pressure of 45 in., which corresponds to a density of 5.5 gr/cm^3. The floats were cleaned and then used to establish X-ray inspection conditions for tungsten carbide impurities. Tungsten carbide fragments of different sizes (+5 mesh, −5/+10 mesh, −10/+18 mesh, −18/+30, −30/+50 mesh, −50 mesh/+80 mesh, −10/+18 mesh, −30/+50 mesh, −50 mesh/+80 and −80 mesh) were attached to two 3- by 5-in. cardboard cards. These cards formed reference samples for the X-ray tests. In tests, the first card was placed on a lucite tray and covered with a known amount of titanium scrap. The second card was then placed on top of the scrap layer. The sample was then X-rayed with a Faxitron Radiographic Inspection System. For the different types of scrap, the parameters investigated were the weight of scrap, beam voltage, exposure time and type of film. The horizontal dimensions of the sample were kept constant to 11- by 14-in., the size of the largest standard format X-ray film.

The best conditions developed for each case are listed in Table 14. Under these conditions, all the tungsten carbide pieces on the reference cards were visible. Increasing the height of the scrap on the tray tended to decrease the detection sensitivity. The maximum amount of scrap for the various lots corresponded to a height of about one inch. The inclusions were more readily detected with Kodak-Type AA film than with Type M film. So-called "Lead-Pack Film" resulted in marginally sharper negatives than AA-type film. This marginal advantage was not enough to warrant use of the Lead-Pack Film which is 50% more expensive than AA film.

TABLE 14
X-RAY TEST CONDITIONS FOR DETECTION OF TUNGSTEN CARBIDE INCLUSIONS IN TITANIUM TURNINGS — AVCO

Lot No.	Bulk Density of Scrap (lb/ft^3)	Weight of Scrap on 11 × 14-in. Tray (gm)	(lb)	Beam Voltage (KV)	Exposure Time (Sec)	Film Type
01	20 to 22	640	1.41	110	30	AA
02	20 to 22 with fines	880	1.94	110	66	AA
03	10 to 12	480	1.06	100	30	AA
05	30 to 33	1120	2.46	110	78	AA

The AVCO Corporation completed radiographic analyses of sample "float" and "sink" byproducts from previous ferrofluid titanium separation tests. The X-ray films were used to define contaminants in the separated fractions, particularly tungsten carbide (WC). On the basis of the radiographic inspection, float and sink samples from six different test runs were selected for further (final) analysis.

Float samples were forwarded to Teledyne Wah Chang to be melted into six 100 lb (approx.) ingots, and then converted to plate for evaluation (X-ray, chemistry, microstructure, mechanical properties). Sink samples were forwarded to P&WA for button melting and chemical analysis. The degree of successful scrap separation of other than WC contaminants attained with the ferrofluid system was determined primarily by chemical analysis.

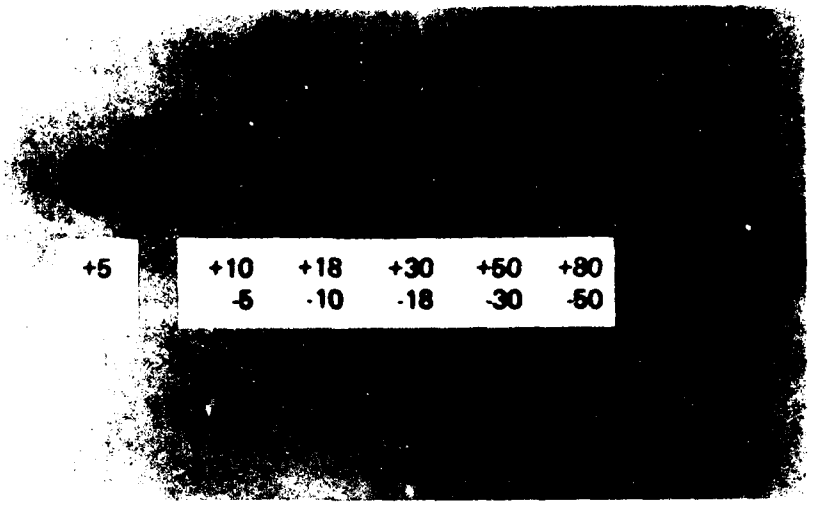
(a) Radiograph Inspection Procedure

Tungsten carbide tool bit fragments, because of their relatively high density and characteristic shape, are readily identified by X-ray. AVCO calibration runs with X-ray equipment proved that WC fragments as small as 0.005-in. square could be identified in an 11- by 14- by 1-in. sample of loose chips. For these reasons, plus the knowledge that successful separation of a tool bit fragment correlates with separation of other contaminants, AVCO used X-ray inspections to identify the most successful (clean) ferrofluid separation runs.

In preparation for the radiographic survey, samples of floats and sinks had been obtained from all the AVCO ferrofluid runs. The volume of floats from any given run was such that 55-gal drums were required for their storage, whereas the sinks could be contained in 5-gal pails. Float samples were obtained by removing a quantity from each basket of floats leaving the degreaser. The sample quantity was at least 10% of the total floats from each run. In addition, any material brushed out of the float basket at the end of a cycle run was added to the X-ray sample. Since only fine contaminants would tend to cling to the basket, it is suspected that this addition of fines tended to give the X-ray sample a somewhat higher-than-representative contaminant level.

Each X-ray sample was stored in a polyethylene bag, and the bags in turn stored in covered five-gallon pails to minimize the possibility of post-processing contamination. Prior to X-ray, the samples were transferred from the bags into two different sets of lucite trays, one set for sinks, the other set for floats. Each tray, 11 in. wide, 14 in. long and 1 in. deep, was appropriately labeled. For baseline reference, various size WC chips were glued to a card and the card placed on top of the sample chips prior to X-ray (Figure 20).

Subsequent to X-ray, the 11- by 14-in. radiographs (X-ray negatives) were examined with a magnifier for inclusions. Suspected indications were marked on the negative, and after identification, measured for size; however, if less than 10 inclusions were observed on a radiograph, the size of each inclusion was recorded, and if more than 10 inclusions were observed, the range of sizes only was recorded. Selected inclusions were removed from the chips in the tray for subsequent analysis. The loaded trays were saved until examination of the radiographs was completed. The contents were then returned to an appropriate float or sink container.



U.S. Standard Sieve Series	Sieve Opening	Wire Diameter
No. 5	0.157	0.0539
No. 10	0.787	0.0354
No. 18	0.0394	0.0223
No. 30	0.0234	0.0154
No. 50	0.0117	0.0085
No. 80	0.0070	0.0052

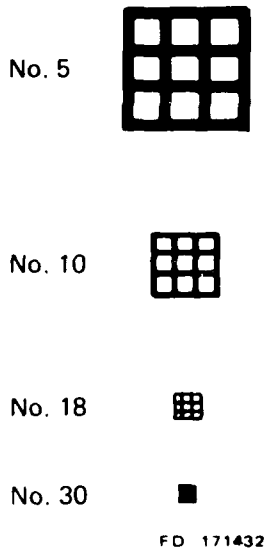


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Figure 20. AVCO Tungsten Carbide Particle Sample Card

(b) Radiographic Inspection Results and Analyses

As noted previously, AVCO split their ferrofluid systems tests (titanium separation) into two parts: Series A-D to establish basic parameters, and Supplementary Tests to establish the validity of those parameters for production operation. Inspection results and analysis for each test series are documented in the following paragraphs.

(c) Test Series A-D X-ray Results

X-ray inspection results of Test Series A-D floats are presented in Table 15. The high level of contaminants in the A-D sinks precluded an inclusion count.

Radiographs of sinks from "blank" runs, i.e., no contaminants added, established that all lots of as-received scrap contained WC plus other contaminants. Radiographs also confirmed the capability of the ferrofluid system to concentrate these contaminants in the sinks. For example, Figure 21 shows a sample of as-received Lot 01 scrap material with only two inclusions identified as WC, while Figure 22 shows a sample of the sinks of Lot 01 "blank" run material with several WC inclusions evident. The two large inclusions shown in Figure 22 were removed, measured for density and photographed (Figure 23). The smaller piece with two holes had a density of 8.63 gm/cm^3 and therefore may be a titanium alloy. In general, Lot 01 material did not appear to contain many chips of foreign materials.

A radiograph of the sinks of Run 29 is presented in Figure 24. This blank run was carried out with as-received high-bulk-density titanium alloy chips (Lot 05). In addition to tungsten carbide, this lot contained chips of somewhat greater opaqueness than titanium, and many sheet trimmings, probably from a punch press operation, that were definitely more opaque than the titanium chips, and were probably superalloy.

The low bulk density titanium chips (Lot 04) were badly contaminated. A radiograph of the sinks of Run 41 is presented as Figure 25. In addition to tungsten carbide, this lot contained not only a significant quantity of foreign chips but also numerous other inclusions, as shown in Figure 26, including sizable fragments of stainless steel and brass. A large number of dense rounded inclusions which did not have the sharp angular facets of tungsten carbide were also observed. These pieces are fairly soft, suggesting a lead-containing alloy.

The presence of these inclusions appeared consistent with the chemical analysis provided with the as-received chips (Table 4), which indicated that all the chip lots had some degree of contamination. In particular, Lot 04 was found to have the highest iron and lead content of the as-received lots.

The most extensive data was obtained with medium-bulk-density titanium alloy chips comprising Lot 01, which was screened, and Lot 03, which was not screened. The contaminated feed mixtures of Lot 01 contained 0.37 weight-percent of deliberately added tungsten carbide chips, that ranged from +5 to −80 mesh in size, in addition to any naturally occurring contaminants. The expected concentration of seeded fragments of various sizes are presented in Table 16. Based on this table, an X-ray sample tray containing 1.4 lb of contaminated feed mixture should therefore contain over 400 detectable (0.005-in. dia) inclusions; Figure 27 is a radiograph of a typical sample of this contaminated feed mixture.

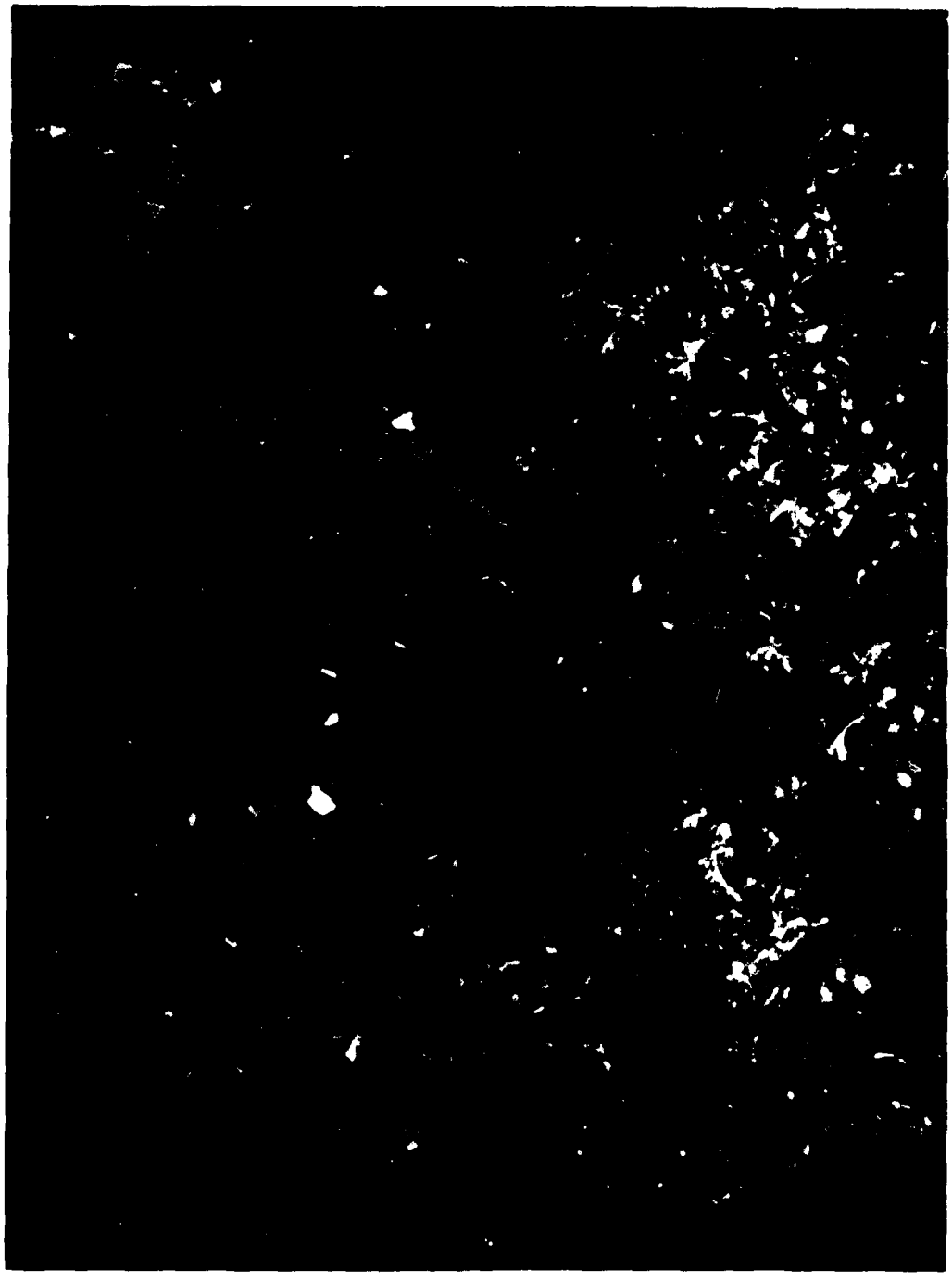
TABLE 15
AVCO EXPERIMENTAL DENSITY SEPARATION RADIOGRAPHIC TEST DATA TITANIUM SERIES A-D.

Feed Material	Run No.	Contam.	Feed Rate lb/hr	Operating Pressure in H_2O	Oper Density (gm/cm³)		Weight of X-ray Sample (lb)	Number of Inclusions Found in each size (in.) Less Than .015	.015 To .029	.030 To .045	.045 To .059	Over .060	Total Number Inclus	Inclus lb	Size of Largest Inclusion	Percent Lost X-ray Data
SERIES A																
Low Oil	2	B*	400	37	4.7	0.018	13.8	2	2	1	0	1	6	0.4	0.050 + 0.200	
Medium Density	1	B	445	40	5.0	0.016	16.8	6	13	4	2	2	27	1.6	0.065	
Ti-6Al-4V Screened	53	B	425	40	5.0	0.015	14.2	21	7	2	1	0	31	2.3	0.050	
	52	B	401	42	5.3	0.014	14.7	13	8	1	0	3	25	1.7	0.020 + 0.075	
	9	C	439	37	4.7	0.020	15.4	6	2	0	0	0	8	0.5	0.025	
	10	C	595	37	4.7	0.033	15.1	19	4	0	0	1	24	1.6	0.105 + 0.225	
	6	C	410	40	5.0	0.014	14.9	16	8	0	0	0	24	1.6	0.025	
	7	C	659	40	5.0	0.022	14.4	9	5	1	0	0	15	1.0	0.030	
	8	C	890	40	5.0	0.030	13.3	4	3	0	0	1	8	0.6	0.055 ID + 0.125 OD**	
	3	C	365	42	5.3	0.011	14.5	13	5	1	1	2	22	1.5	0.060 + 0.250	
	4	C	530	42	5.3	0.015	15.1	76	10	1	0	0	87	5.8	0.030	
	5	C	785	42	5.3	0.022	13.8	18	3	0	0	0	21	1.5	0.150**	
	54	C	1200	42	5.3	0.034	12.4	26	4	2	0	6	38	3.1	0.220 + 0.275	
50 Run 9 Floats	17	C	400	37	4.7	0.018	14.2	9	6	0	0	0	15	1.0	0.020	
50 Run 10 Floats	18	C	550	37	4.7	0.025	13.7	11	3	1	0	0	15	1.1	0.035	
50 Run 6 Floats	14	C	446	40	5.0	0.015	17.7	15	10	1	0	0	26	1.5	0.025	
50 Run 7 Floats	15	C	800	40	5.0	0.020	14.0	22	4	0	0	0	26	1.9	0.020	
50 Run 8 Floats	16	C	785	40	5.0	0.026	13.0	9	6	1	0	1	17	1.3	0.030 + 0.130	
50 Run 3 Floats	11	C	410	42	5.3	0.012	16.1	12	2	0	0	1	15	0.9	0.080	
50 Run 4 Floats	12	C	585	42	5.3	0.017	16.1	14	7	0	0	1	22	1.4	0.105 + 0.160	
50 Run 5 Floats	13	C	800	42	5.3	0.023	12.7	22	1	0	0	0	23	1.8	0.075	
50 Run 54 Floats	55	C	1140	42	5.3	0.033	13.1	20	4	0	1	2	27	2.1	0.200 + 0.210	
SERIES B	20	C*	410	37	4.7	0.019	7.5						167	9.5	0.200	
Low Oil	21	C	550	37	4.7	0.025	13.5	33	31	2	0	2	68	5.0	0.125	
Medium Density	23	C	440	40	5.0	0.023	15.2		165		0	0	165	10.8	0.050 + 0.175	
Ti-6Al-4V	22	C	760	40	5.0	0.025	19.0						129	6.8	0.055	
Not Screened	19	C	475	42	5.3	0.014	21.0	41	78	17	10	4	140	6.7	0.140	
50 Run 20 Floats	25	C	492	37	4.7	0.025	16.4						55	3.4	0.015 + 0.375	
50 Run 21 Floats	26	C	650	37	4.7	0.030	13.1						28	2.2	0.375 + 0.475	
50 Run 23 Floats	28	C	385	40	5.0	0.013	15.7		22	1	0	0	23	1.5	0.025 + 0.050	
50 Run 22 Floats	27	C	695	40	5.0	0.030	19.4						41	2.1	0.025 + 0.335	
50 Run 19 Floats	24	C	612	42	5.3	0.018	19.7						44	2.2	0.100	

TABLE 15
AVCO EXPERIMENTAL DENSITY SEPARATION RADIOGRAPHIC TEST DATA TITANIUM SERIES A-D (CONTINUED)

Feed Material	Run No.	Contam.	Feed Rate lb/hr.	Operating Pressure in. H_2O	Oper. Density gm/cm	ε	Weight of X-ray Sample lb	Number of Inclusions Found in each size run: Less Than 0.015	0.015 To 0.029	0.030 To 0.045	0.045 To 0.059	Over 0.060	Total Number Inclus.	Inclus. lb	Size of Largest Inclusion	Pieces in Last X-ray Tray
SERIES C																
Lot 05	30	B	725	37	4.7	0.021	22		135	0	0	0	135	6.1	0.025	120
High Density	29	B	660	40	5.0	0.015	18.2		108	0	0	0	108	5.9	0.020	105
Ti-6Al-4V																
	34	C	715	37	4.7	0.021	23.5						185	7.9	0.030 - 0.135	175
	35	C	430	40	5.0	0.010	10.4						110	10.6	0.065	100
	33	C	715	40	5.0	0.015	23.7		112	0	2	0	114	4.8	0.050 - 0.200	110
	31	C	955	42	5.3	0.017	27		174			2	176	6.5	0.105	150
	32	C	1090	42	5.3	0.020	23.4						129	5.5	0.030 - 0.080	125
50% Run 34 Floats	39	C	785	37	4.7	0.023	20.4		42	1	0	0	43	2.1	0.055	
50% Run 35 Floats	40	C	620	40	5.0	0.013	16.0		24			1	25	1.6	0.070 - 0.105	15
50% Run 33 Floats	38	C	930	40	5.0	0.020	20.7		45		0	0	45	2.3	0.030	43
50% Run 31 Floats	36	C	935	42	5.3	0.017	27	50	4	1	0	0	55	2.4	0.055	48
50% Run 32 Floats	37	C	1100	42	5.3	0.020	22		112	0	2	0	114	5.2	0.050	110
SERIES D																
Lot 04	41	B*	384	37	4.7	0.026	10.4						160	15.4	0.250 - 0.300	
Low Density																
Ti-6Al-4V																
	46	C	345	37	4.7	0.023	10.4						100	9.6	0.250 - 0.470	
	45	C	130	40	5.0	0.0065	8.7						94	10.8	0.320 - 0.500	
	44	C	365	40	5.0	0.018	10.0						130	13.0	0.300 - 0.425	
	42	C	400	42	5.3	0.017	A 10.4						277	26.6	0.400 - 0.700	
							B 8.2						80	9.8	0.260	
	43	C	695	42	5.3	0.029	9.0						78	8.7	0.180 - 0.355	
50% Run 46 Floats	51	C	395	37	4.7	0.027	9.4						42	4.3	0.450	
50% Run 45 Floats	50	C	202	40	5.0	0.010	7.8						19	2.4	0.325	
50% Run 44 Floats	49	C	390	40	5.0	0.020	9.7						64	6.6	0.250	
50% Run 42 Floats	47	C	365	42	5.3	0.015	9.2						51	7.7	0.200	
50% Run 43 Floats	48	C	665	42	5.3	0.028	9.0						61	6.8	0.470	

*B Blank
C Contaminants Added
**Not WC



FD 171433

Figure 21 Photo Reduction of 11 × 14-in. AVCO Radiograph of Lot 01 Ti Alloy Chips As-Received

FD 171434

Figure 22. Photo Reduction of 11 × 14-in. AVCO Radiograph of Sinks of Lot 01

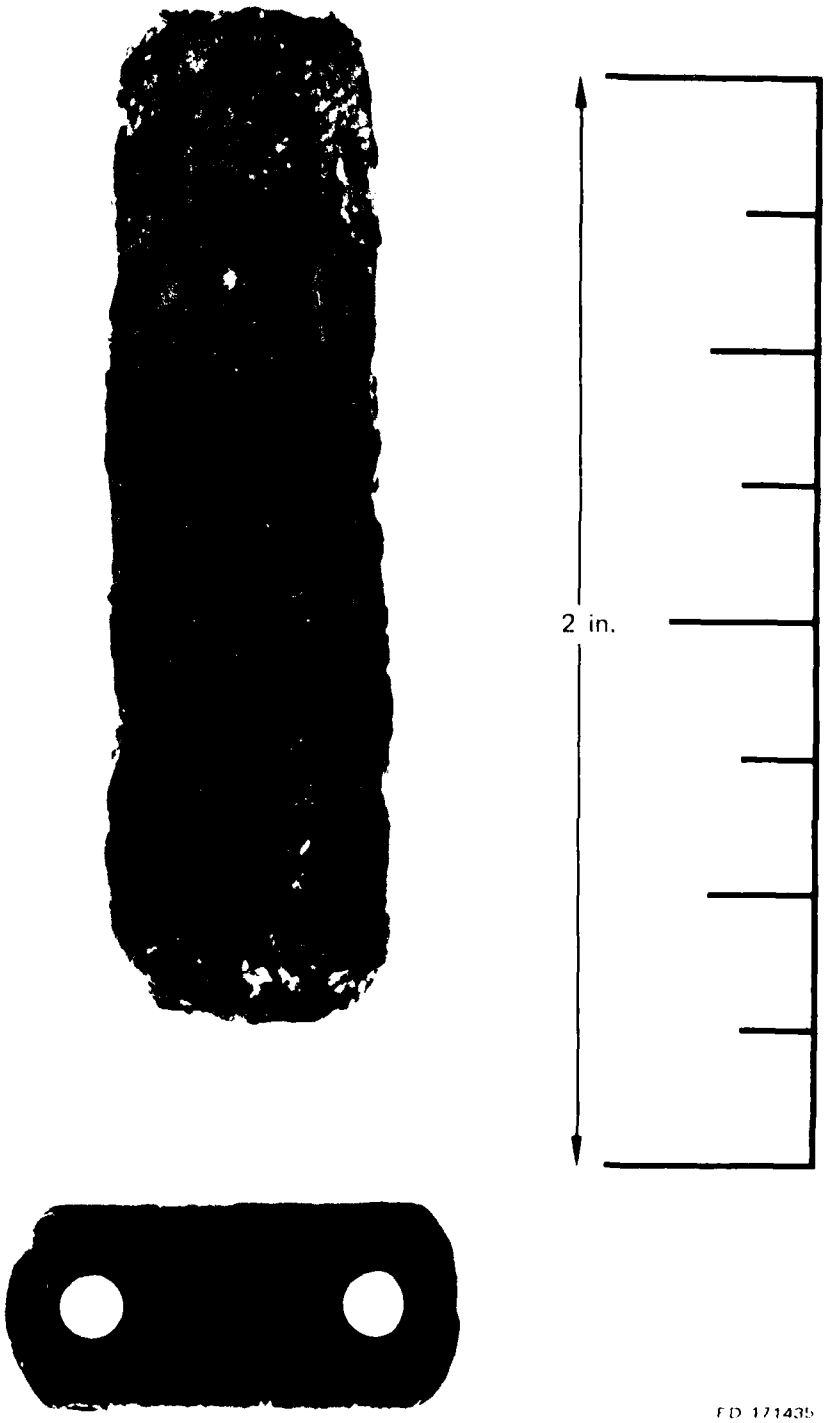
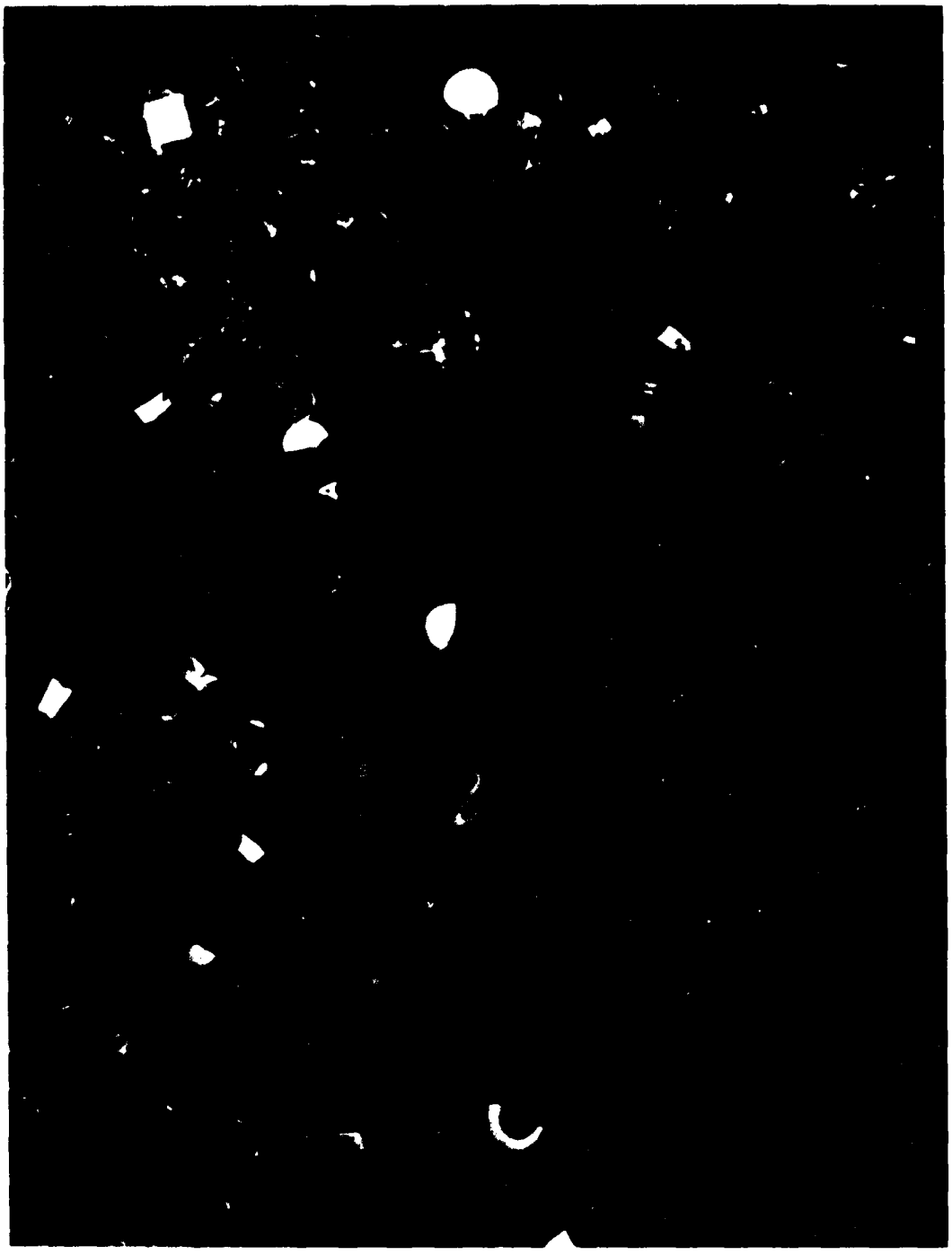
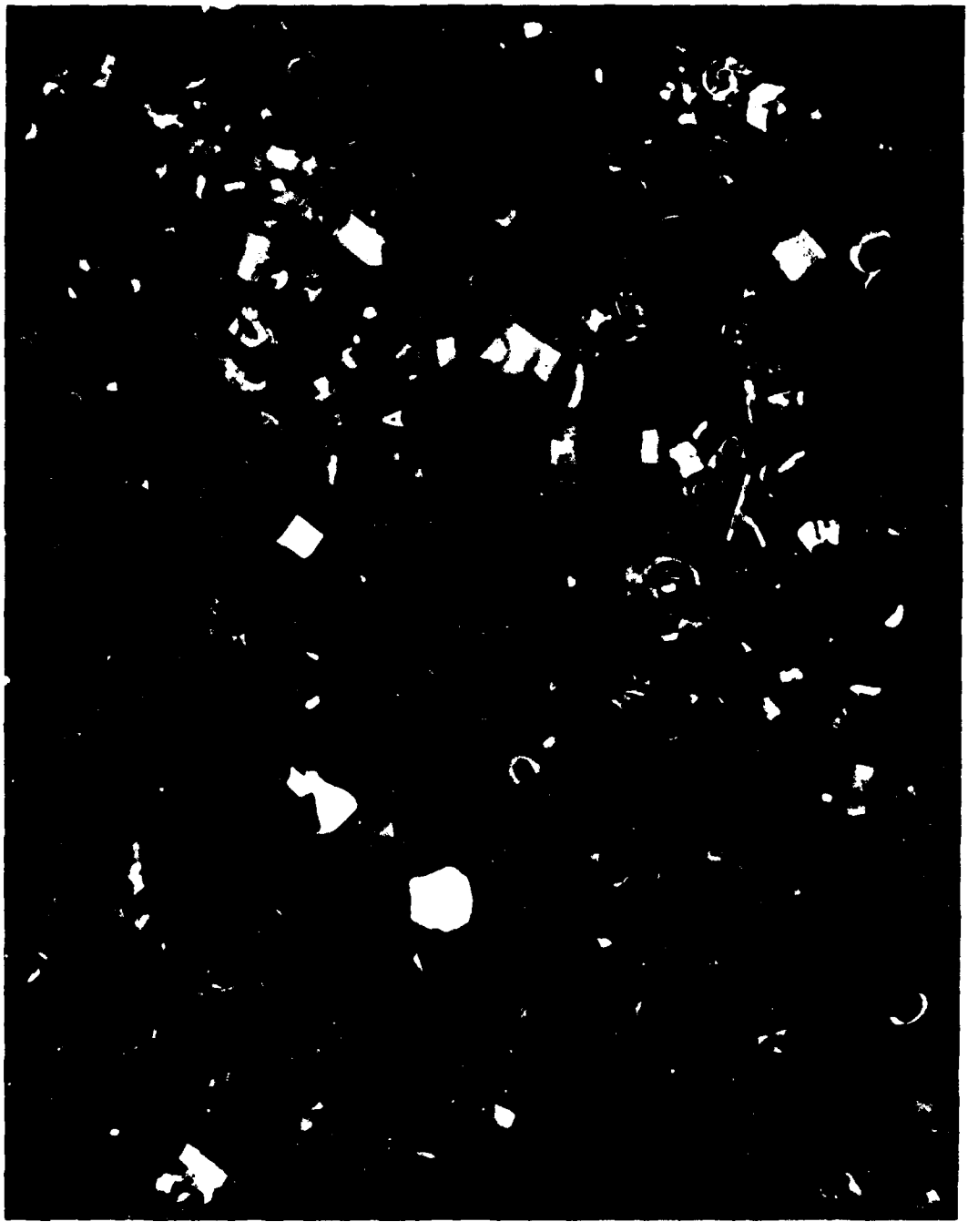


Figure 23. Foreign Metal from Sinks of AVCO Lot 01



FD 171436

Figure 24. Photo Reduction of 11 × 14-in. AVCO Radiograph of Sinks of Run 29, Lot 05



FD 171437

Figure 25. Photo Reduction of 11 × 14-in. AVCO Radiograph of Sinks of Run 41, Lot 04

Figure 26. Inclusions from Sinks of AVCO Run 41, Lot 04

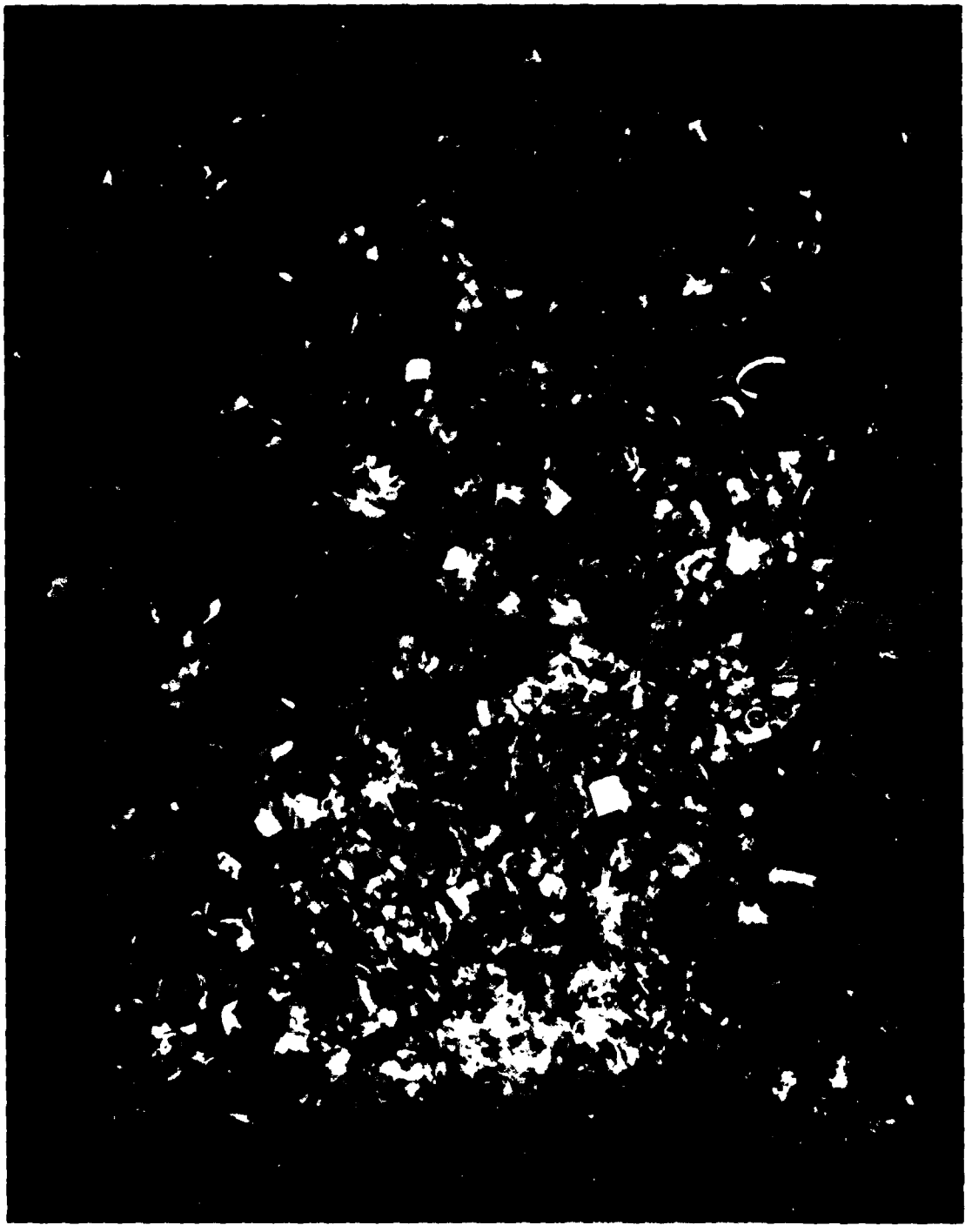
TABLE 16
TUNGSTEN CARBIDE CONTAMINATION ADDED TO SCREENED MEDIUM BULK DENSITY TITANIUM ALLOY CHIPS (LOT 01) AVCO TEST SERIES A

Size of Tungsten Carbide Fragments		*Expected Number of Tungsten Carbide Fragments Per Pound of Titanium Alloy Chips*	
US Mesh	*Mils*	*In Size Range*	*Cumulative Number*
+5	>156	1	1
5 to +10	158 to +78	1	2
10 to +18	79 to +39	4	6
18 to +30	39 to +23	12	18
30 to +50	23 to +12	48	66
50 to +80	12 to +7	236	302
-80	7	3900	4202

In all cases, the processing of contaminated titanium alloy scrap from either Lot 01 or Lot 03 through a ferrofluid sink-float separator resulted in a decrease in the concentration of detectable contaminants. Typically, an initial concentration in excess of 300 inclusions/lb of titanium decreased to values which ranged from 0.5 to 5.8 inclusions/lb titanium for Series A, Lot 01, and from 1.5 to 9.5 inclusions/lb titanium for Series B, Lot 03. This represents a reduction in contaminant level from 30-fold to 600-fold. In comparison, the contamination level of the floats from blank runs ranged from 0.4 to 2.3 inclusions/lb titanium. There were a number of Lot 01 Runs (4, 5, 13, 6, 14, 7, 15, 9 and 17) in which the largest WC inclusion found had a diameter of 0.030 in. or less. Other runs in this series contained one or more WC inclusions larger than 0.030-in. diameter. All the runs processed with Lot 03 as a feed material contained one or more larger WC inclusions. The best results were obtained with Run 9 (Lot 01, ρaf = 4.7 gm/cm^3, feed rate = 439 lb/hr, $\epsilon = 0.020$) in which eight inclusions, all smaller than 0.025-in. dia, were found in 15.4 lb X-ray sample.

The highest level of contaminants and the largest contaminants were found in the floats of Runs 10, 54 and 55 for which $\epsilon \sim 0.33$. It was previously reported that these runs had an unusually large amount of titanium in the sinks. In general, it is observed that the degree of misclassification increases as the difference in density between the ferrofluid and the scrap increases and also as the feed rate to the separator increases. When the degree of misclassification is fairly high, after an initial pass through the separator, a second pass will result in further removal of dense contaminants. When the degree of removal after an initial pass is high, a second pass through the system does not bring about a significant further improvement.

These results indicate that the bulk of the dense contaminants are removed by the ferrofluid density separation process under a wide variety of operating conditions. This was the case even when the ferrofluid separator was operated under conditions where there was significant misclassification of titanium scrap. This is consistent with the fact that the density difference between the contaminants and the ferrofluid was much greater than the density difference between the ferrofluid and the titanium chips. Optimum contaminant removal cannot be expected, however, under conditions which result in a high misclassification of the titanium chips. Process conditions which result in high misclassification are characterized by a high value for ϵ. The very poor results obtained with Lot 01 Runs 10, 54 and 55 ($\epsilon \sim 0.030$) and with Lot 03 Runs 19 to 28 support this conclusion. ϵ values calculated for Runs 19 to 28 (0.013 to 0.030) assumed that the residence time of the chips in the separator was controlled by inertia. Because of the presence of a roughly 10% fines which would have large residence times, the actual value of ϵ is probably higher than calculated. The floats for runs with Lot 03 feed were in general more contaminated than those carried out with Lot 01 feed material.



FD 171439

Figure 27. Photo Reduction of 11 × 14 in. AVCO Radiograph of Sinks of Representative Sample of Contaminated Local Mexican Run 60

The presence of a few large WC fragments in the floats of runs which approach optimum conditions might have been due to recontamination of processed material leaving the separator. The degreasing baskets used to contain the titanium chips in the ferrofluid removal step were lined with a 50-mesh steel screen. Although the baskets were brushed carefully after processing each run, the possibility exists that a few WC pieces could be carried over from a poor run to a subsequent run.

The results obtained with the high-bulk-density Lot 05 titanium chips (Table 15) were qualitatively similar to those obtained with Lot 01; however, the X-ray samples of floats generally contain more inclusions than did the samples from Lot 01. Most of these inclusions were less than 0.030-in. dia. On the total sample basis, the concentration of inclusions found ranged from 1.6 inclusions/lb (Run 40) to 10.6 inclusions/lb (Run 35); however, a large majority of these inclusions occurred in the X-ray tray corresponding to the bottom of the sample pails. If the bottom portion of the samples is discounted, the inclusion concentration is decreased by an order of magnitude.

A possible explanation for these results is that this lot of titanium chips contained many WC fragments that adhered to the chips, due to residual cutting oil, and the chips thus behaved as particles of intermediate density which either sank or floated in the ferrofluid separator depending on the relative sizes of the chip and the adhering WC pieces. Exposure to ferrofluid followed by degreasing of the chips to remove ferrofluid could result in separation of the titanium chip from the tool fragment. The agitation in the handling of the sample container could then allow the fine WC fragments to move down through the chip pile to the bottom of the container. The very fine screen lining of the degreaser baskets would result in retention of these small fragments.

Radiographs of the floats of the various runs carried out with the low-bulk density Lot 04 titanium alloy chips, exhibited a relatively large number of dense inclusions. A number of these inclusions however, had an irregular, somewhat rounded shape rather than the characteristic sharp angular appearance of WC. These inclusions were also larger in size than previously identified WC fragments.

A typical inclusion physically removed from the floats of Run 42 shown in Figure 28, appeared to consist of a multilayered foil with a binder material. The density of one of these inclusions was determined to be 4.477 gm/cm^3. The chemistry of typical inclusions was determined. Similar, but higher, density inclusions were found in the sinks. For example, one inclusion removed from the sinks of Run 4 was found to have a density of 7.526 gm/cm^3. The different apparent densities of these inclusions is due to differences in their porosity. Since the densities were measured by liquid immersion, these measurements reflect closed voids that are not filled with liquid.

The above inclusions were not misclassified by the ferrofluid process. The objects of lower density than the ferrofluid floated, while the denser ones sank. Some of these inclusions have densities that do not differ significantly from the density of the titanium scrap being processed and thus are not readily removed by the ferrofluid process.

(d) Supplemental Test X-ray Results

As noted previously, AVCO derived optimum parameters for operation of the ferrofluid separation process during test series A-D. These parameters, i.e., an ϵ value of less than 0.015, and a small density difference between the ferrofluid and titanium, were used during the five supplementary tests (56 to 60). The chips used for Runs 58 to 60 were Ti-6Al-4V of light, heavy, and medium bulk densities, respectively; chips for Run 56 were Ti-6Al-2Sn-4Zr-2Mo, and Run 57 chips were Ti-5Al-2.5Sn turnings. In view of Series A-D test experience, each lot was subject to screening of fines by AVCO after the addition of contaminants.

FD 171440

Figure 28. Light Inclusion from Floats of AVCO Run 42 (Dimensions: 0.625 × 0.450 × 0.200 in.)

Radiographic inspection was conducted on floats from the supplementary test runs utilizing a procedure identical to that employed on the A-D runs. Radiographic inspection results are found in Table 17. Typical radiographs of the float products for each of the supplementary test runs are presented in Figures 29 through 33.

Radiographic inspection of the floats indicated a high degree of purity in the floats with significantly lower inclusion levels than the float samples obtained from the A-D runs. The largest concentration of inclusions (1.53 inclusions per pound of titanium) was found in floats from Run 58 with Lot 04 feed material. Most of these inclusions did not appear to be tungsten carbide chips.

For the other supplementary test runs, the concentration of inclusions ranged from 0.08 to 0.43 inclusions/lb of titanium. Assuming an average chip weight of 20 mg, this corresponds to a concentration level of less than one inclusion per 10^6 titanium chips. The largest inclusion that could be definitely identified as being tungsten carbide from the radiographs was 30 mils in dia. Other inclusions which were larger than 30 mils were also observed, but these did not appear to be tungsten carbide. Inclusions, typical of this larger type, were physically removed and found to have densities lower than tungsten carbide. An inclusion removed from the floats of Run 58 is shown in Figure 34, and an inclusion removed from the floats of Run 59 is shown in Figure 35. A porous slag-like appearance of both these inclusions may be noted. A photograph of the largest inclusion found in Run 60 is shown in Figure 36. This object is metallic and nonporous in character. These inclusions were chemically analyzed by P&WA

Conclusions drawn from the radiographic examination of the basic and supplementary test runs are listed below:

(a) Contaminants were present in all the lots of titanium turnings as-received, thus confirming the need for a process to remove them.

(b) The ferrofluid density separation process resulted in a significant reduction in contamination level of titanium chips for all the run conditions examined; however, some residual contaminants remained after each run.

(c) The residual concentration of contaminants in the floats is a function of the operating parameters of the separator and the characteristics of the contaminants. The principal mechanism that results in misclassification of particles, appears to be particle interaction, especially rafting. In order to minimize this problem, it is necessary to operate a ferrofluid separator under conditions such that ϵ 0.015 with a ferrofluid density approaching the density of the scrap.

(d) The ferrofluid separation process, utilizing suitable operating parameters, appears to be capable of removing all tungsten carbide fragments larger than 0.030-in. dia as well as a significant fraction of the smaller fragments. Conditions which lead to the removal of tungsten carbide fragments also lead to the removal of stainless steel and superalloy turnings. Porous contaminants which may contain closed voids, and which have an apparent density approaching that of titanium are not effectively removed by the ferrofluid process.

e. *Preparation for Ingot Melting*

Float products from six specific Ti-6Al-4V test runs were selected for melting and further evaluation. These six runs included basic (Series A-D) Nos. 6, 9 and 23, and supplementary test runs 58, 59 and 60.

TABLE 17
AVCO SUPPLEMENTARY TEST RUNS

Feed Material	*Run No* *	*Feed Rate (lb/hr)*	*Operating Pressure (in H_2O)*	*Operating Density (gm/cm³)*		*Weight of X-ray Sample (lb)*	*Less Than 0.010*	*0.010 To 0.019*	*0.020 To 0.030*	*Over 0.030*	*Total Number Inclus*	*Inclus (lb)*	*Size of Largest Inclusion*
Lot 07 35.3 lb/ft³ Bulk Density Ti 6Al-2Sn-4Zr-2Mo	56	375	38 to 39	4.8 to 4.9	0.011	37.0	1	1	1	0	3	0.08	0.030
Lot 08 21.0 lb/ft³ Bulk Density Ti 5Al-2.5 Sn	57	287	37 to 38	4.7 to 4.8	0.012	18.8	6	0	1	1**	8	0.43	0.035 × 0.060
Lot 04 15.6 lb/ft³ Bulk Density Ti 6Al-4V	58	215	37 to 38	4.7 to 4.8	0.013	14.2	7	8	3	6***	22	1.53	0.250
Lot 05 36.3 lb/ft³ Bulk Density Ti 6Al-4V	59	455	37 to 38	4.7 to 4.8	0.012	45.3	2	1	1	1*	5	0.11	0.180
Lot 01 23.2 lb/ft³ Bulk Density Ti 6Al-4V	60	245	37 to 38	4.7 to 4.8	0.010	24.7	5	1	1	1*	8	0.32	0.080

*All runs were artificially contaminated
**Not WC
***One each 0.035, 0.045, 0.055, 0.120, 0.230, and 0.250-in. size

AD-A083 089 PRATT AND WHITNEY AIRCRAFT GROUP WEST PALM BEACH FL G--ETC F/G 13/8
MANUFACTURING METHODS FOR STRATEGIC MATERIALS RECLAMATION,(U)
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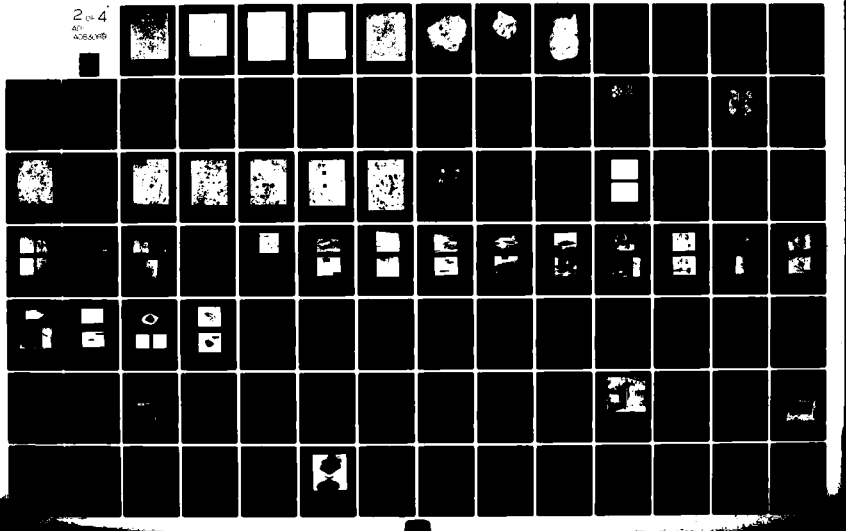
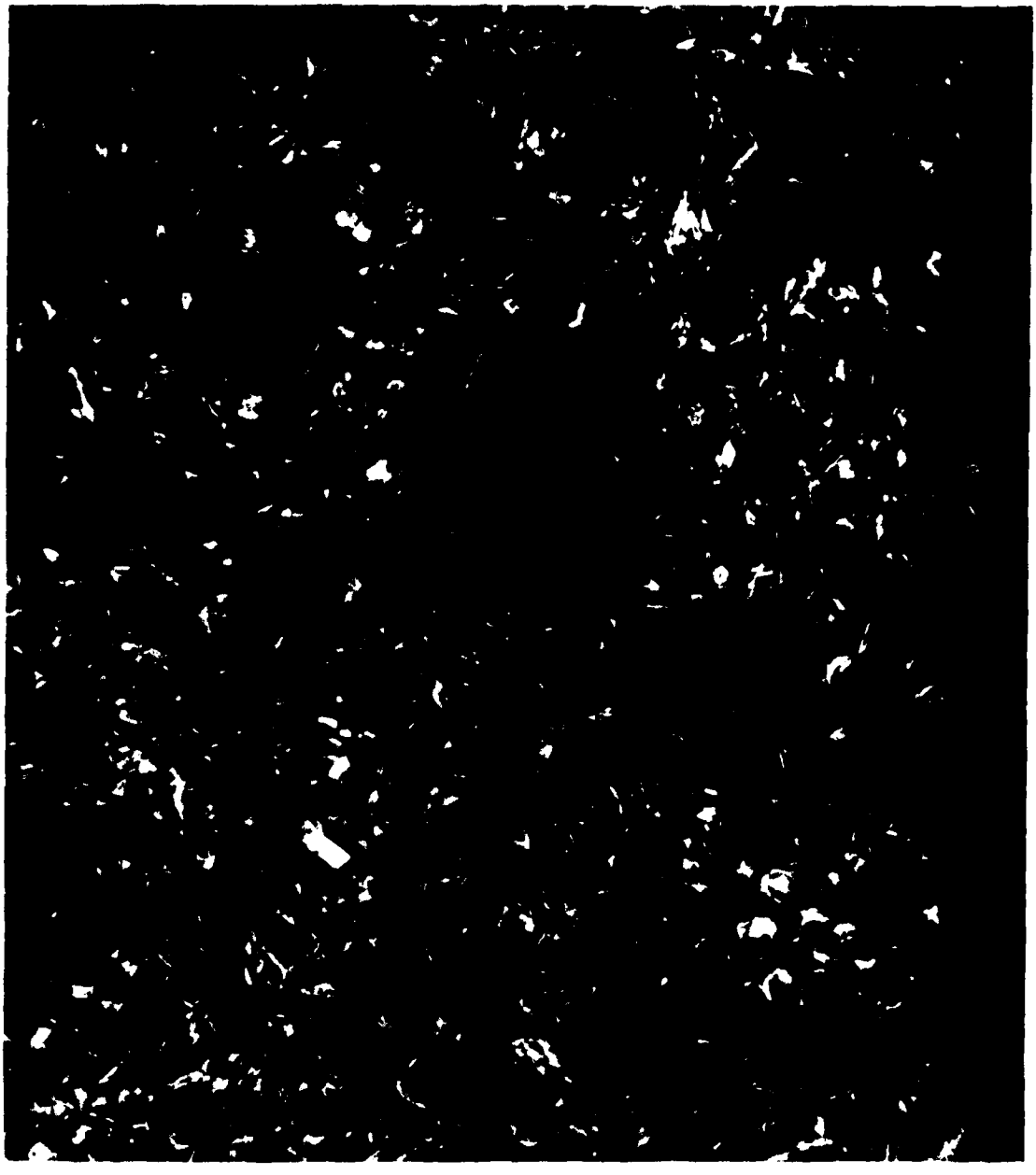


FD 171441

Figure 29. Photo Reduction of 11 × 14-in. AVCO Radiograph of Floats from Test Run No. 56, Lot 07

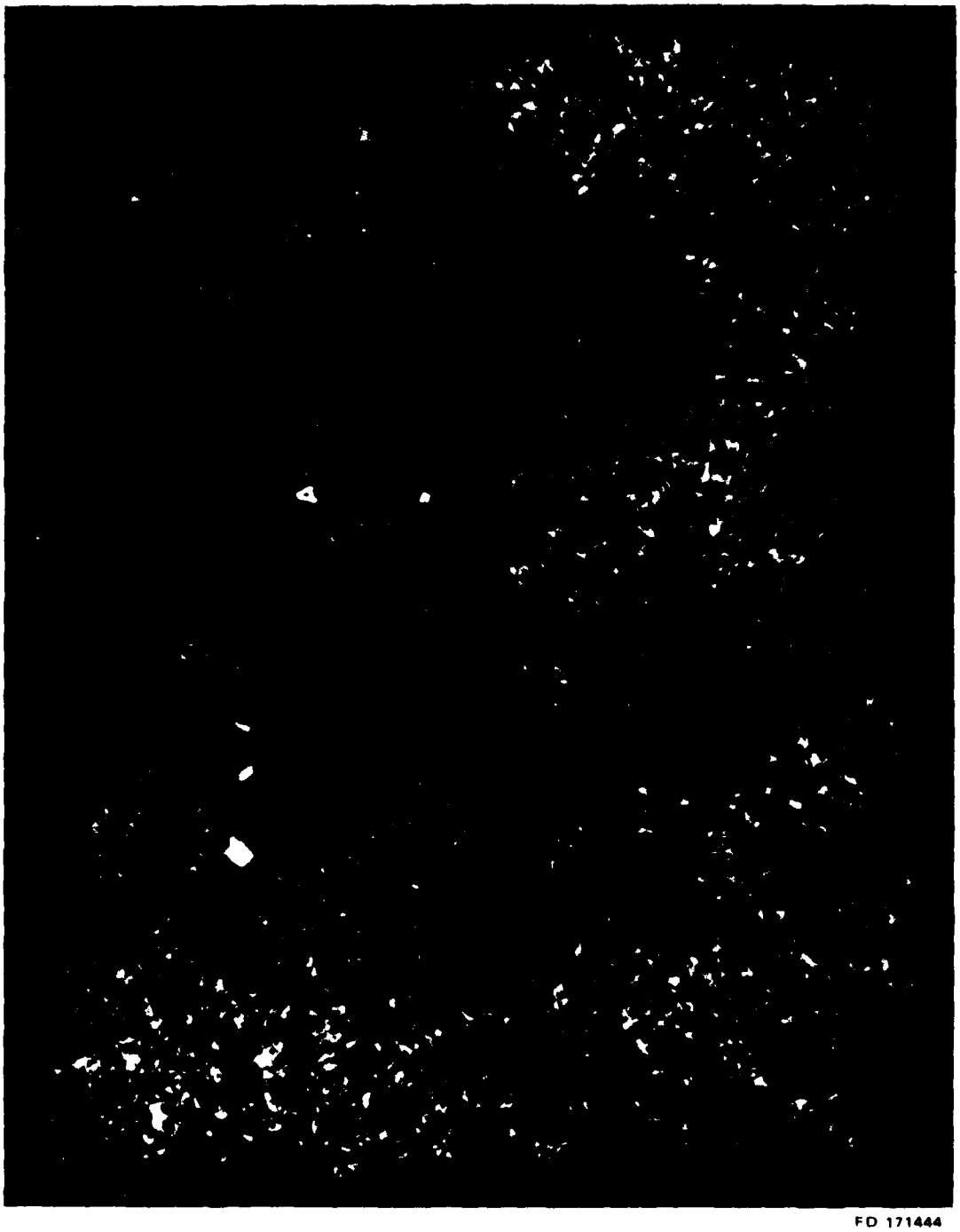


FD 171442

Figure 30. Photo Reduction of 11 × 14-in. AVCO Radiograph of Floats from Test Run No. 57, Lot 08

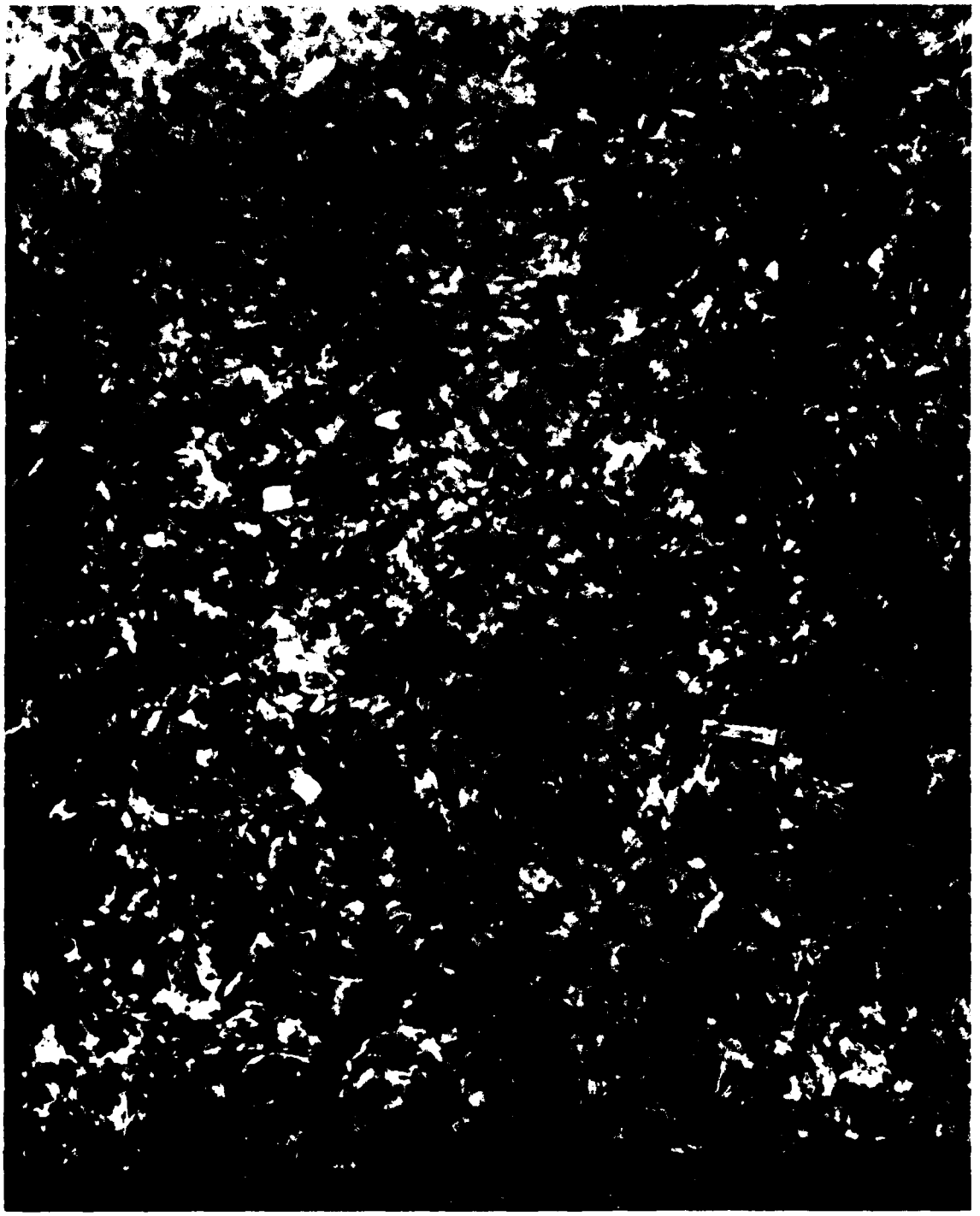
ED 171443

Figure 31. Photo Reduction of 11 × 14-in. AVCO Radiograph of Floats from Test Run No. 58, Lot 04



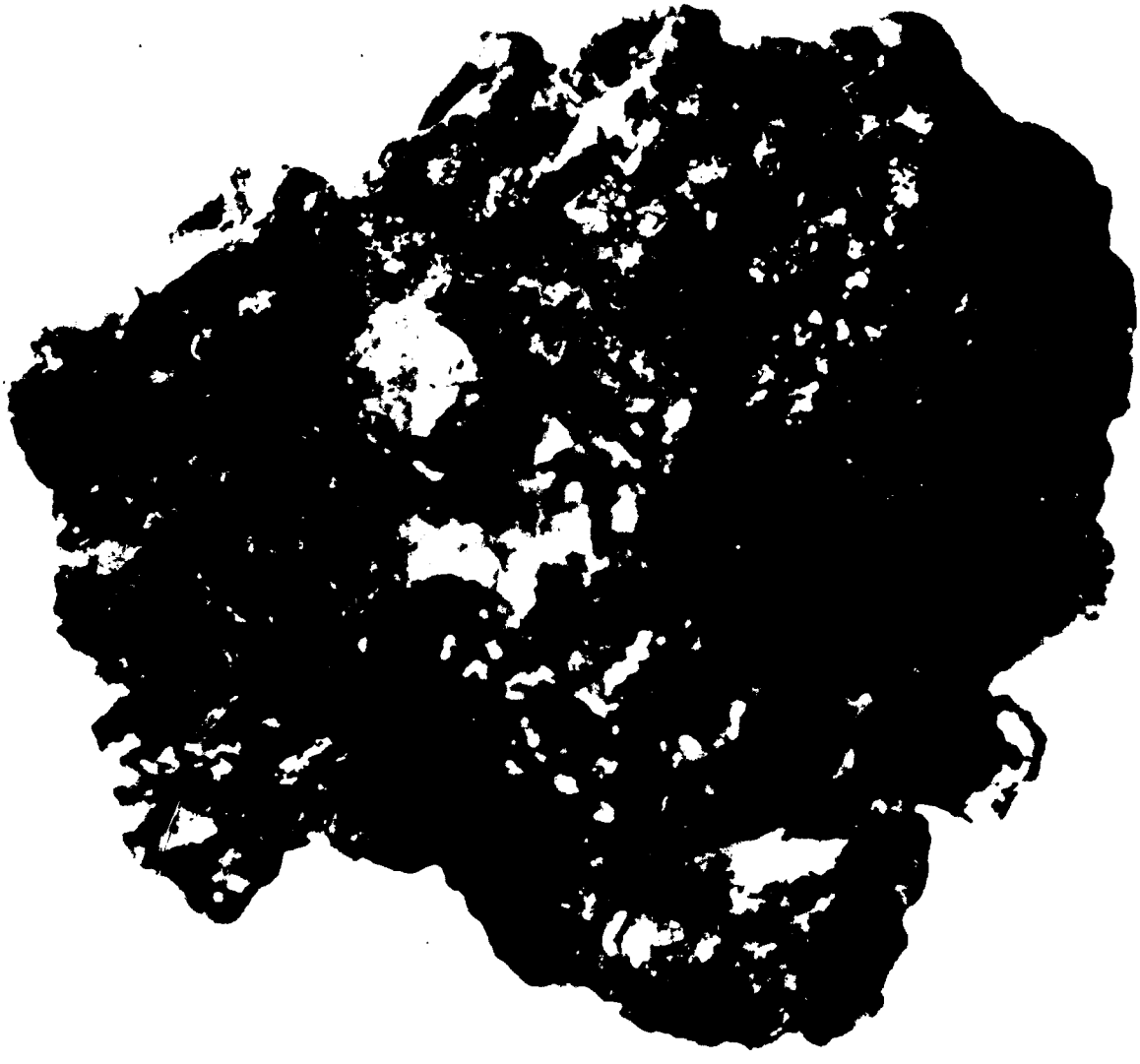
FD 171444

Figure 32. Photo Reduction of 11 × 14-in. AVCO Radiograph of Floats from Test Run 59, Lot 05



FD 171445

Figure 33. Photo Reduction of 11 × 14-in AVCO Radiograph of Floats from Test Run No. 60, [illegible]



FD 171446

Figure 34. Inclusion from Float of AVCO Run 58 (Dimensions: 0.250 × 0.200 × 0.150 in.)

FD 171447

Figure 35. Inclusion from Float of AVCO Run 59 (Dimensions: 0.150 × 0.150 × 0.085 in.)

The basic runs 6, 9 and 23 represented medium bulk density material. Process parameters for runs 6 and 9 approach optimum conditions. Run 23, however, was run at less than optimum conditions and also represented material which had never been screened. It was selected to assess the effect of melting on material which is highly contaminated with fine inclusions. The supplementary runs 58, 59, and 60 represented light, heavy and medium bulk density material, respectively, run at optimum process parameters.

The entire float products from each of these runs was forwarded to Teledyne Wah Chang for melting into six approximately 100-lb consumable melted ingots. These ingots were chemically analyzed and converted to plates which will be further evaluated by X-ray, chemical analysis, mechanical properties and microstructure. These ingots provided a means of thoroughly evaluating the entire separator product, rather than the 10% chip samples previously radiographed. The tendency of very small inclusions, i.e., < 0.030 in., to dissolve in the melt was also determined. The sinks from each of the six selected runs were forwarded to P&WA for button melting and chemical analysis.

FD 171448

Figure 36. Largest Inclusion from Floats of AVCO Run 60 (Dimensions: 0.487 × 0.312 × 0.178 in.)

(f) Light-Density Contaminant Separation Tests

Light density contaminants, such as aluminum, in moderate quantities, are not especially detrimental to Ti-6Al-4V, yet may produce chemistry deviations: other light contamination, e.g., wood chips, rag fragments, etc., are a nuisance during remelting. The capability of the ferrofluid to remove these low-density contaminants was, accordingly, demonstrated in three runs using natural scrap and scrap seeded with aluminum chips.

(1) Test Procedure

Titanium for the "lights" removal runs was selected from lots which had the "heavies" removed in prior runs, and which had been run under near-optimum conditions, to be representative of the practice which is likely to be followed in the future purification of titanium scrap turnings. Aluminum turnings for addition to the titanium turnings were made by AVCO by machining a solid piece of aluminum; thus it was assured that the "contamination" added was, in itself, uniform and free of other contaminants.

A series of runs of titanium turnings from Lot 01 were made at different density settings and the percentages of sinks versus floats were recorded. Similarly, samples of the aluminum turnings alone were run at different density settings. Figure 37 shows the relationship between the percent of floats and ferrofluid apparent density for the titanium and the aluminum respectively. Based on these calibration runs, 3.6 gm/cm^3 ferrofluid apparent density (34.4 in. of water) was selected as best meeting the criteria of maximum separation with minimum misclassification.

The initial test run (No. 269) was conducted utilizing a material lot which had been previously run to remove "heavies." "Lights" removal during this run provided a presumably contamination-free material. Pure Al turnings were subsequently added to this material to provide feedstock for a run (No. 270) in which "lights" were again removed. In order to demonstrate the removal of "lights" from as-received scrap, a second lot of material, which had been previously run to remove "heavies," was run (No. 271) to remove "lights."

(2) Test Results

Data from each of the three tests are listed below:

1. Test No. 269 (Removal of any possible "lights" before addition of aluminum)

Sample Source:	Lot 01, Floats from Test No. 1
Sample Weight:	107 lb
Contamination Added:	None
Ferrofluid Apparent Density:	3.62 gm/cm^3
Actual Feed:	220 lb/hr
Floats:	1.03 lb
Sinks:	98.0 lb
Removed from Separator:	8.2 lb (possibly contaminated by prior runs)

2. Test No. 270 (Removal of Al)

Sample Source:	Sinks from Test No. 269
Sample Weight:	98.0 lb
Contamination Added:	4.5 lb aluminum turnings
Total Sample:	102.5 lb

Ferrofluid Apparent Density:	3.62 gm/cm³
Actual Feed Rate:	325.5 lb/hr
Floats:	5.7 lb
Sinks:	97.2 lb

3. Test No. 271 ("Lights" removal from "as-is" Ti turnings)

Sample Source:	Lot 01, Floats from Test No. 17
Sample Weight:	81.9 lb
Contamination Added:	None
Ferrofluid Apparent Density:	3.62 gm/cm³
Actual Feed Rate:	239 lb/hr
Floats:	0.6 lb
Sinks:	81.3 lb

A 32 lb sample of the sinks from Test No. 270 was hand-sorted to identify any misclassified aluminum which might be present. A total of 1.8 gr of Al was found manually indicating a weight concentration of 0.012%.

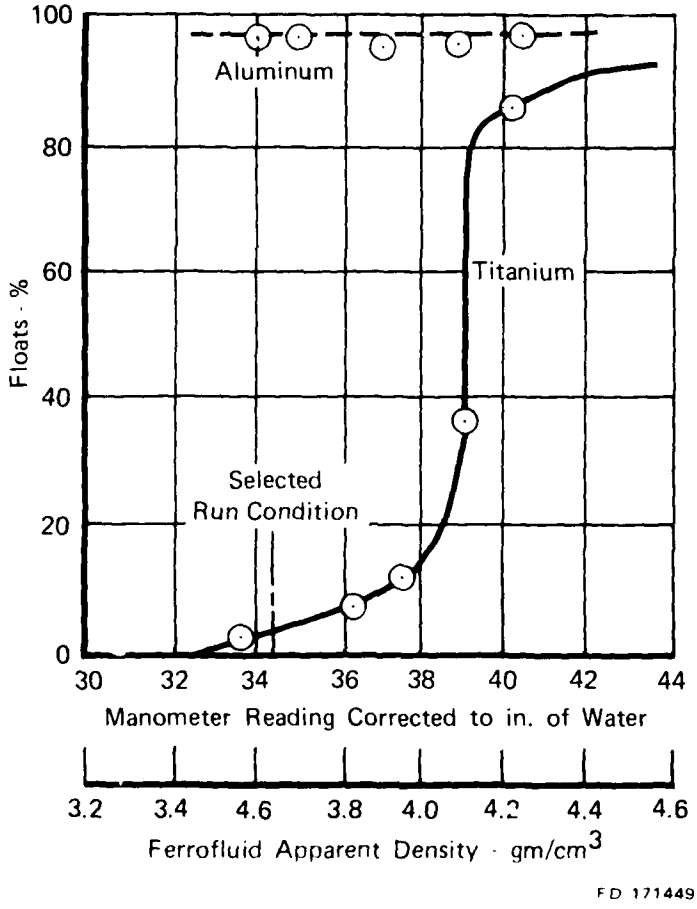


Figure 37 Percentage of Floats from Titanium Turnings and Aluminum Turnings vs Ferrofluid Apparent Density — Preliminary Test — (Lot 01) (FF 1224) (M ~ 205 Gauss)

Preliminary analysis of test data indicates: (1) the "as-is" samples had little, if any, Al and other "lights" present (floats in the proportion of 1.04% in Test No. 269, and 0.7% in Test No. 271 likely reflect predominantly misclassification of Ti, rather than "lights"), and (2) the Al purposely added in Run No. 270 appears to have been almost completely removed (apparently, about 1.2 lb, or about 1% of the Ti went out with the floats, and manual examination of a portion of the sinks indicates only a trace of free Al was left in the sinks).

Chemical analyses of the floats and sinks from tests 270 and 271 was conducted to confirm that the sinks meet Ti-6Al-4V specification and the floats are Al-rich.

g. Separation Test Program (Superalloy)

A separation test program was carried out to establish the manufacturing conditions required to remove foreign fragments from superalloy chips by ferrofluid density separation. The object of these tests was to remove Ti alloy turnings (Ti-5Al-2.5Sn, Lot 08) and lead shot from Waspaloy chips (Lot 09) as supplied by Frankel Co. The general operating conditions established to process Ti chips were used in the superalloy tests, with exceptions as noted in the following paragraphs.

(1) Test Procedure

The ferrofluid separator was operated at a higher apparent density than for the Ti chips because of the higher density of the Waspaloy. As a result, a stronger ferrofluid (AVCO Ferrofluid No. 1248) was used, having a magnetization of 500 gauss, a true density of 1.405 gm/cm^3 and a viscosity of 24 cp. This kerosene-base ferrofluid differs from the Ferrofluid 1224 used in the Ti separation tests only in the concentration of magnetic colloid in suspension.

The purification of the Waspaloy required two separations. The low density contaminants were removed as floats by operating the separator at a density lower than that of the superalloy being processed. The sinks of this separation, which consisted of the superalloy and denser contaminants, were then reprocessed through a ferrofluid separator operating at a density higher than the superalloy. The dense contaminants were in the sinks, and the purified superalloy product was recovered as floats.

The feed mixture was processed through the complete AVCO process, e.g., it was screened at 10 mesh to remove fines and passed over a magnetic drum to remove magnetics. It was found that screening resulted in the removal of about 7% of the as-received material; less than 0.4% was removed by the magnetic drum.

(2) Test Results

Preliminary tests were carried out with small lots of uncontaminated Waspaloy chips (Lot 09) to establish the appropriate range of operating conditions for the optimization runs that would be carried out with larger lots of contaminated material. In these tests, the apparent density of the ferrofluid was varied over a range corresponding to pressure readouts of from 60 to 79 in. H_2O, or an apparent ferrofluid density of 7.6 gm/cm^3 and 10.0 gm/cm^3, respectively as shown previously in Figure 12. The feed rates ranged from 270 to 1230 lb/hr.

Figure 38 shows the relation between the percentage of material floated and ferrofluid apparent density. Essentially all the material sank at an apparent ferrofluid density of 7.6 gm/cm^3 or less, and approximately 85 to 95% of the material floated at a density of 8.9 gm/cm^3. The amount of Waspaloy chips in the floats increased rapidly, with increasing densities between 7.6 and 8.9 gm/cm^3. The neutral buoyancy point for these chips corresponds to an operating pressure of slightly less than 66 in. H_2O. At this pressure, half of the chips floated and half the chips sank (density of Waspaloy = 8.2 gm/cm^3).

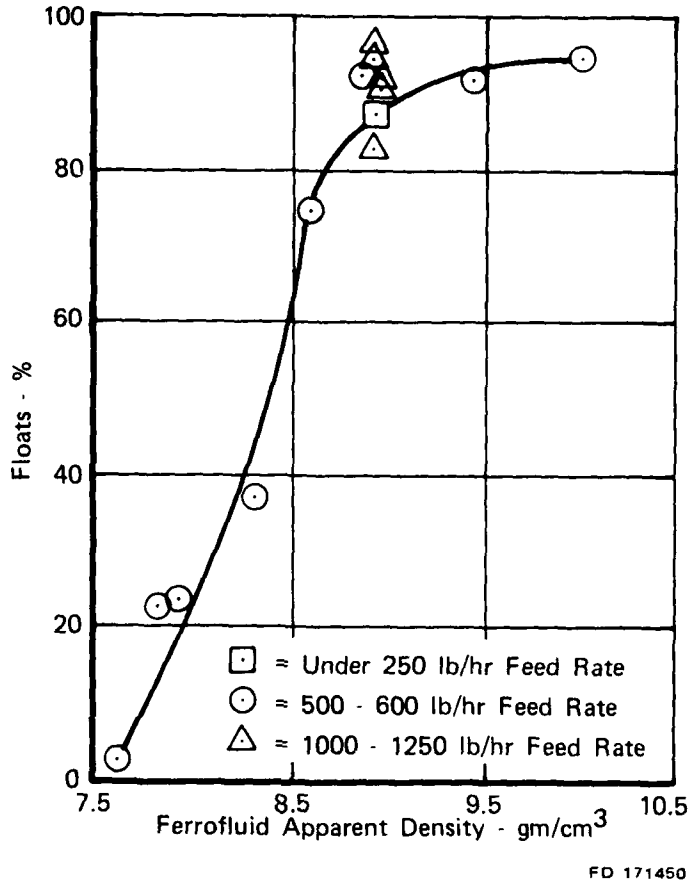


Figure 38. Preliminary Waspaloy Test Runs of AVCO Ferrofluid Separator — Percent of Material Floated vs Apparent Density

Figure 39 shows the effect of feed rate on the classification of the Waspaloy chips at an operating pressure of 70 in. H_2O (8.9 gm/cm³). There was no significant effect of feed rate over the range of 270 to 1230 lb/hr.

Based on these tests, the following operating conditions were selected for further evaluation:

Removal of low-density contaminants

Apparent ferrofluid densities	=	7.2 and 7.6 gm/cm³
Feed rates	=	500 to 600 and 900 to 1000 lb/hr

Removal of high-density contaminants

Apparent ferrofluid densities	=	8.9 and 9.4 gm/cm³
Feed rates	=	500 to 600 and 900 to 1000 lb/hr

A total of thirty test runs were performed in accordance with the summary test sequence presented in matrix form in Table 18. This sequence may be contrasted with the original test plan in Table 8.

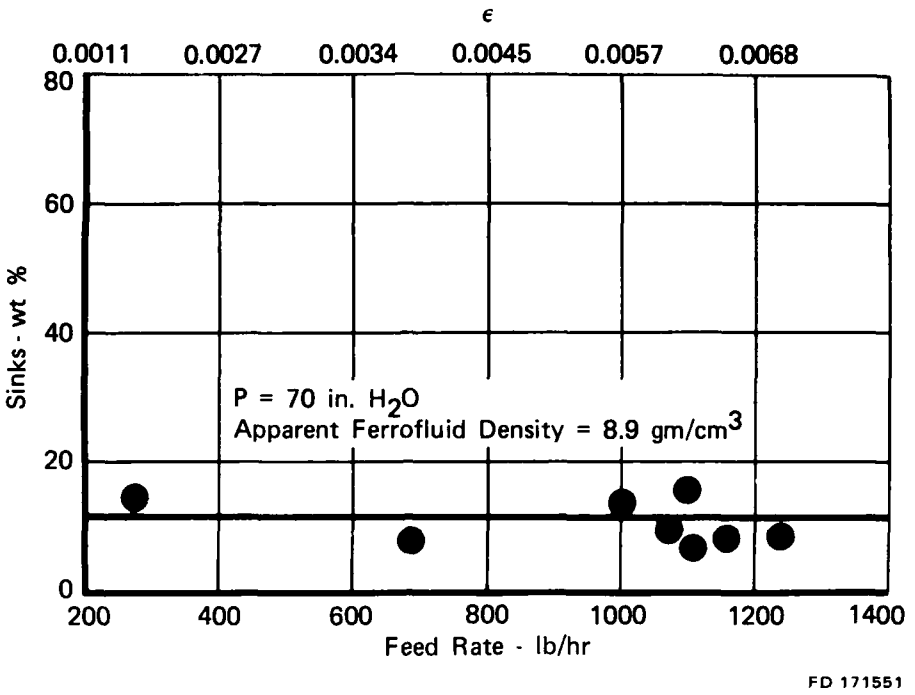


Figure 39. Preliminary Waspaloy Test Runs of AVCO Ferrofluid Separator — Percentage of Sinks vs Feed Rate

TABLE 18
AVCO FERROFLUID SEPARATION SYSTEM TESTS — WASPALOY

OBJECTIVE

Determine effects of apparent density and processing rate on separation efficiency.

SCRAP PROPERTIES

1. Crushed, degreased and dry turnings.
2. Size Range $-3 + \frac{1}{8}$ in.
3. Bulk Density — Typical Median Range

EXPERIMENTS

Apparent Densities in gm/cm³		Processing Rate (lb/hr)		
Removal of Light Contaminants	Removal of Dense Contaminants	400 to 700	700 to 1000	1000
7.9	8.9	101*		
7.6	8.9	109 ABCD		
7.2	8.9	103 AB**		102 AB
7.2	9.4	107 ABCD	108 ABCD	
7.6	9.4	104 AB**	105 AB	106 B
			106 A	

*Separation of Uncontaminated Sample
**Repeat test

A 102 lb sample of screened Waspaloy was used to carry out Run 101. An additional 615 lb of screened Waspaloy was mixed with 30 lb each of Ti-5Al-2.5Sn chips (Lot 08) and lead shot to provide the feed material for Runs 102 to 106 which were single-pass runs at the various operating conditions. Subsequently the remaining 345 lb of screened Waspaloy were mixed with 15 lb each of the two contaminants to provide the feed material for Runs 107 to 110 which were double-pass runs. Figure 41 shows the material balance summary.

The material balances obtained in these tests are summarized in Tables 19, 20 and 21 and include data for the uncontaminated material (single- and double-pass runs). X-ray examinations of the separated samples were not made since it was more economical to obtain lead content as part of the elemental analysis of the melted samples. Consequently, 10 lb samples of all products were sent to P&WA for selective chemical analysis.

(a) Discussion of Results

A thorough assessment of the data requires a consideration of chemical analysis results, although some insight into the separation can be reached by analysis of the yield data.

Purification of Uncontaminated Material

The preliminary tests and Runs 101A and 101B yield information on the processing of uncontaminated material. Very little material floated at apparent ferrofluid densities of 7.6 gm/cm^3 or less, i.e., 0.6 gm/cm^3 or more below the density of Waspaloy. Conversely, a small but significant amount of material sank when the separator was operated at apparent densities significantly higher than the Waspaloy density of 8.2 gm/cm^3. At an apparent density of 10.0 gm/cm^3, 6.8% of the material went into the sinks. There appears to be a response lag on the floats, since not all the material floats even when the separator is operated at densities higher (by about 1.3 gm/cm^3) than that of the scrap. These results indicate that the as-received Waspaloy chips contained a very small impurity concentration of lower density than the Waspaloy and a large high density contaminant concentration. One possible explanation for the response lag on the floats is interference by the sinking dense contaminants. Another interpretation is that some fraction of the Waspaloy chips are slightly more magnetic than the bulk of the chips due to selective work hardening during their prior history. As a result, these chips may simulate the behavior of high density chips.

The uncontaminated test (Run 101A) was run at too high an apparent density of the separating medium. The result was floating an unacceptable large amount of Waspaloy.

This confirms the preliminary test results which show over 20% of material floating at an apparent density of 7.9 gm/cm^3, whereas at an apparent density of 7.6 gm/cm^3 only 1.5% of the uncontaminated (as received) material floated in a preliminary run. As a result, all tests with contaminated feed were run at an apparent density of 7.2 or 7.6 gm/cm^3 to minimize misclassification of the Waspaloy.

As shown in Figure 40, there was little effect of feed rate on the classification of the as-received product. This is not surprising in that the calculated values of ϵ, utilizing the density difference between the operating density and that of Waspaloy, were much lower than the initial value of 0.015. For the runs presented in Figure 40, the values of ϵ ranged from 0.001 to 0.007. These values are much lower than the values that would have been observed for Ti alloy chips processed at these mass feed rates, mostly because the Waspaloy chips have a much higher bulk density.

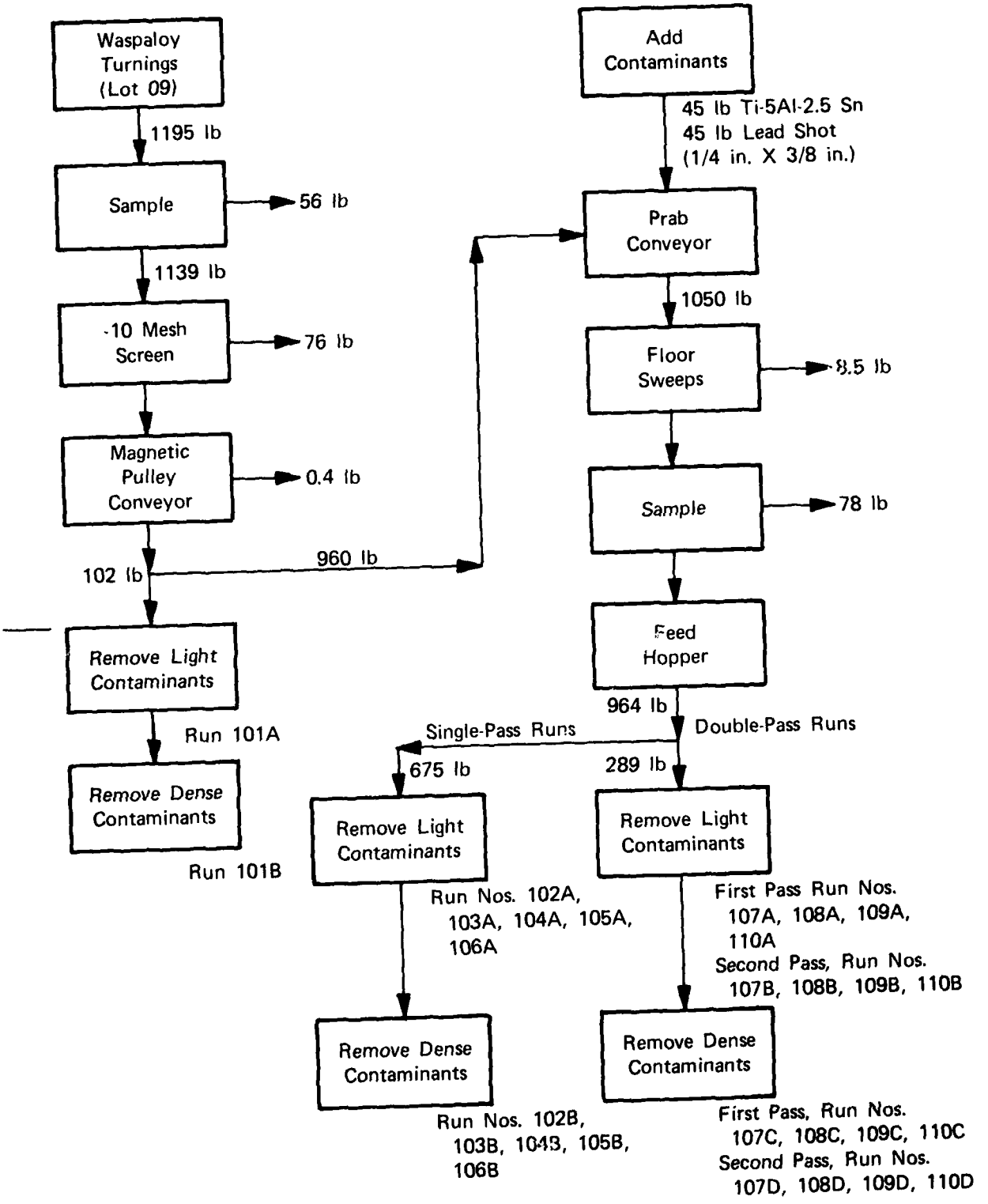


Figure 40. AVCO Ferrofluid Separator Waspaloy Test Runs-Summary Material Balance

TABLE 19
AVCO FERROFLUID SEPARATION TESTS — DATA SUMMARY FOR SINGLE-PASS RUNS WITH AS-RECEIVED WASPALOY CHIPS

Run No.	*101A Lights Removal*	*101B Heavies Removal*	*Preliminary Test 1*	*13*	*12*
Feed Rate lb/hr	506	450	610	585	580
Operating Density gm/cm³	7.9	8.9	7.6	7.9	8.7
Operating Pressure in. H_2O	62	70	60	62	70
Wt Floats, lb	22.6	72.0	0.6	7.3	33.4
Wt Sinks, lb	80.3	8.2	37.2	28.2	2.2
Total Weight, lb	102.6	80.2	37.8	35.5	35.6
Weight % Sinks	78.2	10.2	98.5	79.4	6.2
Product Recovery*, %		70.5			

*Product Recovery = $\frac{\text{Weight of Floats Pass B}}{\text{Weight of Feed Pass A}}$

TABLE 20
AVCO FERROFLUID SEPARATION TESTS OF WASPALOY MATERIAL — DATA SUMMARY FOR SINGLE-PASS TESTS OF CONTAMINATED MATERIAL

Run No.	*102A Lights Removal*	*102B Heavies Removal*	*103A Lights Removal*	*103B Heavies Removal*	*103B'* Heavies Removal Rerun*	*104A Lights Removal*	*104B Heavies Removal*	*104B'* Heavies Removal Rerun*	*105A Lights Removal*	*105B Heavies Removal*	*106A Lights Removal*	*106B Heavies Removal*
Feed Rate lb/hr	970	1040	605	830	575	573	825	575	762	906	900	1260
Operating Pressure, in. H_2O	57	70	57	70	70	60	74	74	60	74	60	74
Apparent Density gm/cm³	7.2	8.9	7.2	8.9	8.9	7.6	9.4	9.4	7.6	9.4	7.6	9.4
Wt Floats lb	5.1	147.2	2.9	89	93.9	9.1	86.2	83.6	11.0	68.0	33.4	43.5
Wt Sinks lb	175.6	21.3	119.6	21.2	15.9	105.6	11.8	12.0	88.0	12.6	66.6	18.6
Total lb	180.7	168.5	122.5	110.2	109.8	114.7	98.0	95.6	99.0	80.6	100.0	62.1
Wt % Sinks	97.5	12.6	97.8	19.2	14.6	92.3	12.1	14.3	89.0	15.7	66	30
Product Recovery %**	81.5		72.8		76.6	75.5		72.8	68.5		43.5	

*Repeat of Previous Test

**Product Recovery = $\frac{\text{Weight of Floats Pass B}}{\text{Weight of Feed Pass A}}$ (100)

		lb	Wt %
Feed Material	Ti-5Al-2.5Sn	30	4.65
	Lead Shot	30	4.65
	Waspaloy	615	Balance
	Total	645	100.0%

TABLE 21
AVCO FERROFLUID SEPARATION TEST OF WASPALOY MATERIAL — DATA SUMMARY FOR DOUBLE-PASS RUNS

Run No.	*107A Lights*	*107B Removal*	*107C Heavies*	*107D Removal*	*108A Lights*	*108B Removal*	*108C Heavies*	*108D Removal*
Pass	1	2	1	2	1	2	1	2
Feed Rate lb/hr	527.0	510.0	518.0	595.0	850.0	950.0	1000.0	880.0
Operating Pressure, in. H_2O	57.0	57.0	74.0	74.0	57.0	57.0	74.0	74.0
Apparent Density gm/cm³	7.2	7.2	9.4	9.4	7.2	7.2	9.4	9.4
Wt Floats lb	0.9	0.6	51.7	45.3	1.0	0.6	56.8	51.7
Floats and Sinks								
Wt Sinks lb	63.8	62.3	8.8	5.08	69.0	65.3	7.5	4.8
Total lb	64.7	62.9	60.5	50.4	70.0	65.9	64.3	56.5

	Wt. lb	Wt. %	Wt. lb	Wt. %
Weight Product, lb and Wt %	45.3	74.7	51.7	78.8
Weight by Product Floats, lb and Wt %	1.5	2.5	1.6	2.4
Weight by Product Sinks, lb and Wt %	13.8	22.8	12.3	18.8
Total Accountable Products, lb	60.6	100.0	65.6	100.0

Run No.	*109A Lights*	*109B Removal*	*109C Heavies*	*109D Removal*	*110A Lights*	*110B Removal*	*110C Heavies*	*110D Removal*
Pass	1	2	1	2	1	2	1	2
Feed Rate lb/hr	520.0	475.0	450.0	570.0	880.0	960.0	960.0	960.0
Operating Pressure, in. H_2O	60.0	60.0	70.0	70.0	60.0	60.0	70.0	70.0
Apparent Density gm/cm³	7.6	7.6	8.9	8.9	7.6	7.6	8.9	8.9
Wt Floats lb	3.0	1.5	46.9	39.2	4.6	1.7	54.2	47.3
Wt Sinks lb	61.2	58.7	10.2	7.4	68.0	64.8	9.5	6.6
Total lb, Floats and Sinks	64.2	60.2	57.1	46.6	72.6	66.5	63.7	54.9

	Wt. lb	Wt. %	Wt. lb	Wt. %
Weight Products	39.2	64.0	47.3	68.0
Weight by Product Floats	4.5	7.4	6.3	9.0
Weight by Product Sinks	17.5	28.6	16.0	23.0
Total Accountable Products	61.2	100.0	69.6	100.0

Feed Mixture	
Waspaloy	345 lb
Ti-5Al-2.5Sn	15 lb
Lead	15 lb
Total	375 lb

Purification of Contaminated Material

For the single-pass runs, 102 though 106, it is possible to compare the total weight of the floats of the A runs, in which low density contaminants are removed, with the weight of Ti alloy scrap added to the master feed batch. In a similar fashion, it is possible to compare the weight of the sinks of the B runs, in which the high density contaminants are removed, with the weight of the lead shot added to master feed batch. It should be recalled that 30 lb each of titanium alloy and lead were added to 615 lb of Waspaloy, resulting in 675 lb of a feed mixture that contained, on the average, 4.45% of each contaminant.

The total weight of the floats from Runs 102A through 106A was 61.5 lb. This is roughly twice the weight of the added Ti indicating a greater degree of misclassification in these runs than was found in the floats of preliminary runs carried out at apparent densities of 7.6 gm/cm³; i.e., 31.5 lb/615 lb or about 5% appears to be misclassified, or five times as much as before. The weight fraction of the floats was much lower in the case of Runs 102A and 103A than for Runs 104A to 106A.

The higher percentage of floats in the later runs may be partially accounted for by segregation of the Ti chips in the feed hopper. There were fewer Ti chips in the feed to the first Run 102, which was drawn from material originally present at the bottom of the hopper, than in the feed to the last Run 106, which was drawn from material originally at the top of the chip pile in the hopper. In addition the weight of floats from Run 106A was larger than the 30 lb of Ti chips added to the original feed mixture.

The total weight of the sinks from Runs 102B to 106B was 80.4 lb. This is roughly 2.7 times the weight of lead shot initially added. The ratio of the weight of the sinks less the weight of the added lead to the weight of the Waspaloy chips initially added to the feed is equal to 50.4/615 or 8.2%. This overall percentage of Waspaloy chips in the sinks agrees with percentage of sinks found in the preliminary tests with uncontaminated Waspaloy at apparent densities of 8.9 gm/cm^3 and 9.4 gm/cm^3. There was no systematic increase or decrease in the weight of the sinks of progressive runs, indicating that there was no severe segregation of the lead shot in the Waspaloy. However, the weight fraction sinks for the last Run 106B was much higher than for the other runs. While this result might have been due to operation at a higher feed rate (1,260 lb/hr) than was used in the other runs, it may also have been due to the presence of significantly more lead shot.

The same general observations apply to Runs 107 to 110, where the scrap was subjected to four passes, two floats and two sinks. The total weight of the floats from Runs 107B to 110B was 17.9 lb. This number is only slightly higher than the weight of Ti chips (15 lb) added to the feed mixture, indicating that little (i.e. 2.9/345 = 0.8%) Waspaloy has been misclassified as was found in the preliminary runs operated at the same pressures. In addition, it was noted that the weight of the floats increases with progressive runs and that the weight of the by-product floats for the last Run (110) was four times the weight of the by-product floats for the first Run (107). The possible segregation of the titanium chips in the feed hopper was also likely for these runs, although less severe, most probably because the feed batch was smaller.

The total weight of the sinks from Runs 107D to 110D was 65.6 lb. Subtracting the 15 lb of lead added to the feed produces a nominal misclassification of 50.6/345 or about 14.5% of the feed Waspaloy. This is higher than the average value found for the one pass runs. The significance of these results will depend on the analysis.

The weight of the input to a given run is slightly higher than the combined weight of the floats and sinks from that run (Table 21). This was because of a small residual amount of material left in the separator. This amount is noncumulative and would not be of significance in processing large quantities of scrap.

The purified products from Runs 102 and 104 were sent to Teledyne-ALLVAC in Monroe, N. C., for melting into ingots. At the same time, 16 samples from various runs were sent to P&WA for selective chemical analysis.

(b) Conclusions

1. In the various artificially contaminated runs, Ti chips were definitely present in the by-product floats, and lead balls were present in the by-product sinks. The principal product looked clean.

2. The operation of the separator was smooth due to the high bulk density of the chips.

3. The concentration of low density contaminants in the as-received scrap was low. The presence of higher density contaminants is likely.

4. There was some indication that the composition of the feed varied for different runs because of segregation of Ti scrap in the feed hopper. Because of this possibility, it was not possible to definitively sort the effects of feed rate, operating pressure and number of passes on the purification process. It appears that overall conclusions drawn from the Ti chip purification test program would also be applicable to the superalloy tests.

2. Frankel Company Fluidized Bed Process

An improved method of dense media separation using a dry fluidized bed was developed by the Frankel Company. U. S. Patent 3,610,415 describes this process for the separation of dense particulate metals from Ti turnings and chips. As originally conceived, the process used a fluidized bed of lead shot capable of producing effective media densities up to 6.0 gm/cm^3. In pilot scale experiments, the separation of tungsten carbide tool bits from titanium turnings was demonstrated. The process is not capable of separating denser metals, such as lead alloys, from superalloys. To avoid the potential difficulties with the use of a lead media, an alternative method is utilized to separate titanium from heavier particles. A thick bed of Ti particles is fluidized with air in a conventional manner. Scrap material is continually introduced while being removed from overflow discharge regions at the top of the bed. When heavier particles sink to the bottom of the bed, they are removed laterally by vibratory conveyance.

a. Program Plan Details (Phase I)

Frankel Co. conducted a series of test separation experiments to evaluate the effect of scrap variables and process variables associated with the fluidized bed separation process. Contaminants similar in type size and concentration to those described for the ferrofluid test program were added to the scrap. Test lots of 100 lb each were processed under varying conditions (Table 22). Processing included crushing, cleaning, magnetic separation, screening, fluidized bed processing, X-ray/chemistry checks and ingot melt. Ingots were evaluated as previously described in the *AVCO ferrofluid separation program plan*.

TABLE 22
FRANKEL FLUIDIZED BED SEPARATION SYSTEM PROPOSED TEST VARIABLES

Run No.	*Alloy*	*Particle Size (in.)*	*Bulk Density (lb/cu ft)*	*Method of Cleaning*	*Contaminants**	*Fluidized Bed Process*
1	6Al-4V	- ¼ + 8 Mesh	20 to 30	Detergent	WC	1
2	6Al-4V	- ¼ + 8 Mesh	20 to 30	Detergent	WC	3
3	6Al-4V	- ¼ + ⅛	20 to 30	Detergent	WC	1
4	6Al-4V	- ⅛ + 8 Mesh	20 to 30	Detergent	WC	1
5	6Al-4V	¼ + ⅛	20 to 30	Detergent	WC	3
6	6Al-4V	¼ + ⅛	20 to 30	Detergent	Wasp + SS	1
7	6Al-4V	¼ + ⅛	20 to 30	Detergent	Wasp + SS	3
8	6Al-4V	¼ + ⅛	10 to 15	Detergent	WC	1
9	6Al-4V	¼ + ⅛	35 to 40	Detergent	WC	1
10	8Al-1Mo-1V	¼ + ⅛	35 to 40	Detergent	WC	1

*WC — Tungsten carbide tool chips
Wasp — Waspaloy
SS — Stainless Steel

b. Frankel Co. Fluidized Bed Density Separation Process

(1) System Technique

A method of dense media separation utilizing a dry fluidized bed was developed by the Frankel Co. (U. S. Patent 3610415). This process is capable of separating dense particulate matter from Ti chips, and with modification has also been used to separate Ti chips from nickel base alloys.

Figure 41 is a schematic layout of the Frankel Co. fluidized bed separation system. The unit is designed to feed contaminated chips to a vibrating screen. By operator control of chip feed rate, airflow, screen movement, and screen angle, these chips are separated into heavy and light components. Details of the operation are described in the following paragraphs.

The screen is adjusted at an angle so that one end is higher than the other, i.e., an inclined plane. Initial operation requires establishment of a chip-to-screen feed rate which will match the capability of the screen to "transport" the chips, followed by actuation of airflow through the screen. With continued feeding, a wedge-shaped chip bed is built up, with a lip at the lower end of the screen providing the base to the wedge. An output chute is located at the base of the inclined plane and a reject chute is located at the top of the inclined plane. Airflow through the screen is adjusted to provide sufficient air to fluidize the bed of chips on the screen. Lighter Ti chips will float on the top of the bed and be carried down the inclined plane by gravity, overflowing the lip into the output chute. Heavier contaminants will settle to the bottom of the bed, come in contact with the screen and be carried up the screen to the reject chute. Some of the larger, dense particles, especially if they have a smooth surface, will not be carried by the screen to the reject chute and will remain near the bottom of the bed. If airflow through the screen is excessive most or all material will overflow the lip and exit at the output chute, or, in an extreme condition, be blown out of the unit and scattered. If airflow through the screen is inadequate, the reject rate will be excessive. In the extreme case of no airflow, all material will be carried to the reject chute.

No airflow is used when starting the unit. Following activation of the airflow and the establishment of a fluidized bed, airflow is adjusted to establish a reasonable rejection rate; however, rejection of noncontaminated titanium chips will always occur. Material which has been removed through either the output chute or reject chute up to this point is returned to the hopper and the actual separation run is initiated.

The selection of a rejection rate is based on operator judgment and experience. It is necessary to reject a minimum amount of noncontaminated material to assure removal of all contaminants. A reject rate should correspond with the anticipated difficulty of separation, e.g., a high rate for separation of fine chips. In general, the reject rate can vary from 0.25 to 10%, depending on the size, shape, and density of the feed material and contaminants. When the run ends, the airflow is turned off and the bed material (including accumulated contaminants) is carried out the reject chute.

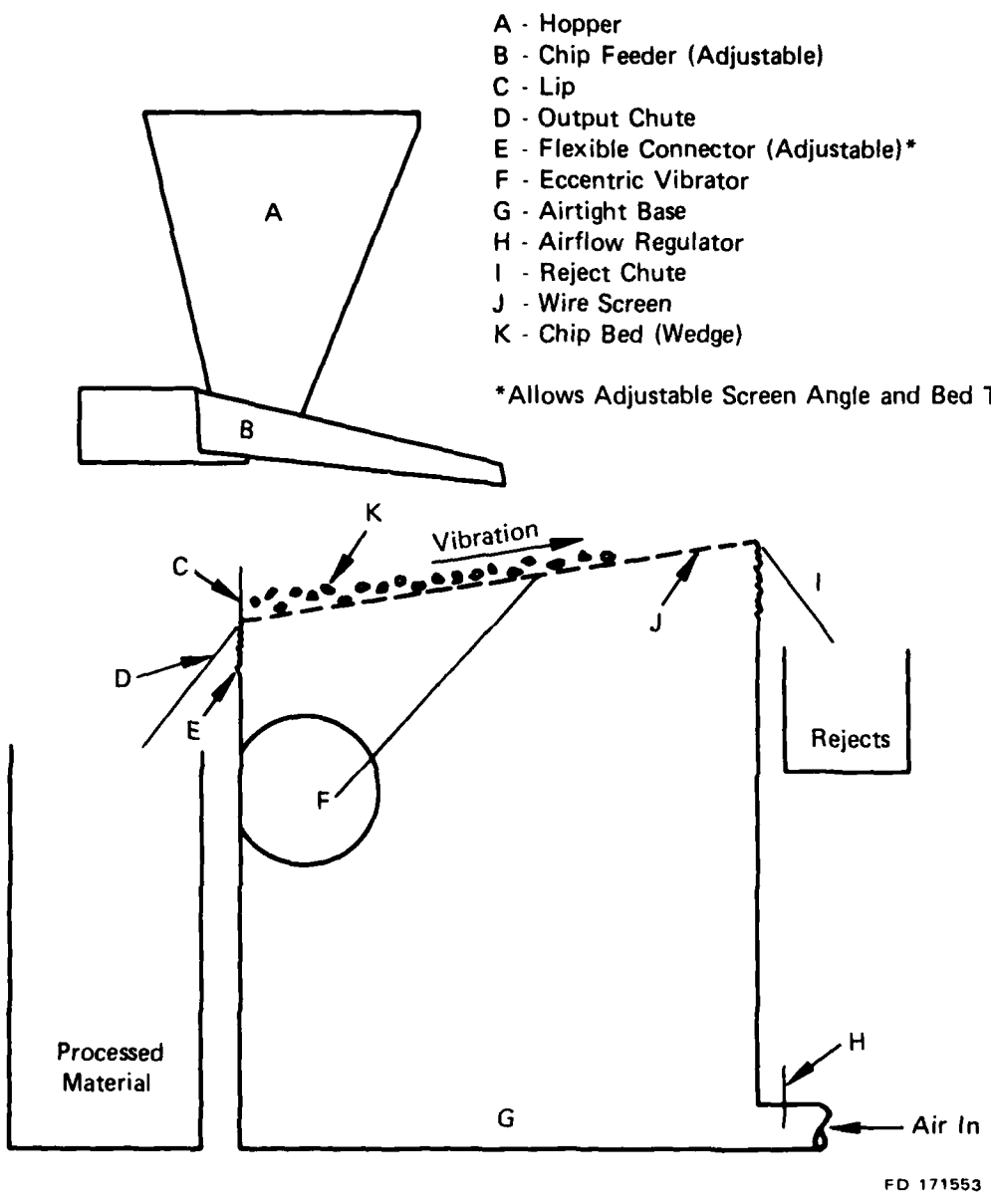


Figure 41. Schematic of Frankel Fluidized Bed Unit

(2) Separation Test Program

(a) Test Procedures

Frankel Co. conducted a series of ten fluidized bed test runs. With a few exceptions, the program adhered to previously published plans (Table 22). High density Ti-5Al-2.5Sn chips were substituted for Ti-8Al-1Mo-1V chips in the final run because of greater availability of the 5Al-2.5Sn alloy, and therefore, the high density 6Al-4V run was deleted. In addition, only Waspaloy, rather than Waspaloy and stainless steel, was added as a contaminant for Run No. 9 to demonstrate removal of medium density contaminants. It was noted that Waspaloy and stainless steel have similar densities, and that a more positive evaluation could be made for Waspaloy in a reclaimed product, since the 6Al-4V composition does not normally have a nickel content. Two of the runs included an addition of W, Mo and Ta, along with the planned WC. Test parameters are summarized in Table 23.

TABLE 23
FRANKEL FLUIDIZED BED ACTUAL TEST PARAMETERS

Run No.	*Alloy*	*Bulk Density (lb/ft³)*	*Chip Size Range (in.)*	*Contaminate*	*No. of Passes*	*Reject Rate (%)*	*Feed Rate (lb/min)*
1	6Al-4V	25	+ 10 Mesh/ [illegible]	WC	1	5	5
2	6Al-4V	25	+ 10 Mesh/ [illegible]	WC	3	5	5
3	6Al-4V	25	+ 10 Mesh/ [illegible]	WC	1	7	5
4	6Al-4V	25	+ [illegible]/ [illegible]	WC	1	2[illegible]	5
5	6Al-4V	25	+ [illegible]/ [illegible]	WC	3	1	7
6	6Al-4V	25	+ [illegible]/ [illegible]	WC, W, Mo, Ta	3	1[illegible]	5
7	6Al-4V	25	+ [illegible]/ [illegible]	WC, W, Mo, Ta	1	2	5
8	6Al-4V	11	+ [illegible]/ [illegible]	WC	1	[illegible]	3
9	6Al-4V	25	+ [illegible]/ [illegible]	Waspaloy (4%)	1	10	5
10	5Al-2.5Sn	41	+ [illegible]/ [illegible]	WC	1	2	5

Ten equal assortments of tungsten carbide particles were prepared, each consisting of eight large pieces (+1/2 in.), 20 medium size pieces (−1/2/+1/4 in.), 20 small pieces (−1/4 in./+10 mesh) and 20 very small pieces (−10/+30). Figure 42 shows one of these assortments utilized for each 100 lb test run, except for Run No. 9 (Waspaloy-contaminated run). All tests in this series were run with a 10 deg screen angle and a 1.5 in. thick bed (lip height). Details of each test are noted in the following paragraphs.

Test Run No. 1

One hundred pounds of medium bulk density (25 lb/ft³) 6Al-4V titanium chips were mixed with one assortment of tungsten carbide particles. The contaminated chips were loaded into a hopper and fed onto a fluidized bed unit with a vibratory feeder at the rate of 5 lb/min. Airflow was adjusted on the unit to obtain a 5% rejection rate.

Test Run No. 2

A second 100 lb lot of contaminated medium density 6Al-4V titanium chips were processed with the same test parameters as Test No. 1 (5 lb/min feed rate and 5% rejection rate). After completion of the run, the unrejected portion was replaced in the hopper and the run repeated without changing any of the controls. After completion of this run, the unrejected portion was re-run for a third time in the unit, again without changing any of the controls. The rejects from the three passes were kept separate.

Figure 42. Typical Tungsten Carbide Tool Bit Fragments Added to Each Test Run by Frankel Co.

In preparation for Tests 3 and 4, medium density 6Al-4V titanium chips (200 lb) were mixed with two assortments of tungsten carbide particles. After mixing the batch, the entire 200 lb were screened on a $\frac{1}{4}$ in. screen, resulting in approximately 100 lb of $\frac{3}{8}$ in. and 100 lb of $+\frac{3}{8}$ in. portions. It was noted that a $\frac{1}{4}$ in. screen will pass approximately $\frac{3}{8}$ in. size titanium chips due to the strongly elongated shape of crushed turnings.

Test Run No. 3

The $\frac{3}{8}$ in. portion of chips was processed at a feed rate of 5 lb/min, but required a rejection rate of 7%, due to the smaller particle size.

Test Run No. 4

Due to the larger particle size, the $+\frac{3}{8}$ in. portion permitted an airflow adjustment obtaining only a $2\frac{1}{2}$% rejection rate using a feed rate of 5 lb/min. The remaining test runs were all screened to remove material smaller than $\frac{3}{8}$ in. after the addition of contaminants. The removal of these fines was expected to increase the potential for getting a high quality reclamation product.

Test Run No. 5

Test run No. 5 was performed with medium bulk density 6Al-4V Ti chips ($+\frac{3}{8}$ in.), which were processed at a feed rate of 5 lb/min with airflow adjusted to give 1% rejection rate. After completion of the run, the output portion was replaced in the hopper and the run was repeated

without changing any of the controls. After completion of the second pass, the output portion was re-run for a third time, again without changing of the controls. The rejects from the three passes were kept separate.

Test Run No. 6

In addition to the tungsten carbide particles, a similar size assortment of tungsten, molybdenum and tantalum particles were added to the chips before screening. After processing, the output portion of the $+^{3}/_{8}$ in. chips (feed rate of 5 lb/min and a rejection rate of 1.5%) was replaced in the hopper and the run was repeated twice, without changing of the controls. The rejects from the three passes were kept separate. Between the second and third passes, the bed was removed and kept separate.

Test Run No. 7

Test run No. 7 was performed with medium bulk density 6Al-4V Ti chips contaminated with tungsten carbide particles and a similar size assortment of tungsten, molybdenum and tantalum particles before screening. The $+^{3}/_{8}$ in. portion of chips was processed at a feed rate of 5 lb/min, and airflow was adjusted to give a 2% rejection rate.

Test Run No. 8

Test run No. 8 was performed with low bulk density (11 lb/ft^3) 6Al-4V Ti chips ($+^{3}/_{8}$ in.) which were processed at a feed rate of 3 lb/min and a rejection rate of $^{1}/_{2}$%.

Test Run No. 9

Test run No. 9 was performed with medium bulk density 6Al-4V Ti chips ($+^{3}/_{8}$ in.) with the addition of 4 lb of Waspaloy contaminant and no WC added before screening. The chips were processed at a feed rate of 5 lb/min and a rejection rate of 10%.

Test Run No. 10

Test run No. 10 was performed with high bulk density (41 lb/ft^3) 5Al-2.5Sn titanium chips contaminated with tungsten carbide particles. The $+^{3}/_{8}$ in. portion of chips was processed at a feed rate of 5 lb/min and the airflow was adjusted to give a 2% rejection rate.

Test results are summarized in Table 24.

TABLE 24
FRANKEL FLUIDIZED BED TEST RESULTS

Run No.	Input (lb)	Rejects			Bed (lb)	Output Good Quality (lb)	Total Recovery (lb)
		Pass 1 (lb)	Pass 2 (lb)	Pass 3 (lb)			
1	100	4.6			4	87	96
2	100	5.0	1.5	1.0	5	85	97.5
3	100	7.0			5	80	92
4	100	2.5			4	92	98.5
5	100	1.1	0.3	0.12	4	93	98
6	100	1.4	0.5	0.06	10*	87	98
7	100	2.0			3.5	94	99.5
8	100	0.5			4	95	99.5
9	100	10.25			4	83	97
10	100	2.0			7	90	99

*Bed removed after 2nd pass and new bed established for 3rd pass.

Visual inspection indicated that the rejects of each run contained WC particles in amounts apparently equal or greater than that added and additionally contained some nickel-base alloy material, stones, pieces of wood, titanium solids (narrow long pieces) and titanium chips. Typical reject constituents are shown in Figure 43. Visual inspection of the reclaimed material did not reveal any foreign substance.

Figure 43. Rejects from Frankel Fluidized Bed Test No. 1 Consisting of Hand-Picked Portions of Material Other Than Titanium Chips: Tungsten Carbide Particles (Right Side), Stones, Wood Pieces and Other Nonmetallic Particles (Upper Left Side), Long Solid Titanium Spears (Lower Left Side), and Nickel Alloy Chips and Small Magnetic Particles (Lower Middle)

(b) Conclusions

X-ray and chemical analyses are required to make firm conclusions on the efficiency of the fluidized bed separation method, since an accountability of contaminants added versus contaminants in the rejects would be impractical and inconclusive. Tentative conclusions which can be reached from this data include the following:

1. The advantage of three processing cycles versus one processing cycle is not clear, except possibly as a safety measure.

2. The expected advantage of fines removal is not clear and may not be justified due to material losses.

3. Some contaminants of low density, e.g., wood, appear to have physical properties which enable their removal by the fludizied bed.

Frankel conducted radiographic inspection of separated fractions, i.e., reclaimed material, reject material and bed material from the series of 10-test runs described above. X-ray results were analyzed to determine the removal of WC contaminants. Chemical analyses were conducted on a chip sample from the run containing Waspaloy contamination. Based on these results, four test runs were selected for further evaluation. Reclaimed material from these runs were forwarded to Teledyne-Wah Chang for ingot melting, conversion and evaluation identical to that applied to the six ferrofluid-separated samples. Details of this activity are as follows.

(3) Titanium Separation Test — Radiographic Inspection

(a) Radiographic Inspection Procedure

Samples of reclaimed material and rejects for all WC-seeded runs were radiographically inspected. In addition, bed material from a number of runs was sampled and radiographically inspected. The reclaimed material was represented by an approximate 10% sample, while the reject sample represented all or a substantial portion of the rejects.

The X-ray inspection of the rejects, processed or bed material was performed by placing the material to be X-rayed on cardboard trays measuring approximately 11 by 14 by 1 in. Typically, seven-to-nine trays were required for the reclaimed material sample. The cardboard trays were placed on two sheets of X-ray film. A calibration sample containing WC chips of different sizes was placed on top of the chip tray. A double film was used to detect any possible film defect which may appear as dense particles. As a result, two complete sets of X-rays were obtained. After comparing the two sets, one set was forwarded to P&WA. Reclaimed and reject material from Run No. 9, which contained Waspaloy rather than WC contamination, was sampled and then aqua regia leached to determine the nickel content.

(b) Radiographic Inspection Results

The X-rays of reclaimed material revealed no dense particles in any of the runs. A typical radiograph (from Run No. 6) is shown in Figure 44. The radiographs of the reject material indicate clearly the presence of dense particles as summarized in Table 25. Radiographs of rejects for Run Nos. 1 and 2 are shown in Figures 45 and 46 respectively. It may be noted that the X-rays of Run No. 3 rejects indicate at least 131 dense particles, considerably more than were used to salt the material in the appropriate size range. This indicates that in addition to the dense particles added for test purposes, additional particles occurring naturally in the chips were also removed. X-ray examinations of the bed material indicate, as expected, a high number of large, dense particles which because of their size and shape adhered to the reject screen rather than being carried on the screen to the reject chute. Radiographs of bed material from Run Nos. 6 and 8 are shown in Figures 47 and 48. These results indicate that the fluidized bed is very efficient in removing contaminants of the size (>0.025 in.), and concentration contained in these runs. A radiograph of Run No. 7 rejects (Figure 49) shows many dense particles. Some of these particles were removed (Figure 50) and identified by spectrographic analyses. The analysis indicated pieces of naturally-occurring titanium, low-melting alloy and pieces of added contaminants (tungsten carbide, tungsten, molybdenum and tantalum). The naturally-occurring "titanium" and "low-melting alloy" contaminants identified by Frankel in their rejects appear to be identical to contaminants found in the floats and sinks from the AVCO ferrofluid separation process.

FD 171556

Figure 44. Photo-Radiograph of Frankel-Processed "Good" Material (Test Run No. 6)

TABLE 25
SIZE/QUANTITY OF HIGH-DENSITY MATERIAL FOUND IN FRANKEL CO. REJECTS

		Rejects (in.)					*Bed (in.)*		
Run No.	*Pass No.*	*Less Than 0.080*	*0.080 To 0.150*	*0.150 To 0.300*	*0.300 To 0.500*	*Over 0.500*	*0.150 To 0.300*	*0.300 To 0.500*	*Over 0.500*
1	1	15	14	21	7	6			
2	1	10	13	18	14	7			
	2	0	1	0	0	0			
	3	0	0	0	0	0			
3	1	46	56	29	0	0			
4	1	5	12	28	18	13			
5	1	0	0	10	2	2			
	2	0	0	0	0	1			
	3	0	0	0	0	0			
6	1	0	2	17	11	17	0	1	10
	2	0	0	0	0	0			
	3	0	0	0	0	0			
7	1	1	9	19	20	5	2	6	16
8	1	3	11	31	17	0	0	2	6
9	1	Not X-Rayed							
10	1	0	0	3	4	1	0	2	6

As noted previously, feedstock for all runs was screened following seeding of contaminants and prior to processing. Material from Run 9, which was contaminated with 4% Waspaloy, was analyzed for nickel content in order to determine the degree of success in removal of contaminants other than WC. The reclaimed material sample contained approximately 0.15% Nickel, while the rejects contained 7.0% Nickel. This result indicates that the removal of nickel-base alloy chip contamination or other similar chip contaminations, while not completely efficient, can be performed to a reasonably satisfactory degree. It should be noted that the specification for Ti-6Al-4V material allows a combined total of 0.40% nonspecified elements.

Conclusions as to the relative merits of test parameters utilized in the various runs are difficult to make, since no dense particles were detected in radiographs of the reclaimed material. Screening of the material feedstock prior to processing appears to be desirable to eliminate fines; however, the mesh-size employed must be evaluated in terms of yield and economics. Multiple processing cycles do not appear to be necessary to attain quality, but if economically viable may still be desirable to increase process reliability.

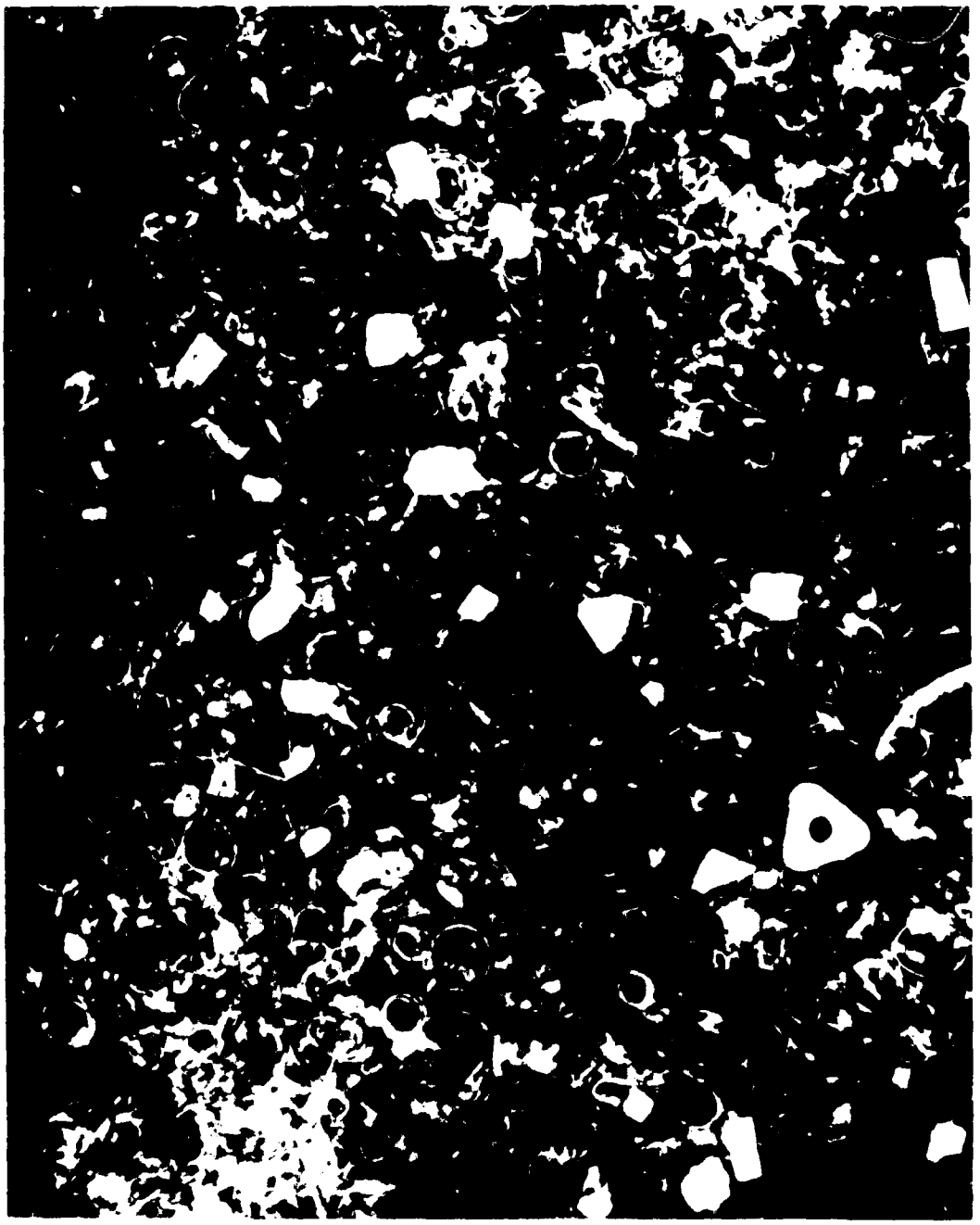
(c) Selection of Ingot Melt Stock

The reclaimed product of four test runs was selected for melting and subsequent evaluation, and the total quantity from each run was sent to Teledyne-Wah Chang for melting. The same detailed evaluation was performed as that applied to the product of the ferrofluid separation melt.

The materials selected for ingot melt were from fluidized bed Run Nos. 1, 2, 5 and 8, and as such, represent specific processing conditions. Run Nos. 1 and 2 represent single- and triple-pass processing respectively. Run No. 5 represents a potentially optimum condition, with rescreening of the material prior to processing plus triple-pass processing. Run No. 8 demonstrates processing of a lower bulk density material than other runs, which is relatively abundant in the scrap market.

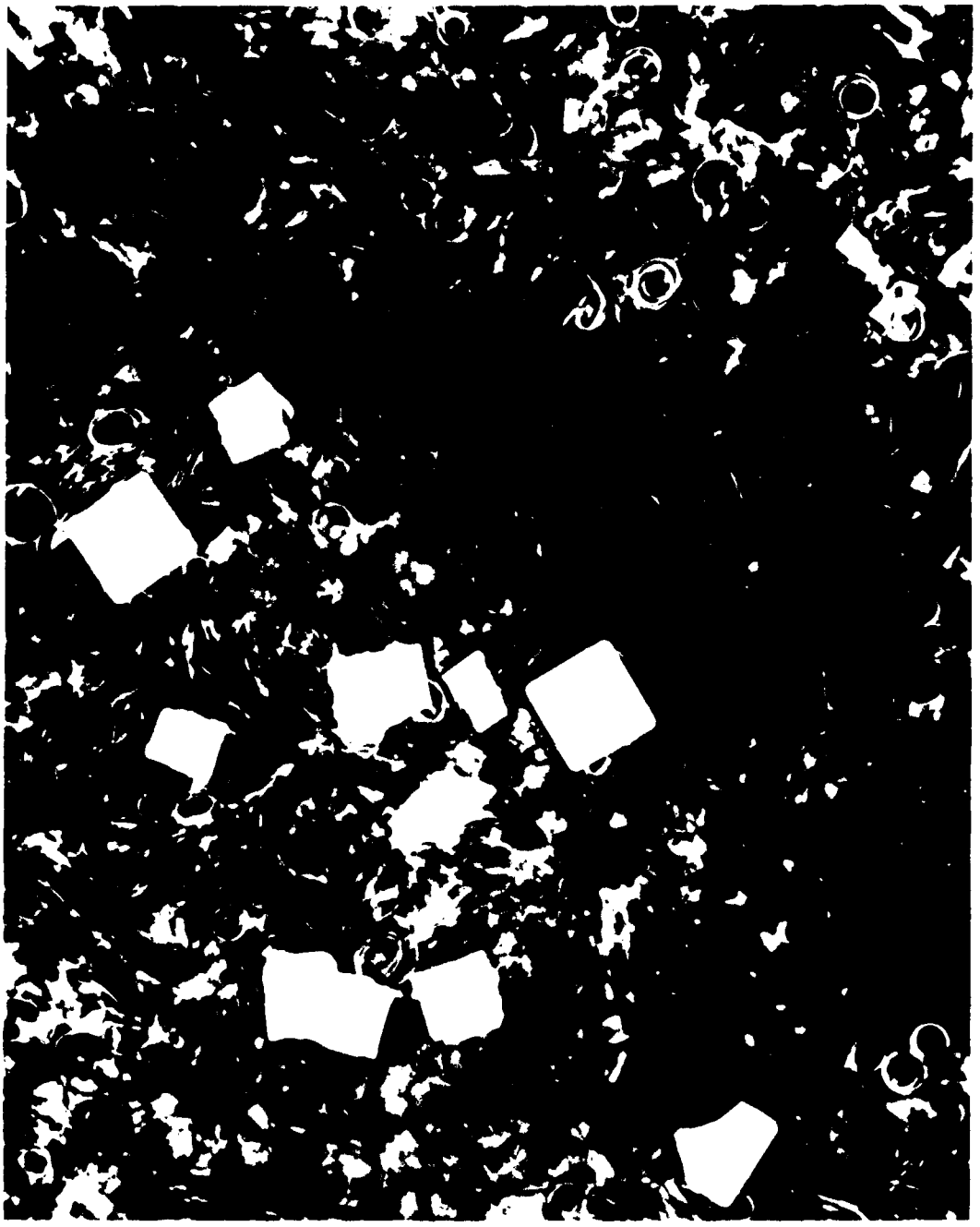
FD 171557

Figure 45. Photo-Radiograph of Frankel Processed "Reject" Material (Test Run No. 6)



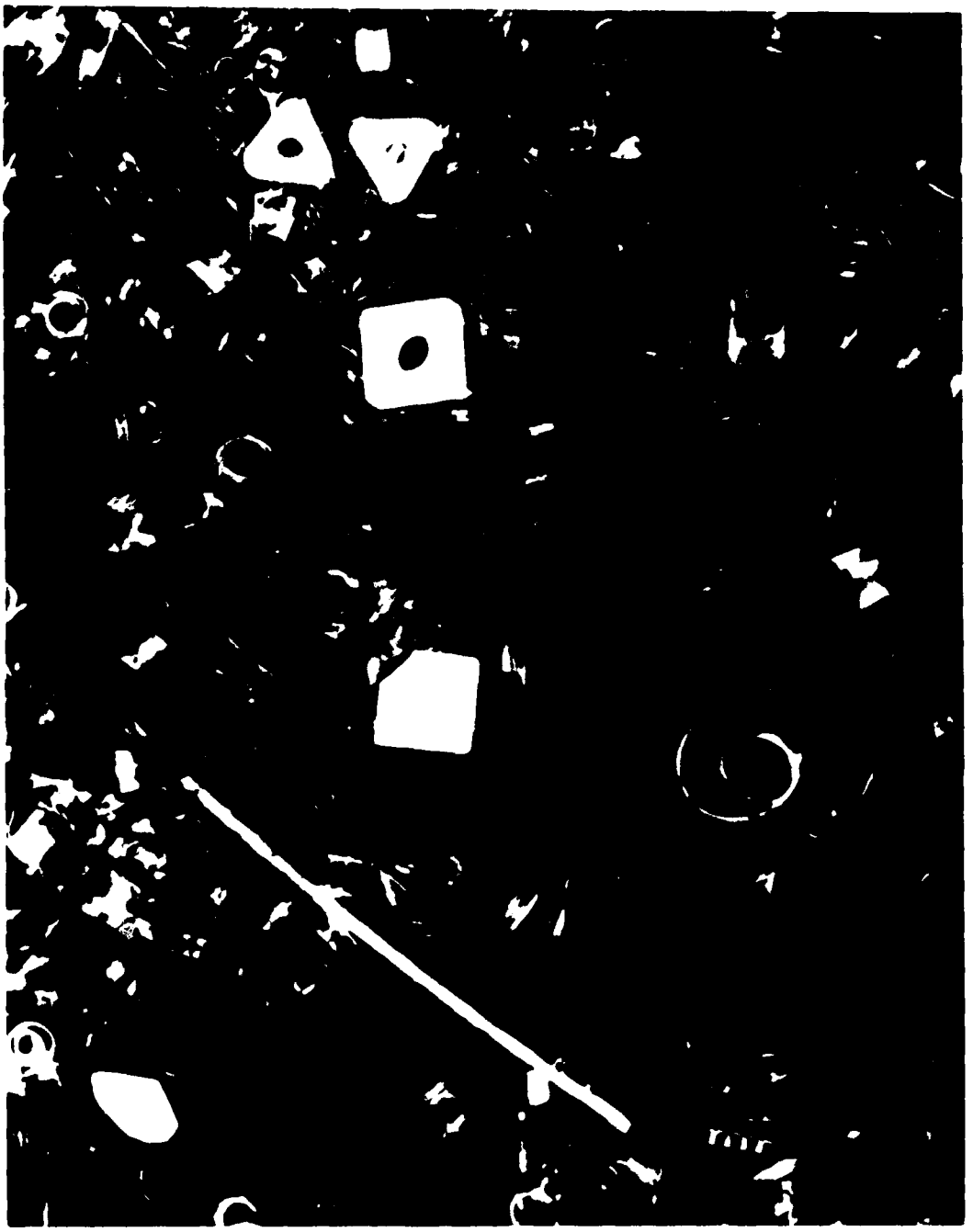
FD 171558

Figure 46. Photo-Radiograph of Frankel-Processed "Reject" Material (Test Run No. 2, Pass No. 1)



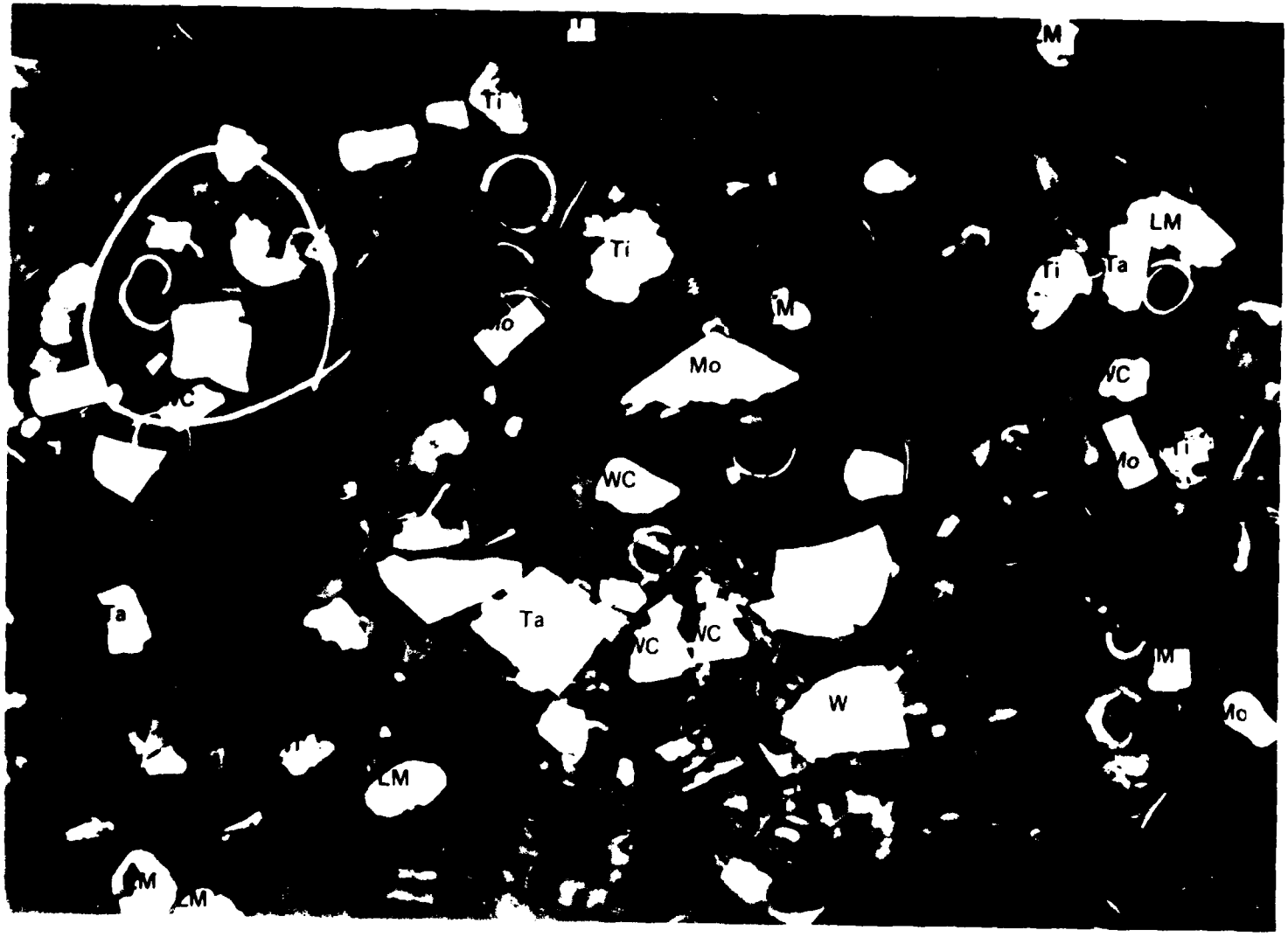
FD 171559

Figure 47. Photo-Radiograph of Frankel-Processed "Bed" Material (Test Run No. 6, Pass No. 1 and 2)



FD 171560

Figure 48. Photo-Radiograph of Frankel-Processed "Bed" Material (Test Run No. 8)



FD 171561

Figure 49. Photo-Radiograph of "Rejects" from Frankel Run No. 7 Identified by Spectroscopy

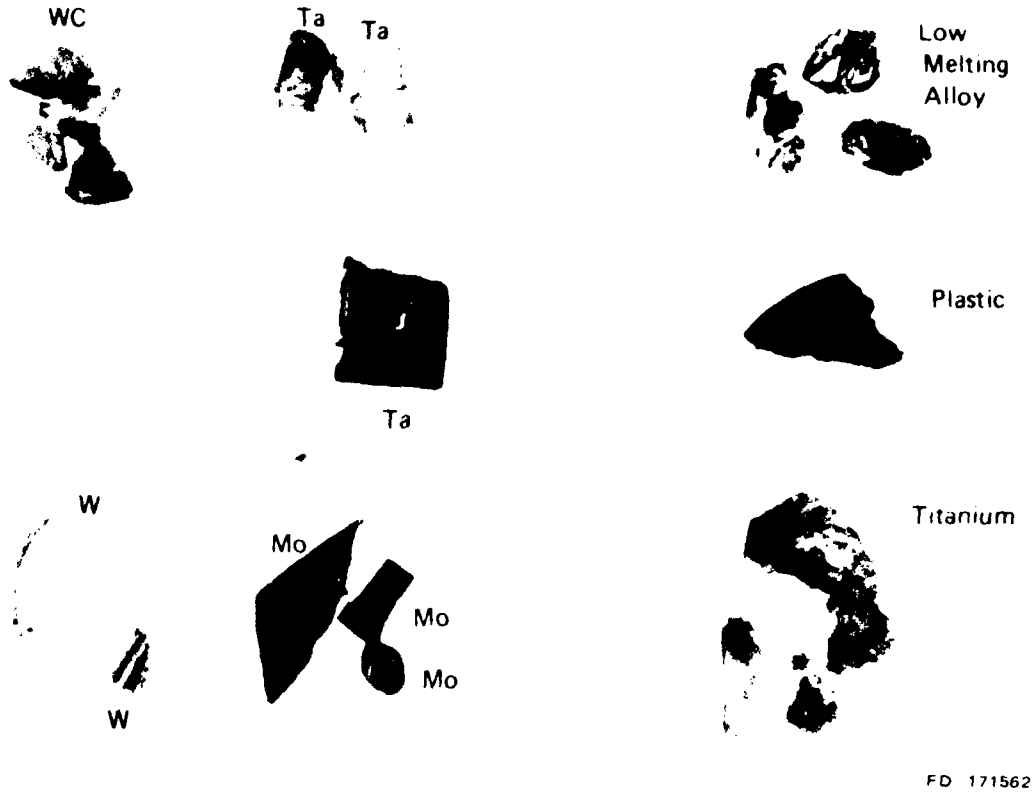


Figure 50. Rejects from Frankel Run No. 7

3. P&WA Evaluation of Density Separation Processes (AVCO Ferrofluid and Frankel Fluidized Bed)

a. Evaluation of Titanium Separations

To complete the study, Ti-base scrap that had been artificially contaminated and separated by either the AVCO ferrofluid density process or the Frankel fluidized bed separation process was melted into 100 lb ingots, converted into plate and evaluated by radiography, chemistry, metallography, particle analysis and mechanical properties.

(1) Melting and Conversion Procedures

Six lots of ferrofluid processed Ti-6Al-4V chips from AVCO and four lots of fluidized bed processed chips from Frankel were processed by Teledyne. In order to compact the chips into briquettes of sufficient strength for consumable melting, it was necessary to blend Ti sponge with several lots (Table 26). The chips were pressed into 1-in. high by 6½-in. dia briquettes and electron beam welded into consumable electrodes. Single vacuum consumable melting at 7000 amps resulted in 9-in. dia ingots. The ingots were lathe turned, heated to 2000°F and upset forged to 2-in. thick "pancakes." These were spot ground, reheated to 2000°F and rolled to ¾-in. thick plate.

TABLE 26
TELEDYNE TITANIUM MATERIALS
SUMMARY (INGOT MELTING)

Chip Source	*Run No.*	*Chip Wt * (lb)*	*Ti Sponge Added (lb)*
AVCO	6	100.5	33.5
AVCO	9	111.0	36.5
AVCO	23	65.5	4.5
AVCO	58	70.0	24.0
AVCO	59	88.0	0
AVCO	60	78.0	24.0
Frankel	1	76.0	28.0
Frankel	2	76.5	25.5
Frankel	5	87.0	29.0
Frankel	8	91.0	33.0

* Approximately 5 lb of chips used as bottom charge for consumable melting

(2) Analyses/Results

Each rolled plate was fully radiographed by Teledyne using a double film technique to protect against occasional film defects. The sensitivity level was set to detect a 0.015-in. defect

Table 27 summarizes the total quantity and size of high density inclusions detected by radiography in each rolled plate. Figure 51 shows the radiographs of two clusters of high density inclusions that were not subsequently identified for composition. These radiographs are typical of the larger inclusions detected.

AVCO had performed a total of 60 separation experiments during this contract, as described earlier. AVCO Run No. 6 and 9 are preliminary separation runs at near optimum process parameters. No. 23 was less than optimum but selected to assess the effect of melting on material which was highly contaminated with fine inclusions. AVCO Run No. 58 through 60 were processed at optimum parameters for low, high and medium density chips respectively

Frankel had conducted a total of 10 separation experiments during this contract. Frankel Run Nos. 1 and 2 were made at identical fluidized bed operating parameters and consisted of single and triple passes respectively. Run No. 5 was a triple-pass run made with rescreening after contaminant seeding; this run is less desirable for the intent of this contract (maximum reclamation of strategic materials) than the optimum Frankel Run No. 2 for medium density chips. Run No. 8 was conducted on low density chips.

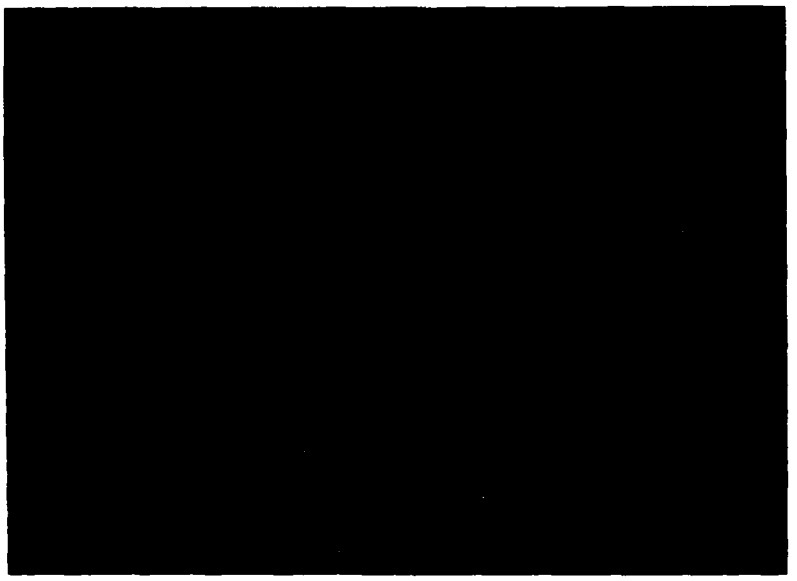
Cursory examination of Table 27 would imply that AVCO separation experiments were less effective in removing small (< 0.020-in.) high density particle inclusions; however, this implication is not correct. AVCO contaminant seeding contained many WC "fines" in this size range while Frankel seeding was of a coarser nature (see seeding ranges, Table 27). It appears that rescreening after contaminant seeding (AVCO Run No. 58 through 60 and Frankel No. 5), was ineffective in subsequent removal of contaminant fines. Contaminant removal was also aided by additional passes as shown by comparison of Frankel Runs No. 1 and 2.

In summary, based on Table 27 data, it is apparent that both separation processes are quite effective in removal of most of the seeded contaminants, but neither process demonstrated complete particle removal during this study.

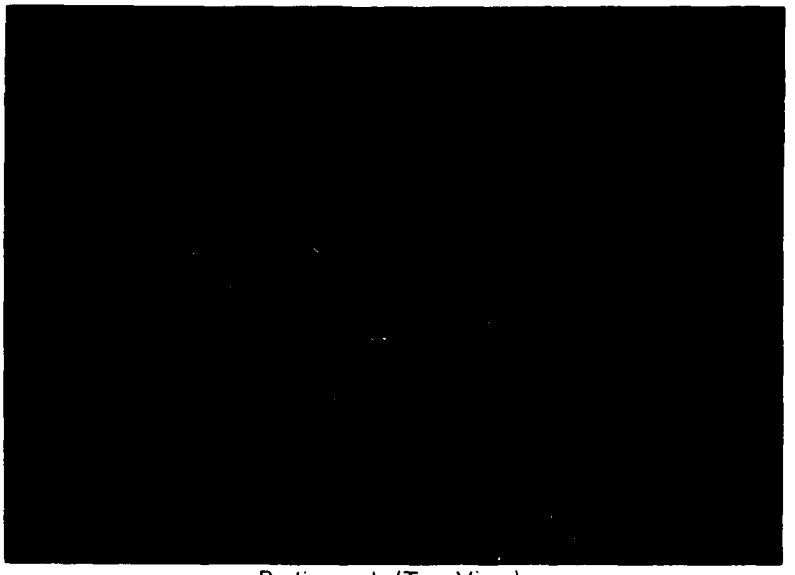
TABLE 27
NUMBER OF HIGH-DENSITY INCLUSIONS VS SIZE RANGE IN TITANIUM ROLLED PLATE

AVCO Run No		Screened at Frankel 10 Mesh (0.0787 × 0.0787) (Fines are Rejected)	WC Seeded	Screened After Seeding Before Separating (Fines are Rejected)	No. Passes During Sepn	Particle Size Range, In. 0.010	0.010 To 0.020	0.020 To 0.030	0.030 To 0.040	0.040 To 0.060	-0.060	Largest Dimension Found	Probe Results
						AVCO WC SEEDING RANGE					-0.150 in.		
6		Yes	Yes	No	1	10	28	12	3	1*	0		*Ti rich, Ti rich
9		Yes	Yes	No	1	0	3	1	0	1	2	0.150	
23	Med ρ	No	Yes	No	1	0	4	1	0	2	0		
60		Yes	Yes	Yes No. 10 mesh (0.0787 in.)	1	5	7*	0	0	1	9		*WC, WC
58	Low ρ	Yes	Yes	Yes No. 5 mesh (0.157 in.)	1	3	2	9*	2	1	1	0.150x 0.010	*Cb rich
59	High ρ	Yes	Yes	Yes No. 10 mesh (0.0787 in.)	1	0	1	1	0	0	0		
FRANKEL Run No						FRANKEL WC SEEDING RANGE					-0.5 in.		
1		Yes	Yes	No	1	0	5	2	7	2	2*	0.250x 0.010*	*Ta rich
2	Med ρ	Yes	Yes	No	3	0	0	2	5	1	0		
5		Yes	Yes	Yes (- in.)	3	0	0	5*	4	1	0		*WC
8	Low ρ	Yes	Yes	Yes (- in.)	1	0	0	1	0	0	0		

*Particle(s) selected for electron microprobe analysis



Mag 10X Radiograph (Top View)
Avco Plate No. 58



Radiograph (Top View)
Frankel Plate No. 1

FD 171563

Figure 51 Radiographs of High-Density Inclusions Detected in Rolled Plate Titanium

Based on severe reductions in component fatigue life in both laboratory and service experience that were attributed to high density inclusions, P&WA Materials Control will not permit any such inclusions in highly stressed components. Therefore, the quantity of high density inclusions detected in rolled plate of both separation processes (Table 27), although small in cumulative number, are still unacceptable by P&WA standards. However, there is strong evidence, to be discussed later, that either of these separation processes in combination with a nonconsumable melting process could effectively remove all high density inclusions.

(b) Chemical Analysis

Complete chemical analysis was obtained from the top and bottom of each ingot by Teledyne. P&WA performed complete chemical analysis of each rolled plate fabricated from these ingots. Chemistry data for both AVCO and Frankel separation processes, as applied to medium density chips, are given in Table 28, and as applied to both low and high density chips, in Table 29. A review of both chemistry tables indicates consistent correlation between the subcontractor and the P&WA analyses.

Tables 28 and 29 show that oxygen contamination prevails in all cast ingots and in converted, rolled plate fabricated from both AVCO and Frankel separated chips. Contamination by oxygen was consistently noted in all raw material scrap chips, and as discussed earlier can be attributed to prior processing (i.e., heat treatment, machining). Since the separation processes evaluated do not presently include surface conditioning, no improvement in oxygen contamination level was anticipated. An appropriate pickling operation could reduce this oxygen contamination for both processes.

There is evidence of Fe contamination exceeding the 0.3% specification maximum in AVCO medium and low density chip separations. The raw material scrap chips utilized in this program (Table 30) contained evidence of iron contamination (0.18 to 0.57% Fe) after magnetic scrap separation. AVCO subsequently seeded 1.6% Fe in their chips (in the form of AISI 304 stainless) before performing their ferrofluid separation. The range of Fe content (0.19 to 0.34%) after separation and ingot melting/conversion slightly exceeds the specification maximum. This indicates that AVCO was reasonably effective in reducing the combined natural and artificial (seeded) Fe contamination, but not completely effective. Frankel, using the same naturally Fe-contaminated raw material chips, effectively attained the Fe specification level after separation and ingot melting/conversion. However, since Frankel did not artificially seed with Fe contaminant, their separation requirements, with respect to Fe, were less severe than those of AVCO.

The mild deviation in V content (3.40% analyzed vs 3.50% specification minimum) found in the ingot/converted plate fabricated from the high density chips (AVCO Run No. 59) was expected since the raw material used in this separation was slightly deficient (3.49%) in V content.

In general, and from an ingot melter's viewpoint, all aforementioned deviations in chemistry, both elemental and interstitial, could be considered acceptable for remelting by blending with less than 50% virgin raw materials.

(c) Metallographic Analysis

Figures 52 and 53 are photomicrographs showing typical microstructures taken in the longitudinal direction of the rolled plate product fabricated from medium density raw material chips after the AVCO and Frankel separation processes, respectively. Likewise, Figure 54 shows typical longitudinal photomicrographs of rolled plate product fabricated from both low and high density raw material chips after the separation processes.

TABLE 28
CHEMISTRY OF TITANIUM ROLLED PLATE FROM MEDIUM DENSITY CHIPS

		AVCO Plate No. 6	AVCO Plate No. 9	AVCO Plate No. 23	AVCO Plate No. 60		Frankel Plate No. 1		Frankel Plate No. 2		Frankel Plate No. 5	
	PWA 1215 Spec	Corrected Chem(1) 23.7% Sponge Teledyne(2)	Corrected Chem 23.9% Sponge Teledyne	Corrected Chem 5.8% Sponge Teledyne	Corrected Chem 22% Sponge PWA(3)	Teledyne	Corrected Chem 25.7% Sponge PWA	Teledyne	Corrected Chem 23.8% Sponge PWA	Teledyne	Corrected Chem 24.0% Sponge PWA	Teledyne
Al	5.50 to 6.75%	6.47	6.45	6.26	6.28	6.35	6.33	6.51	6.30	6.56	6.32	6.45
V	3.50 to 4.50	3.73	3.66	3.63	3.59	3.73	3.77	3.97	3.81	3.93	3.95	4.22
Si	0.1 Max	–	–	–	0.08		0.08	—	0.07	—	0.08	—
Fe	0.3 Max	0.27	0.34	0.33	0.29	0.32	0.23	0.19	0.24	0.23	0.20	0.20
O	0.2 Max	0.50	0.62	0.52	0.33	0.45	0.32	0.32	0.33	0.33	0.30	0.37
C	0.1 Max	0.053	0.054	0.047	0.31	0.23	0.04	0.04	0.07	0.05	0.04	0.03
N	0.05 Max	0.014	0.015	0.016	0.012	0.012	0.011	0.008	0.010	0.011	0.012	0.009
H	0.015 Max	—	-		0.006		0.008	-	0.012	—	0.007	—
B	0.003 Max	–	-		0.003		0.003	-	< 0.003	—	< 0.003	—
Cu	0.1 Max	< 0.05	< 0.05			< 0.05	—	< 0.05	—	< 0.05	—	< 0.05
Pb		—	—		< 0.001	-	< 0.001	-	< 0.001	—	< 0.001	—
W		—	–		< 0.06	-	< 0.07	-	< 0.07	—	< 0.07	—
Cb		—	-		< 0.06		< 0.07	—	< 0.07	—	< 0.07	—
Zr		< 0.10	< 0.10	< 0.10	< 0.01	< 0.10	< 0.007	< 0.10	< 0.007	< 0.10	< 0.001	< 0.10
Sn		< 0.05	< 0.05	< 0.05	< 0.01	0.05	< 0.1	0.10	< 0.1	0.10	< 0.7	0.21
Ni		< 0.05	< 0.08	< 0.05	< 0.06	< 0.06	< 0.07	< 0.05)	< 0.07	< 0.05	< 0.01	< 0.05
Mo		< 0.10	< 0.10	< 0.115	< 0.06	0.13	< 0.07	0.13	< 0.07	< 0.10	< 0.007	0.10
Mn		< 0.005	< 0.005	< 0.005	< 0.01	< 0.005	< 0.007	< 0.005	< 0.007	< 0.005	< 0.007	< 0.005
Cr		< 0.05	0.05	< 0.05		< 0.05	—	< 0.05	—	< 0.05	—	< 0.05

NOTE: (1) All chemistry corrected for % sponge addition
(2) Teledyne chemistry is average of ingot top and bottom
(3) PWA chemistry is from rolled plate

TABLE 29
CHEMISTRY OF TITANIUM ROLLED PLATE FROM LOW AND HIGH DENSITY CHIPS

		AVCO Plate No. 58 (Low-Density Chips) Corrected Chem(1) For 27% Sponge		*Frankel Plate No. 8 (Low-Density Chips) Corrected Chem For 25.6% Sponge*		*AVCO Plate No. 59 (High-Density Chips) Uncorrected Chem (0% Sponge)*	
	PWA 1215 Spec	*PWA(3)*	*Teledyne(2)*	*PWA*	*Teledyne*	*PWA*	*Teledyne*
Al	5.50 to 6.75%	6.57	6.44	6.45	6.52	6.5	6.45
V	3.50 to 4.50	3.83	3.67	3.90	4.06	3.4	3.40
Si	0.1 Max	0.07	-	0.08		0.05	--
Fe	0.3 Max	0.34	0.29	0.21	0.21	0.20	0.19
O	0.2 Max	0.37	0.52	0.31	0.33	0.28	0.47
C	0.1 Max	0.05	0.055	0.04	0.027	0.04	0.035
N	0.05 Max	0.015	0.014	0.011	0.010	0.015	0.017
H	0.015 Max	0.005	-	0.008		0.004	--
B	0.003 Max	< 0.003		0.003		< 0.002	--
Cu	0.1 Max	--	-	--	< 0.05		--
Pb		< 0.001	-	< 0.001		< 0.001	--
W		< 0.07	-	< 0.07		< .05	--
Cb		< 0.07		< 0.07	-	< 0.05	--
Zr		< 0.01	0.17	< 0.007	-	< 0.001	< 0.10
Sn		< 0.01	< 0.05	< 0.1	0.097	< 0.01	< 0.05
Ni		< 0.1	< 0.05	< 0.07	< 0.05	< 0.05	< 0.05
Mo		< 0.07	0.13	< 0.07	< 0.10	< 0.05	0.11
Mn		< 0.01	< 0.005	< 0.007	< 0.05	< 0.01	< 0.005
Cr		-	< 0.05		0.05		< 0.05

NOTE: (1) All chemistry corrected for % sponge addition
(2) Teledyne chemistry is average of ingot top and bottom
(3) PWA chemistry is from rolled plate

TABLE 30
ANALYSIS OF TITANIUM CHIP RAW MATERIALS

PWA	*1215 Spec (%)*	*Medium Density (23.2 lb/ft³)*				*High Density (36.3 lb/ft³)*	*Low Density (15.6 lb/ft³)*
		FRANKEL	*AIRCO*	*AIRCO by PWA*	*AVCO by PWA*	*FRANKEL*	*FRANKEL*
Al	5.50 to 6.75	6.55	6.07	6.4	6.5	6.70	6.50
V	3.50 to 4.50	3.70	3.82	3.9	3.8	3.49	3.80
Si	0.1 Max			0.05	0.05		-
Fe	0.3 Max	0.38	0.18	0.21	0.18	0.38	0.57
O	0.2 Max		0.27	0.26	0.28		
C	0.1 Max		0.036	0.12	0.05		
N	0.05 Max	0.045		0.011	0.013	0.033	0.06
H	0.015 Max			0.011	0.008	-	-
B	0.003 Max			0.02	0.002		
Cu	0.1 Max						
Pb		0.05		< 0.001	< 0.001	< 0.05	0.18
W				0.05	< 0.05		
Co		0.05	0.05	0.05	< 0.05	0.05	< 0.05
Zr		0.07		< 0.01	0.01	0.05	0.10
Sn		0.09	0.66	0.01	0.01	0.05	0.5
Ni		0.30	0.47	0.1	0.1	0.25	0.10
Mo		0.15	0.065	0.05	0.05	0.22	< 0.05
Mn		0.05		0.01	0.01	0.05	0.05
Cr		0.09	0.024			0.08	< 0.05
Mg			0.05				

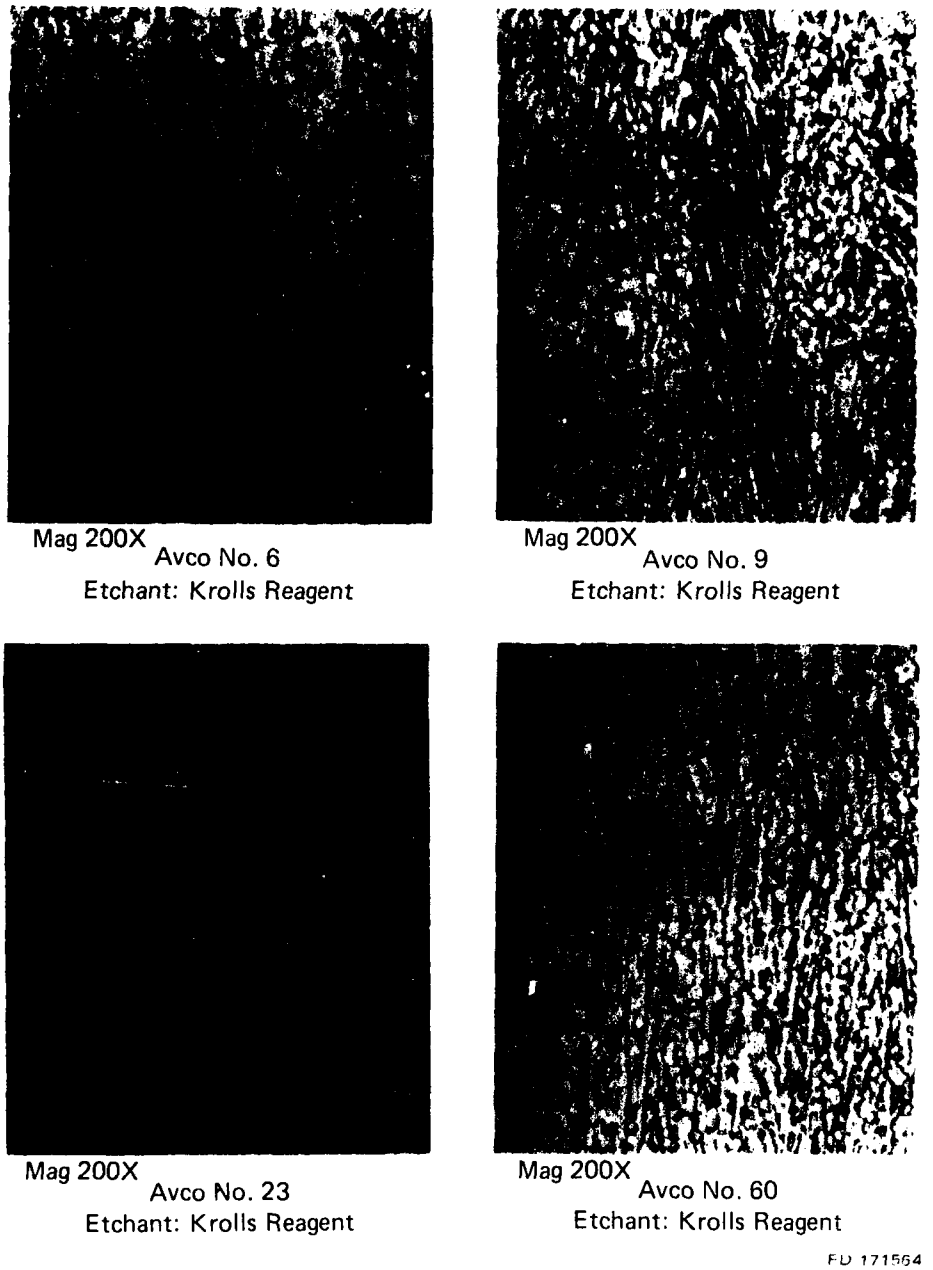


Figure 52. Photomicrographs of Rolled Plate Fabricated from AVCO Separation-Processed Medium-Density Titanium Chips

Mag 200X Frankel No. 1
Etchant: Krolls Reagent

Mag 200X Frankel No. 2
Etchant: Krolls Reagent

Mag 200X Frankel No. 5
Etchant: Krolls Reagent

ED 171565

Figure 53. Photomicrographs of Rolled Plate Fabricated from Frankel Separation-Processed Medium-Density Titanium Chips

Mag 200X

Low Density Avco No. 58
Etchant: Krolls Reagent

Mag 200X

Low Density Frankel No. 8
Etchant: Krolls Reagent

Mag 200X

High Density Avco No. 59
Etchant: Krolls Reagent

FD 171566

Figure 54. Photomicrographs of Rolled Plate Fabricated from Density Separation-Processed Low- and High-Density Titanium Chips

Analysis of these microstructures show transformed beta with elongated alpha platelets and prominent prior beta grain boundaries. These structures are caused by fabricating above the beta transus level of the Ti alloy and would be unacceptable to P&WA standards for critically stressed rotating components. The reported Teledyne forging and rolling process parameter of 2000°F was responsible for this structure.

(d) Particle Analysis

Based on the quantity of high density inclusions detected by radiography of the rolled plate, P&WA selected several representative inclusions for subsequent metallographic and electron microprobe analysis. Specific inclusions selected for this study are identified in Table 27.

The original radiographs were utilized to crudely locate the inclusion in the rolled plate, and the suspect area was sectioned and radiographed a second time to accurately locate the particle. After metallographic sectioning to expose the particle and documentation (light metallography), the particle composition was identified by X-ray energy spectroscopy using the electron microprobe. In this identification, both secondary electron image photographs (to show surface topography) and backscatter electron image photographs (atomic number analyses) were obtained.

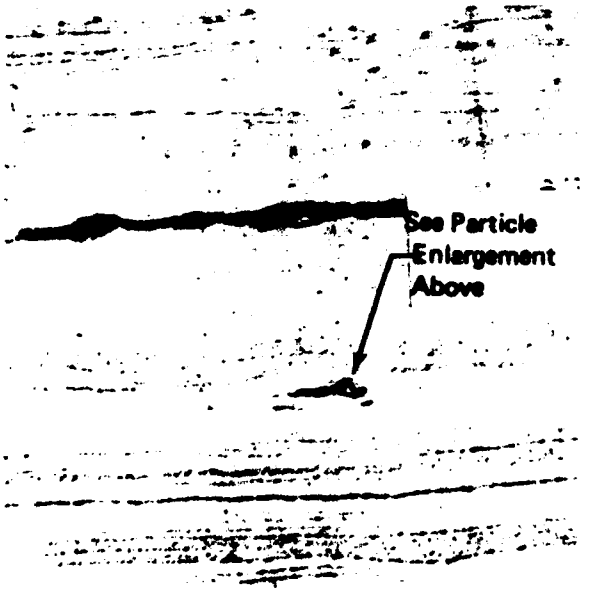
Table 31 summarizes the identification of particles that were analyzed as typical radiographic high density inclusions in the rolled plates. Subsequent metallographic analyses revealed four particles of lower density, and these particles were identifed and included in Table 31.

Figures 55 through 61 show light and electron photomicrographs and radiographs, where applicable, of these inclusions. Figures 62 through 68 show X-ray energy spectroscopic data (from the scanning electron microprobe) used to identify inclusion elements.

TABLE 31
IDENTIFICATION OF FOREIGN PARTICLES DETECTED IN ROLLED PLATE

Rolled Plate	*Particle Photo*	*Microprobe Data*	*Remarks*
AVCO No. 6	Figure 55	Figure 62	(1) Titanium-rich inclusion, many laminations
AVCO No. 6	Figure 56	Figure 63	(1) Titanium-rich inclusion (2) Al and Mg-rich inclusion (3) Ti-rich inclusion with Mo, Sn, and Al
AVCO No. 58	Figure 57	Figure 64	(1) Cb-rich inclusion with Hf, Ta or W
AVCO No. 60	Figure 58	Figure 65	(1) WC cluster inclusion
AVCO No. 60	Figure 59	Figure 66	(1) WC cluster inclusion
Frankel No. 1	Figure 60	Figure 67	(1) Ta wire inclusion
Frankel No. 5	Figure 61	Figure 68	(1) WC inclusion

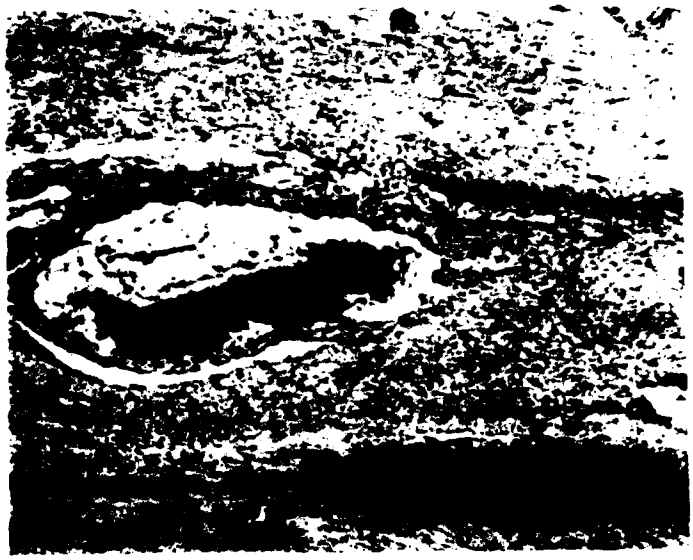
(Krolls Etch) Mag 100X



Longitudinal View (Krolls Etch) Mag 15X

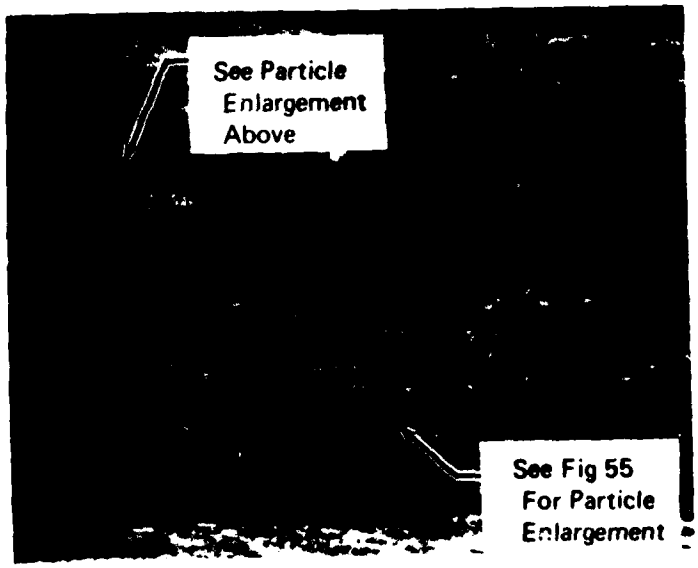
FD 171567

Figure 55. Foreign Particle Found in AVCO No. 6 Rolled Titanium Plate Type Two



Mag 100X

(Krolls Etch)

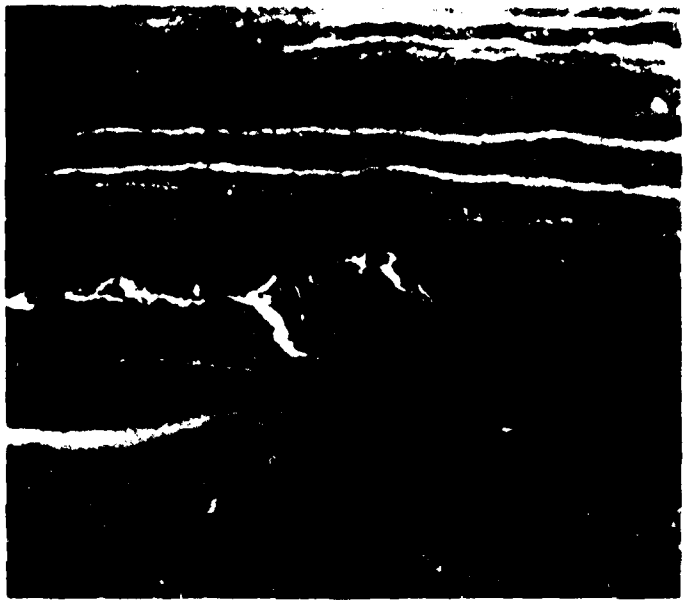


Mag 8X

Longitudinal View (Krolls Etch)

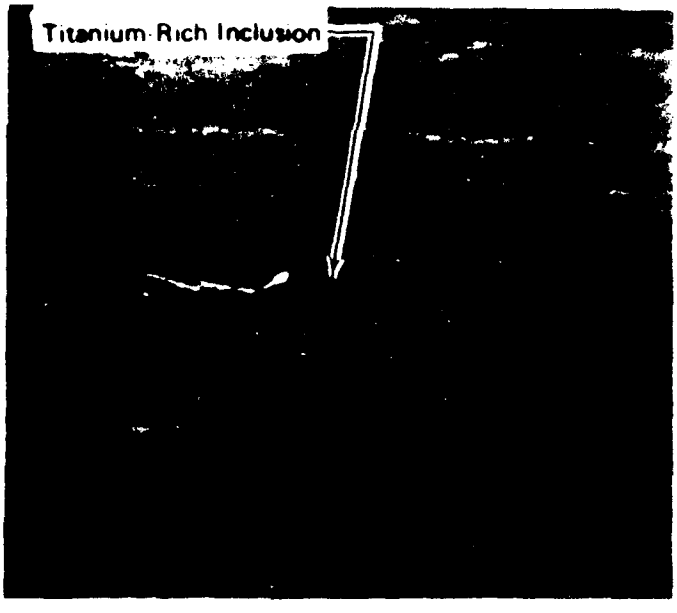
FD 171568

Figure 55. Foreign Particle Found in AVCO No. 6 Rolled Titanium Plate Type Two (Continued)



Mag 100X

Secondary Electron Image



Mag 100X

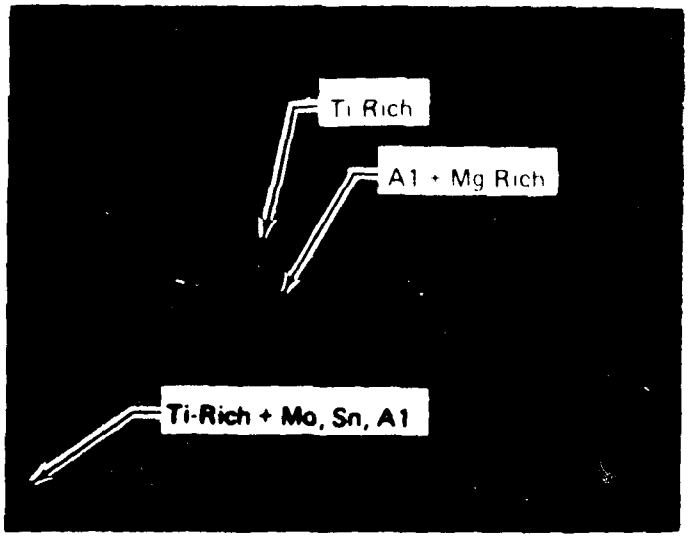
Backscatter Electron Image

FD 168209

Figure 55. Foreign Particle Found in AVCO No. 6 Rolled Titanium Plate (Continued)

Mag 100X

Secondary Electron Image



Mag 100X

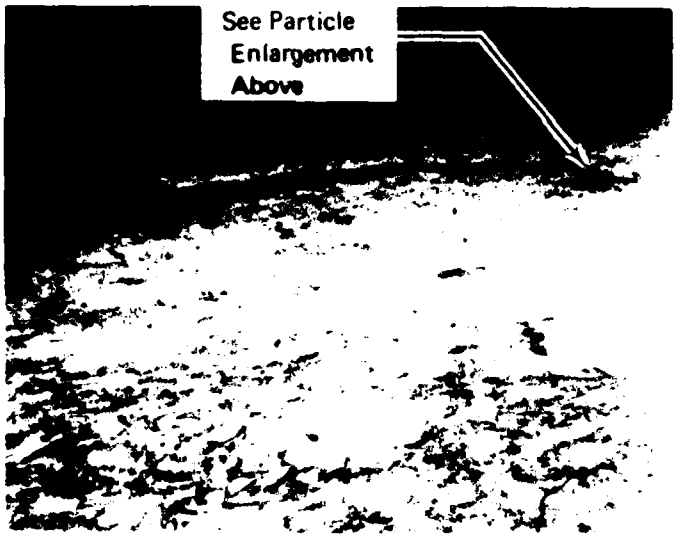
Backscatter Electron Image

FD 168211

Figure 56. Foreign Particle Found in AVCO No. 6 Rolled Titanium Plate Type Two

Mag 100X

(Krolls Etch)



Mag 8X

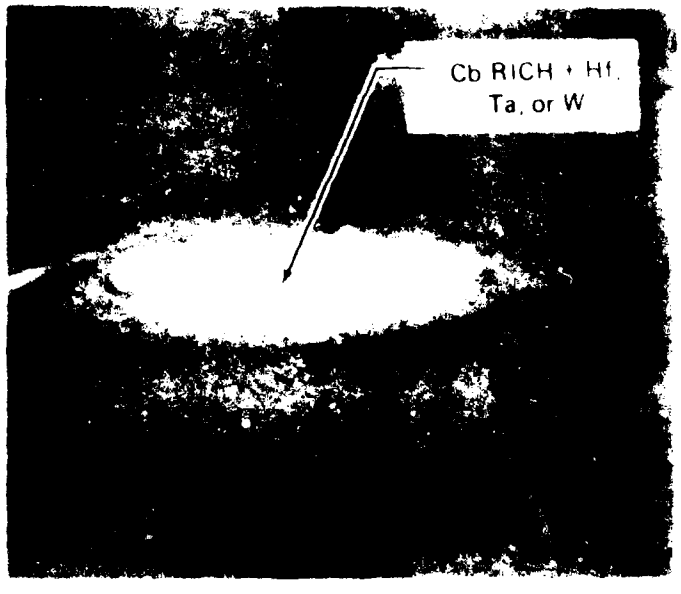
Transverse View
(Krolls Etch)

FD 171569

Figure 57. Foreign Particle Found in AVCO No. 58 Rolled Titanium Plate

Mag 100X

Secondary Electron Image



Mag 100X

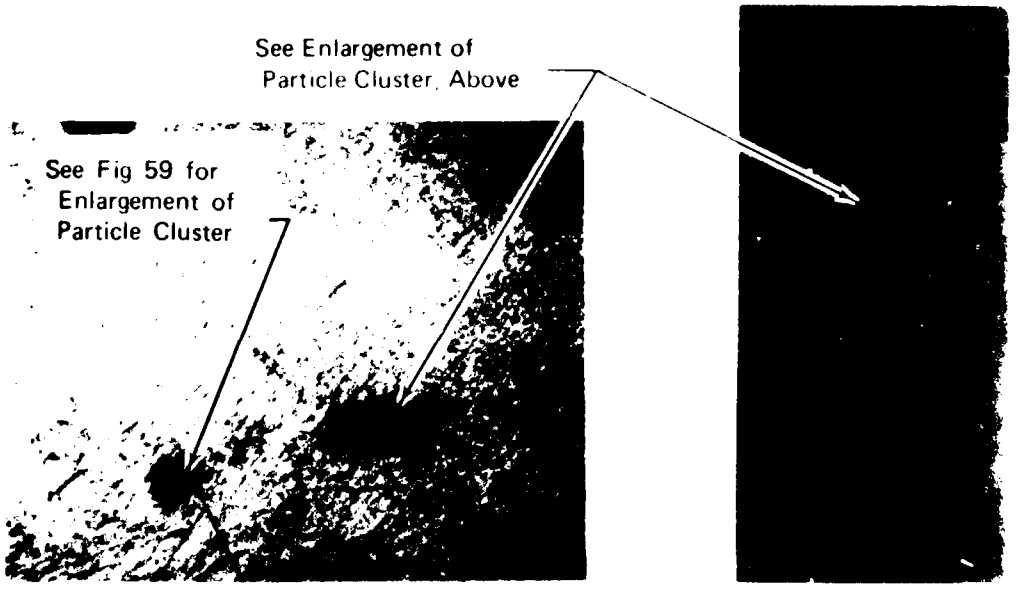
Backscatter Electron Image

FD 168212

Figure 57. Foreign Particle Found in AVCO No. 58 Rolled Titanium Plate (Continued)

Mag 100X

(Krolls Etch)



Mag 8X

Top View (Krolls Etch)

Mag 10X

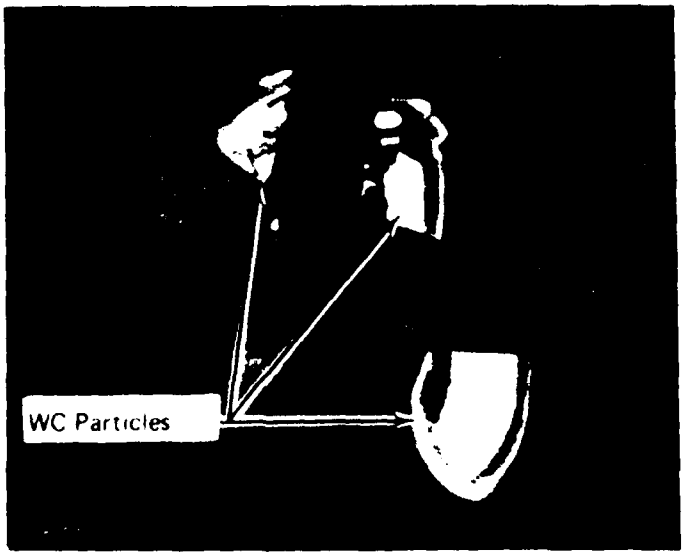
Radiograph (Top View)

FD 121520

Figure 58. Cluster of Foreign Particles Found in AVCO No. 60 Rolled Titanium Plate

Mag 100X

Secondary Electron Image



Mag 100X

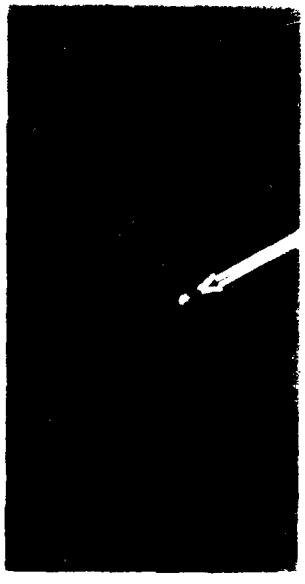
Backscatter Electron Image

FD 168213

Figure 58. Cluster of Foreign Particles Found in AVCO No. 60 Rolled Titanium Plate (Continued)

Mag 100X

Top View (Krolls Etch)



See Enlargement of
Particle Cluster, Above

Mag 10X

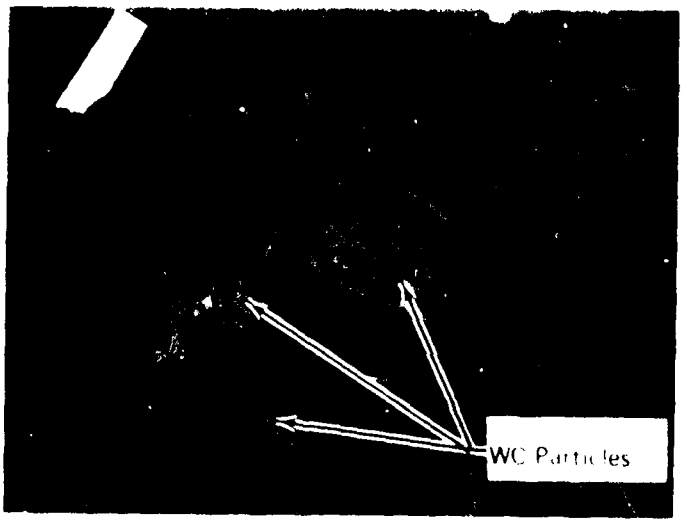
Radiograph (Top View)

FD 171571

Figure 59. Cluster of Foreign Particles Found in AVCO No. 60 Rolled Titanium Plate

Mag 100X

Secondary Electron Image



Mag 100X

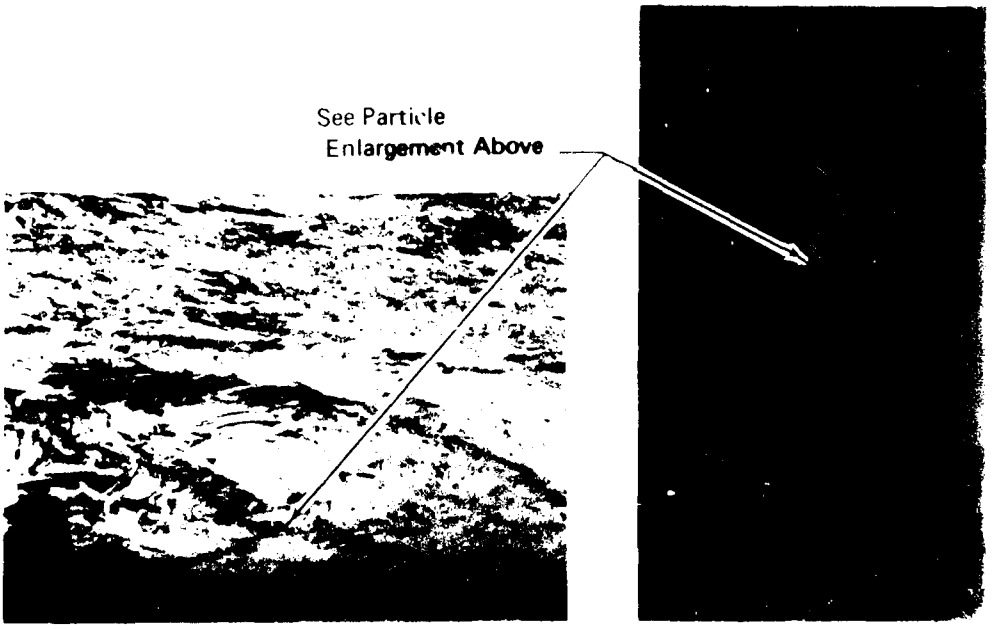
Backscatter Electron Image

FD 168214

Figure 59. Cluster of Foreign Particles Found in AVCO No. 60 Rolled Titanium Plate (Continued)

Mag 100X

(Krolls Etch)



Mag 8X

Transverse View
(Krolls Etch)

Mag 10X

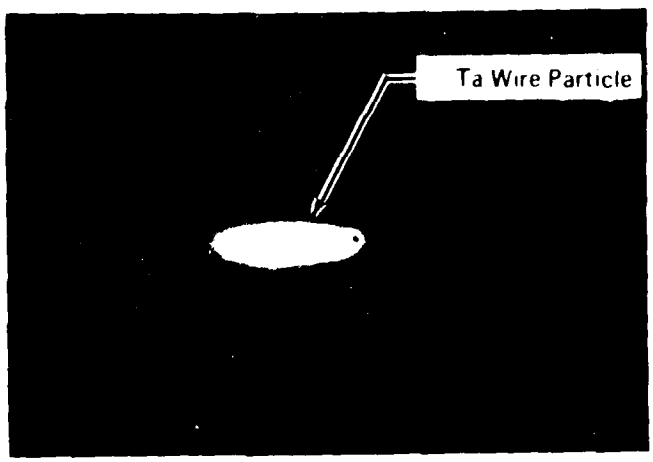
Radiograph
(Top View)

ED 171572

Figure 6b. [illegible] Enlargement Above

Mag 100X

Secondary Electron Image



Mag 100X

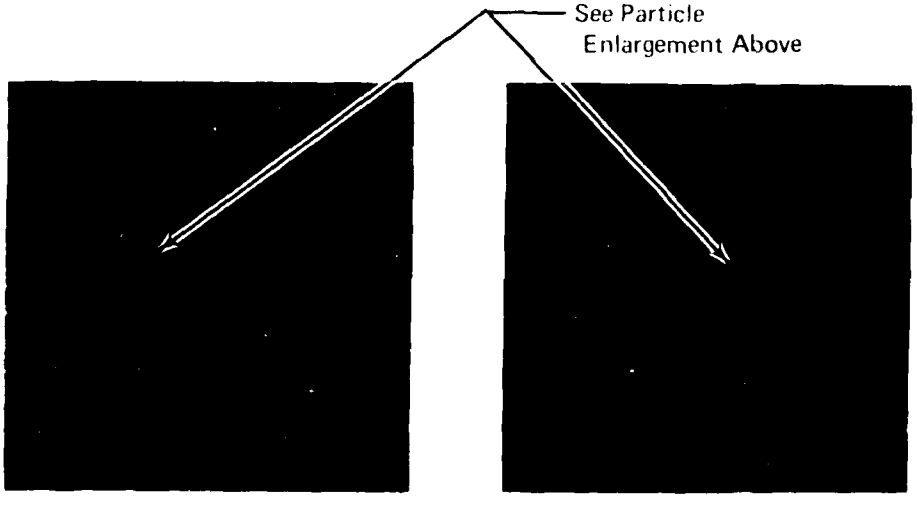
Backscatter Electron Image

FD 168215

Figure 60. Foreign Particle Found in Frankel No. 1 Rolled Titanium Plate (Continued)

Mag 100X

(Krolls Etch)



Mag 8X

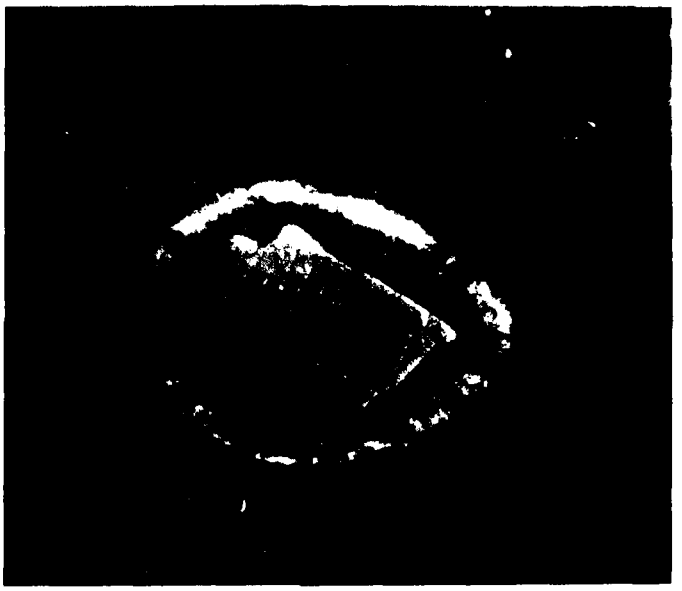
Transverse View (Krolls Etch)

Mag 10X

Radiograph (Top View)

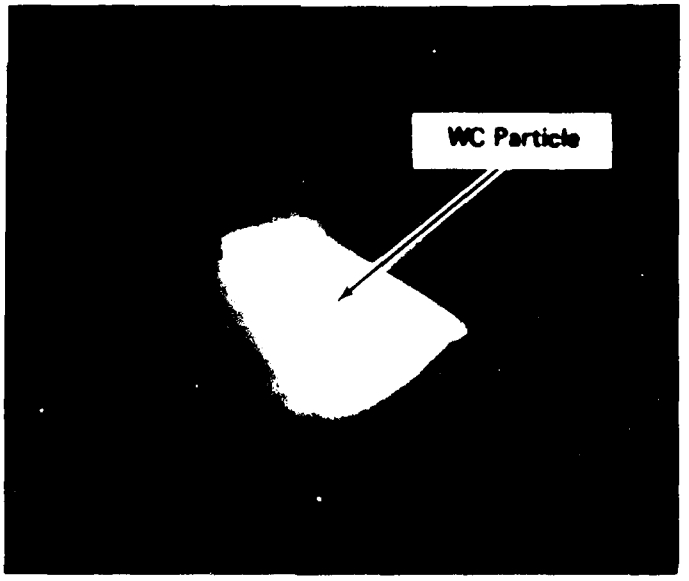
ED 171573

Figure 61. Foreign Particle Found in Frankel No. 5 Rolled Titanium Plate



Mag 100X

Backscatter Electron Image



Mag 100X

Secondary Electron Image

FD 168216

Figure 61. Foreign Particle Found in Frankel No. 5 Rolled Titanium Plate (Continued)

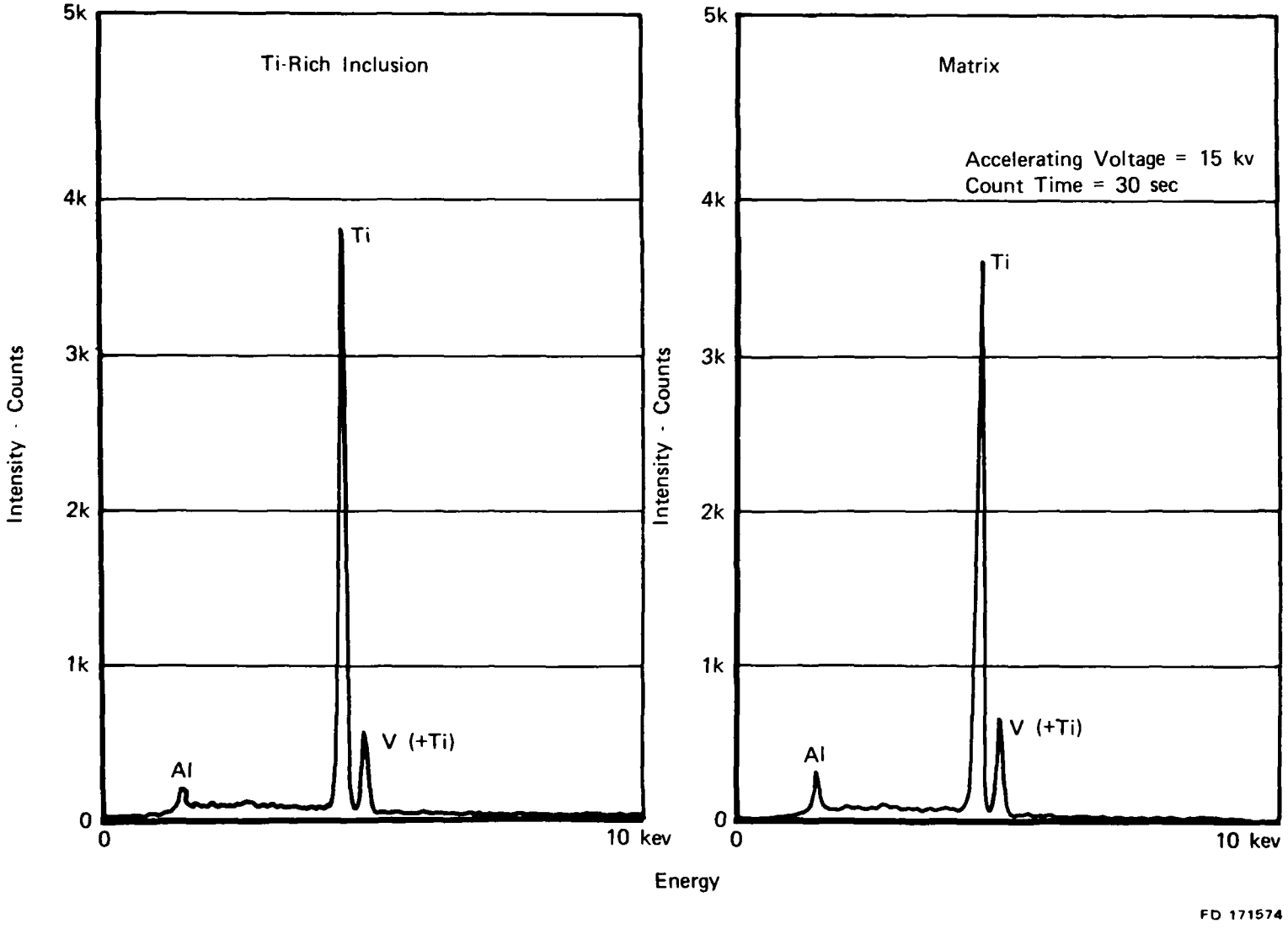


Figure 62. Inclusion and Matrix Microprobe Data from AVCO No. 6 Rolled Titanium Plate

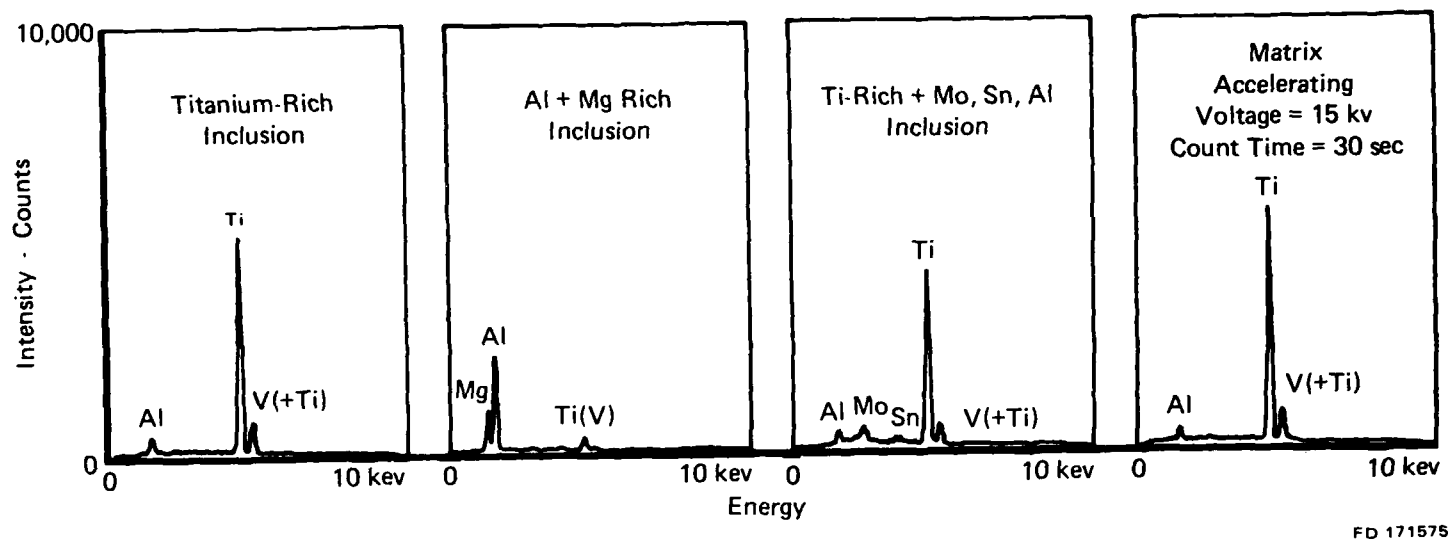


Figure 63. Inclusion and Matrix Microprobe Data from AVCO No. 6 Rolled Titanium Plate

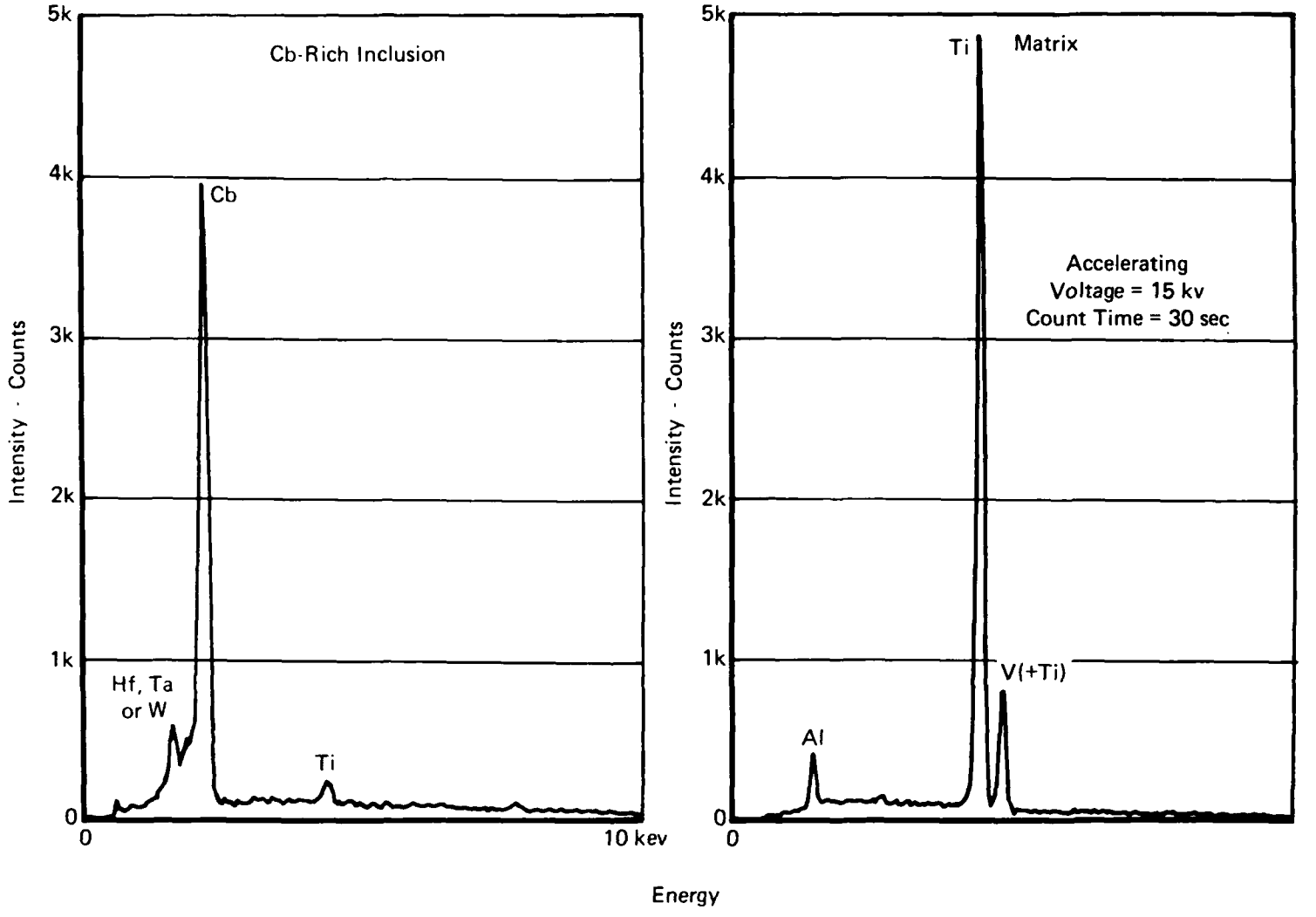


Figure 64. Inclusion and Matrix Microprobe Data from AVCO No. 58 Rolled Titanium Plate

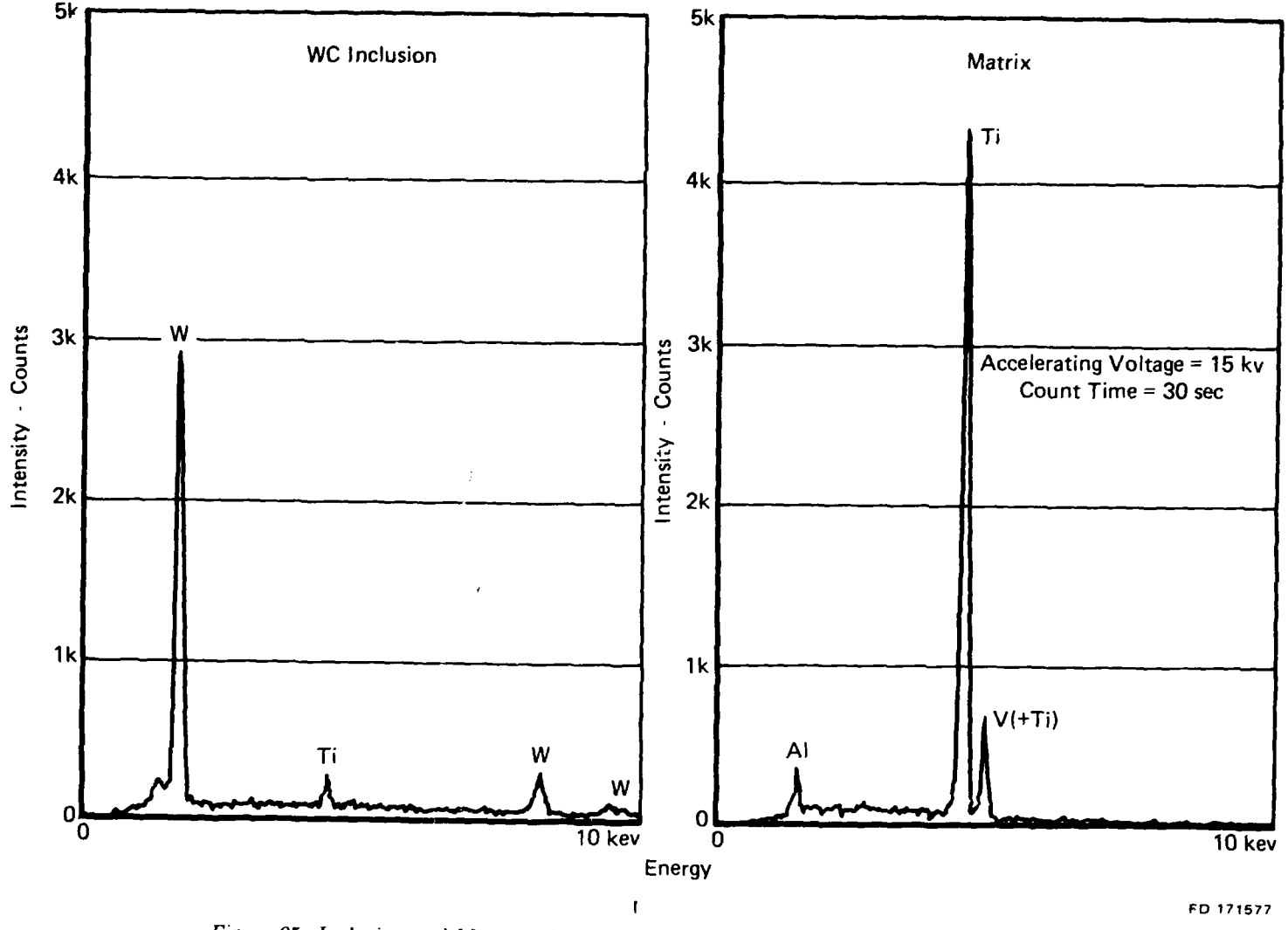


Figure 65. Inclusion and Matrix Microprobe Data from AVCO No. 60 Rolled Titanium Plate

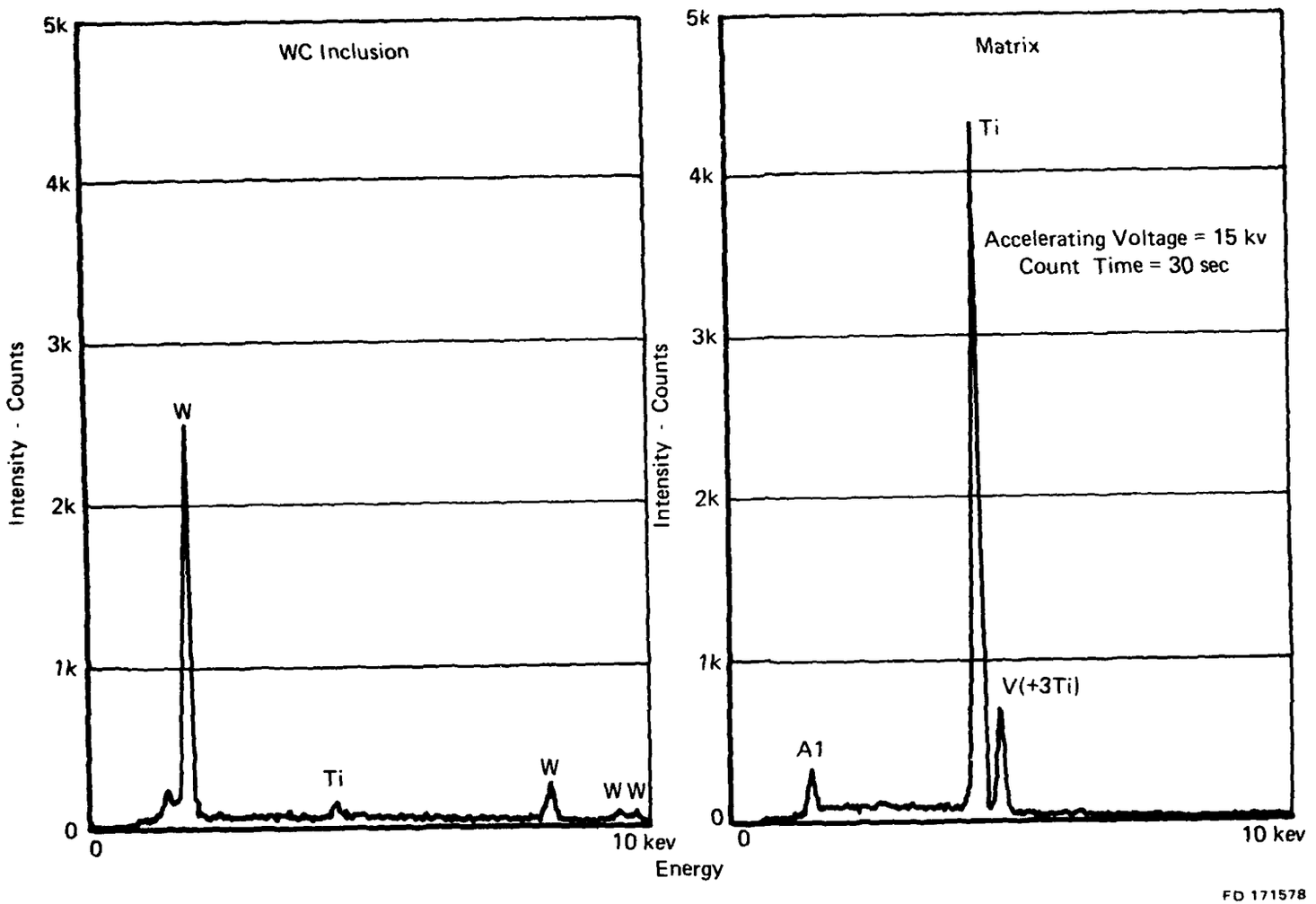


Figure 66. Inclusion and Matrix Microprobe Data from AVCO No. 60 Rolled Titanium Plate

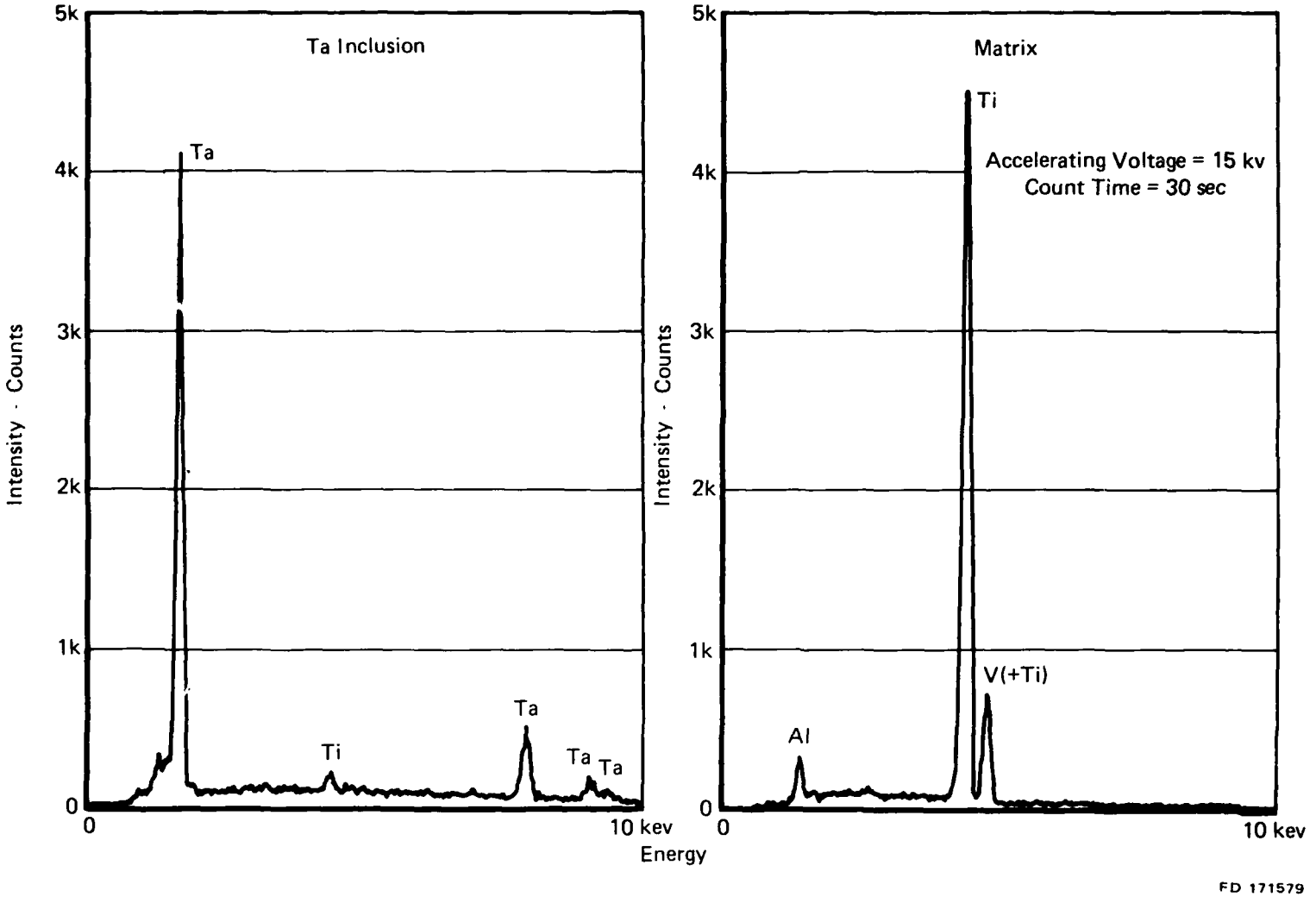


Figure 67. Inclusion and Matrix Microprobe Data from Frankel No. 1 Rolled Titanium Plate

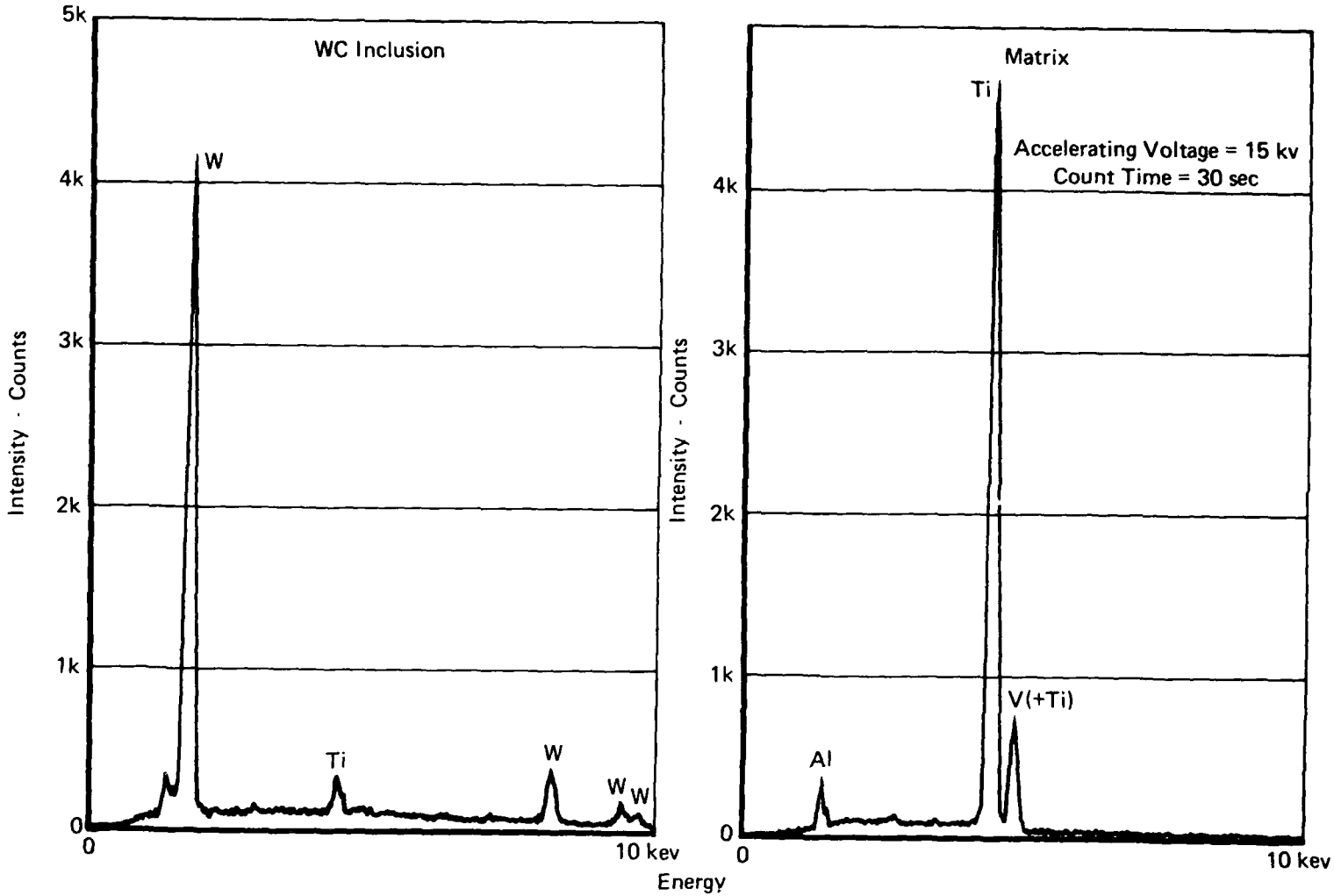


Figure 68. Inclusion and Matrix Microprobe Data from Frankel No. 5 Rolled Titanium Plate

(e) Mechanical Properties

Tensile specimens (0.190-in dia by 1.3-in. gage length) were tested according to ASTM specifications. Notched stress-rupture test specimens were machined according to AMS specification with a notch root diameter of 0.178 in. and a root radius of 0.006 in. A load was applied to produce a stress of 170 ksi.

Room temperature tensile and stress-rupture data are tabulated in Table 32 for both AVCO and Frankel fully heat-treated rolled plates. In general, these data show tensile strength levels, both UTS and YS, exceeding specification minimums and ductility, both % El and %RA, below specification minimums. Likewise, notched stress-rupture minimum lives are often below specification requirements.

TABLE 32
MECHANICAL PROPERTY DATA OF TITANIUM ROLLED PLATE

	PWA 1215 Spec. (Min)	AVCO						Frankel			
		No. 6	No. 9	No. 23	No. 58	No. 59	No. 60	No. 1	No. 2	No. 5	No. 8
RT Tensile	130	148.6	155.4	171.7	156.5	169.7	163.6	142.0	146.9	134.5	140.5
UTS (ksi)		133.6	156.0	174.3	154.0	171.2	162.5	143.2	148.8	134.3	140.2
		128.2	152.8	175.2	155.2	170.7	160.0	143.0	146.7	134.6	140.4
0.2% YS	120	145.7	149.3	168.8	150.6	163.0	159.8	134.7	139.4	128.2	132.1
(ksi)		140.7	149.3	170.4	150.5	165.5	160.2	138.0	142.1	128.3	131.7
		126.7	147.6	172.0	150.5	163.9	160.4	136.2	139.4	129.0	132.5
Elongation	10	2.6	12.3	8.0	11.7	10.0	11.3	11.0	12.2	7.7	9.3
(%)		2.6	10.1	8.0	11.0	10.8	10.4	12.2	12.0	8.4	11.7
		0.5	11.2	2.8	11.6	8.5	12.3	9.5	13.4	8.2	9.4
Rate Average	25	2.7	31.7	12.5	27.6	22.3	37.9	21.1	22.8	24.7	22.2
(%)		4.5	27.9	14.0	23.5	24.2	40.4	33.2	22.8	20.8	23.2
		–	25.8	6.2	28.0	22.0	31.7	21.4	22.9	19.0	17.3
RT Notched S/R**											
Hr life at 170 ksi	5	0.0	25.0	0.0	25.0	0.1	25.0	113.0	113.0	20.0	25.0
		0.1*	88.0	0.0*	88.0	0.2*	88.0	25.0	25.0	0.0	25.0

*Retest at 160 ksi
**< 5 Hr = Failure
>5 Hr = Test Discontinued

There are several pertinent factors that have adversely affected ductility in this program, namely forging/conversion procedure and heat treatment conditions. Forging/conversion procedure was conducted at 2000°F which is clearly above the beta transus level (Figures 52 through 54). This factor will increase strength while seriously reducing ductility. Following the forging/conversion procedure, the ¾-in. thick plate was heat-treated to the specification conditions (solution + quench + age) used for large, highly stressed components. Unfortunately, the high quench rate imposed on the relatively thin plate tends to increase the aging response leading to a decrease in ductility. Minor influences on properties may have resulted during melting procedures (i.e., sponge Ti additions without compensating alloy addition) which could adversely affect stress-rupture properties.

(3) Conclusions — Titanium Separation (Phase I)

1. Both separation processes (ferrofluid and fluidized bed) appear equally effective in removal of most contamination, but neither process can presently remove all contamination.

2. Any high interstitial or residual elements can be diluted to specification level by less than 50% virgin addition.

3. Both separation processes, as evaluated, result in a chip product containing minor, but unacceptable, levels of high density particles.

4. The unacceptable, retained high density particles should be completely removed by a subsequent nonconsumable process. This Electron Beam melting conclusion is based on nonconsumable melting data generated during a previous Air Force Contract (Rotating Electrode Melting).

(4) Review of Supplementary Data

Analyses were made on particles that were retained in the separated recovered floats of AVCO Run No. 42, 58, 59, and 60. These particles had densities similar to that of the titanium scrap being processed and thus were not removed during the ferrofluid process. The particles were located by radiography, manually removed from the chips and chemically identified. Specific identification of the particles is shown in Table 33.

Titanium analyses were made to determine the relative amount of Ti chips that are misclassified and disposed of as sinks in the AVCO process. Data are given in Table 34. Results show that the majority of Ti chips are recovered in the floats and only a small amount (1-5%) were misclassified and disposed of as sinks.

Sinks from AVCO Run No. 270 (i.e., light density contamination separation tests) seeded with 4.5 lb of Al turnings marginally exceeded the 6.75% Al maximum specification level by analyzing as 6.9% Al. In comparison, unseeded Run No. 271 contained 6.6% Al. These results imply that Al separation was very effective, but complete Al separation was not attained.

TABLE 33
COMPOSITION OF RETAINED PARTICLES IN AVCO-SEPARATED/RECOVERED FLOATS

AVCO Run No.	*Ref. Figure No. of IR-162-4-IV*	*Chemical Analysis*	*Remarks*
42	11	Pb base + Sb + Fe + Ti	Porous, low-melting "type" alloy used for fixturing
58	17	Ti base + Sn + Zr + Al + Fe + V	Porous slag of Ti base
59	18	Zr	Porous slag of Zr
60	19	Ti	Solid chunk of Ti

TABLE 34
LEVEL OF MISCLASSIFICATION IN AVCO TITANIUM SEPARATIONS

AVCO Run No.	Total Weight Chips Processed (lb)	Total Rejected Sinks: Seeded Contaminant (lb)	Total Rejected Sinks: Ti Chips (lb)	Ti Misclassification: (Ti Chips Rejected / Ti Chips Total) × 100 (%)
6	241.6	11.4	4.7	1.9
9	252.6	11.1	13.5	5.3
23	179.9	4.2	6.5	3.6
58	67.9	1.8	0.5	0.7
59	103.0	2.9	4.1	4.0
60	95.5	6.3	2.3	2.4

b. Evaluation of Superalloy Separation

Waspaloy scrap that had been artificially contaminated with lead shot and Ti alloy turnings and separated by the AVCO ferrofluid process was melted into 50 lb ingots, converted into bar and evaluated by NDI, chemistry, metallography and mechanical properties.

(1) Melting and Conversion Procedures

Two lots (Run Nos. 102 and 104) of Avco ferrofluid processed Waspaloy chips were button melted and analyzed. A 50-lb vacuum induction melt was made and cast into an electrode from each lot using standard Teledyne Allvac melting procedures for Waspaloy revert. An analysis for carbon was performed during the melt with carbon additions being made to bring the concentration of this element up to the desired level.

The cast electrodes were conditioned and vacuum consumable arc remelted into 4-in. diameter ingots. The ingots were 2100°F homogenized and forged to 2¼ in. square. The billet ends were cut, macroetched and cleaned. The forged billets were rolled to a ½-in. dia bar which was then straightened, centerless ground and etched in preparation for Zyglo and ultrasonic inspection.

(2) Analyses/Results

(a) *Nondestructive Inspection*

Fluorescent penetrant inspection by Teledyne detected minor mechanical surface defects on the centerless ground bar which were subsequently removed by spot grinding. Conventional Teledyne ultrasonic inspection of the centerless ground bar to a 2/64-in. dia flat bottom hole standard revealed no internal defects.

(b) *Chemical Analysis*

Complete chemical analysis was obtained by Teledyne from the separated chips at the bottom of both cast electrodes, and also from the top and bottom of both forged 2¼-in. square billets. P&WA performed complete chemical analysis of both rolled ½-in. dia bars, and these data are presented in Table 35. A review of these data indicate consistent correlation between subcontractor and P&WA analyses.

TABLE 35
CHEMISTRY OF WASPALOY PRODUCTS AFTER AVCO SEPARATIONS

	AVCO Run No. 102					*AVCO Run No. 104*				
PWA 7007 Spec.	*Separated Chips (Teledyne)*	*VIM Electrode Bottom (Teledyne)*	*Billet Top (Teledyne)*	*Billet Bottom (Teledyne)*	*Rolled Bar (PWA)*	*Separated Chips (Teledyne)*	*VIM Electrode Bottom (Teledyne)*	*Billet Top (Teledyne)*	*Billet Bottom (Teledyne)*	*Rolled Bar (PWA)*
Cr 18 – 21	18.60	18.68	18.60	18.70	19.1	18.60	18.58	18.64	18.45	18.5
Co 12 – 15	13.45	13.50	13.53	13.50	13.1	13.30	13.40	13.47	13.52	12.9
Mo 4.5 – 5.0	4.05	4.05	4.06	4.06	4.1	4.10	4.00	4.02	4.02	3.9
Ti 2.75 – 3.25	3.68	3.46	3.41	3.42	3.4	3.35	3.35	3.37	3.38	3.3
Al 1.20 – 1.60	1.35	1.35	1.33	1.39	1.2	1.36	1.30	1.31	1.30	1.2
Zr 0.02 – 0.12	0.06	0.06	0.06	0.06	0.07	0.06	0.06	0.06	0.06	0.07
B 0.003 – 0.010			0.004	0.004	0.006		0.004	0.004	0.004	0.006
C 0.02 – 0.10	0.046	0.046	0.038	0.038	0.04	0.047	0.045	0.038	0.037	0.03
Ni 0.25 Max	0.02	0.01	0.01	0.01	0.003	0.02	0.01	0.01	0.01	0.004
Si 0.25 Max	0.07	0.07	0.05	0.04	0.07	0.07	0.06	0.05	0.05	0.08
S 0.020 Max		0.0022	0.0032	0.0029	0.006		0.0037	0.0035	0.0026	0.01
Fe 2.00 Max	0.96	1.02	1.02	1.03	0.70	0.97	1.01	1.02	1.02	0.60
Cu 0.10 Max	0.02	0.02	0.03	0.02	0.05	0.02	0.02	0.02	0.03	0.05
Bi 0.00005 Max					0.000003					0.000003
Pb 0.00010 Max	0.00003	0.0001	0.0001	0.0004	0.0001	0.0003	0.0001	0.0001	0.0003	0.0001
Sn					0.0001					0.0001
Te					0.000003					0.000002
Tl					0.0001					0.0001
P	0.005	0.006	0.005	0.005		0.005	0.006	0.008	0.007	
W	0.02	0.03	0.03	0.03		0.02	0.03	0.03	0.03	
V	0.14	0.11	0.11	0.11		0.12	0.10	0.10	0.10	
Sn	0.0246	0.0088	0.0089	0.0097		0.0049	0.0082	0.0080	0.0076	
Cb	0.06	0.05	0.05	0.05		0.05	0.06	0.05	0.05	
O			0.0024	0.0024				0.0025	0.0039	
N			0.0020	0.0028				0.0031	0.0025	
Ni R*	R	R	R	R	R	R	R	R	R	R

R* Remainder

Both Waspaloy chip runs were seeded with 4.65% of Pb and 4.65% of Ti-5Al-2.5Sn contaminants prior to the ferrofluid separation. Analyses of Table 35 data indicates that complete removal of Pb contaminant was attained during separation, but that some Ti contaminant was retained. The analyzed range of Ti content (3.30 — 3.68%) consistently exceeded the specification maximum of 3.25%. This Ti increase cannot be attributed to contaminated raw material chips which analyzed as 2.80-3.00% Ti.

These data (Table 35) indicate an appreciable but incomplete removal of Ti seeded contaminant during the separation process of both runs. The ingot melter expressed an opinion that Waspaloy chips with the relatively minor excess of Ti found in these runs could be utilized in production melting with appropriate dilution by other raw materials.

(c) Metallographic Analysis

Typical microstructures of Waspaloy rolled bar fabricated from AVCO separated chips are shown in Figure 69 photomicrographs. Microstructures of both separation runs (Nos. 102, 104) appear identical with no defects or irregularities noted. An interesting observation is the fine, uniform grain structure (ASTM 10-11) of the rolled bar as evidenced in both longitudinal and transverse microsections. The desirable fine-grained structure implies a high degree of controlled work was applied during the conversion and rolling operations.

(d) Mechanical Properties

Tensile specimens (0.190-in. dia by 1.3-in. gage length) were tested according to ASTM specifications. Smooth (0.125-in. dia by 1-in. gage length) and notched (0.178-in. notch root diameter by 0.006-in. root radius) were machined according to AMS specification. A load was applied to produce a stress of 80 ksi at 1350°F. Room and elevated temperature tensile data and stress-rupture data are tabulated in Table 36 for AVCO fully heat-treated, rolled bars.

A review of these data showed that room and 1000°F elevated temperature tensile data as well as 1350°F notched stress-rupture data, all exceeded Waspaloy specification minimums. There were several instances of 1350°F smooth stress-rupture life and ductility that did not attain specification minimums. Reduction in these properties is consistent with P&WA Waspaloy experience that fine grain tends to increase room and elevated temperature tensile strength while reducing ductilities. Waspaloy fabricated in this program had an extraordinarily homogeneous fine grain size which could readily influence ductility and smooth stress rupture lives.

(3) Conclusions — Waspaloy Separation (Phase I)

Waspaloy Separation, Phase I conclusions are:

1. The AVCO ferrofluid separation process is very effective in removing objectionable high density materials, such as lead, from superalloys.

2. The AVCO ferrofluid separation process is capable of removing most, but not all, of the Ti contaminant in Waspaloy.

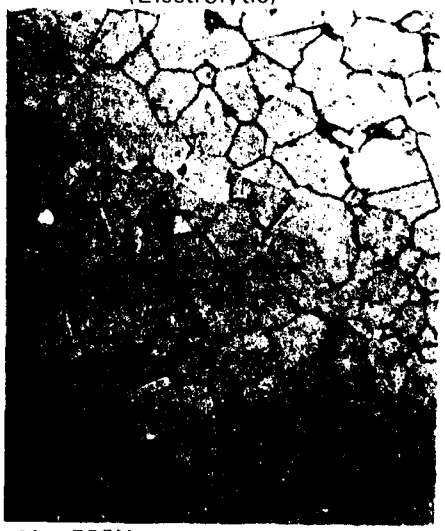
3. Process acceptability, therefore, is dependent upon practical virgin dilution requirements to attain melt specification chemistry. Acceptability would appear likely, since about a three-to-one dilution in melting of a given raw material would be the maximum acceptable limit for production melting.

4. Although partial success was noted in AVCO laboratory separation between mixed superalloys (only INCO 901 was separable from A-286, INCO 718, INCO 901 and Waspaloy mix), it is unlikely that present commercial scale ferrofluid separation between superalloys can be effectively attained.

Mag 500X
Avco No. 102 Longitudinal View
Etchant: 50% HCl/50% CH_3OH
(Electrolytic)

Mag 500X
Avco No. 102 Transverse View
Etchant: 50% HCl/50% CH_3OH
(Electrolytic)

Mag 500X
Avco No. 104 Longitudinal View
Etchant: 50% HCl/50% CH_3OH
(Electrolytic)



Mag 500X
Avco No. 104 Transverse View
Etchant: 50% HCl/50% CH_3OH
(Electrolytic)

ED 171581

Figure 69. Photomicrographs of Rolled Bar Fabricated from AVCO Separation-Processed Waspaloy Chips

TABLE 36
MECHANICAL PROPERTY DATA OF WASPALOY
ROLLED BAR

	PWA 1007 Spec. (Min)	*AVCO Bar No. 102*	*AVCO Bar No. 104*
RT Tensile			
UTS (ksi)	180	214.2	208.3
		212.8	209.1
0.2% YS (ksi)	125	157.2	151.4
		160.7	152.5
% Elongation	15	21.0	28.4
		20.4	19.5
% RA	18	44.5	48.2
		49.8	47.9
1000°F Tensile			
UTS (ksi)	160	198.8	195.5
		200.0	191.8
0.2% YS (ksi)	110	157.1	140.7
		152.2	137.7
% Elongation	15	15.7	16.8
		17.6	17.5
% RA	18	25.9	26.3
		24.2	25.0
1350°F S/R (80 ksi)			
Smooth Life (hr)*	23	29.4	38.7
		17.6	11.0
Smooth % Elongation	12	17.6	14.5
		10.1	19.0
Notched Life (hr)**	Smooth	50.0	49.0
		87.4	86.4

*Tested to Failure
**Test Discontinued

(4) Review of Supplementary Data

Analyses were made to determine the relative amount of Waspaloy chips that are misclassified and disposed of as floats during removal of "lights," and as sinks during removal of "heavies." Data presented in Table 37 shows that the original addition of light (Ti-5Al-2.5Sn) and heavy (Pb) seed contaminants to the entire lot of Waspaloy chips for subsequent portioning into test runs caused some degradation of seed contaminants. In effect, a higher ratio of seed contaminants was retained in the final chip-plus-contaminant runs than in the earlier runs. These data indicate that for effective superalloy separation, roughly an equal weight of Waspaloy chips and seeded contaminants must be removed. Also, there is evidence that a higher level of Waspaloy misclassification occurred during removal of heavies than during removal of lights.

TABLE 37
LEVEL OF MISCLASSIFICATION IN AVCO WASPALOY SEPARATIONS

AVCO Run No.	Total Weight Chips Processed (lb)	Total Rejected Floats: Seeded Contaminant (Ti-5 Al-2.5 Sn) (lb)	Total Rejected Floats: Waspaloy Chips (lb)	Waspaloy Misclassification: (Waspaloy Chips Rejected / Waspaloy Chips Total) × 100 (%)
102	180.7	2.2	2.9	1.6
103	122.5	1.0	1.9	1.6
104	114.7	3.3	5.8	5.0
105	99.0	6.6	4.4	4.4
106	100.0	16.7	16.7	16.7
		Total Rejected Sinks: Seeded Contaminant (Pb) (lb)	Total Rejected Sinks: Waspaloy Chips (lb)	
102	168.5	5.1	16.2	9.6
103	109.8	3.0	12.9	11.7
104	95.6	3.2	8.8	9.2
105	80.6	3.9	8.7	10.8
106	62.1	10.4	8.2	13.2

(5) Separation of Mixed Superalloys

An experiment was conducted to determine the capability of the AVCO ferrofluid process to separate one superalloy from another, specifically, superalloys A286 (Tinidur), INCO 901, INCO 718 and Waspaloy. Flotation characteristics were investigated in a small AVCO laboratory magnet. Although these alloys differ only slightly in true density, it was postulated that the differences in the magnetic properties of the various alloys might permit a separation according to an effective magnetic density. Pertinent data are given in Table 38.

The magnetic contribution $\Delta\rho_m$, to the apparent density of the material is given by:

$$\Delta\rho_m = \frac{\mu H - 1}{4\pi} \quad \frac{H\Delta H}{g}$$

where

$\Delta\rho_m$ = increase in apparent density (g/cm³),
H = magnetic field level (oersted),
ΔH = magnetic field gradient (oersted/cm),
g = acceleration of gravity (cm/sec²).

Calculations of $\Delta\rho_m$ and the effective density, ρ_e, of the material based on the handbook values of permeability, the predicted magnet properties for AVCO's large magnet (H = 5000 oe and ΔH = 200 oe/cm) and the small laboratory magnet (H = 5000 oe and ΔH = 500 oe/cm) are listed in columns 3, through 6 of Table 38. These values indicate that sink-float separation may be possible between certain components and that separability is enhanced by a high magnetic field gradient.

TABLE 38
MAGNETIC PROPERTIES OF SELECTED SUPERALLOYS

Column No	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*
Alloy	*Actual[1]* Density gm/cm³*	*Permeability[1]* μ_{max} at 200 oe*	*$\Delta\rho_{sa}$ Calculated μ_{max} H=5000 oe ΔH=200 oe/cm gm/cm³*	*$\rho_{effective}$ Calculated μ_{max} H=5000 oe ΔH=200 oe/cm gm/cm³*	*$\Delta\rho_{sa}$ Calculated μ_{max} H=5000 oe ΔH=500 oe/cm gm/cm³*	*$\rho_{effective}$ Calculated μ_{max} H=5000 oe ΔH=500 oe/cm gm/cm³*	*Permeability[2]* μ5000 at 5000 oe*	*$\Delta\rho_{sa}$ Calculated μ_{max} H=5000 oe ΔH=500 oe/cm gm/cm³*	*$\rho_{effective}$ Calculated μ_{max} H=5000 oe ΔH=500 oe/cm gm/cm³*	*Actual ρ_a for Flotation*
A 286	7.92	1.01-1.05	0.81-4.1	8.7-12	2.02-10.1	9.9-18.1	1.003	0.61	8.53	8.9
INCO 901	8.23	1.008	0.65	8.9	1.62	9.85	1.002	0.41	8.64	11.6
INCO 718	8.21	1.001	0.08	8.3	0.20	8.41	1.005	0.20	8.41	8.8
Waspaloy	8.20	1.012	0.97	9.17	2.43	10.60	1.003	0.06	8.26	8.8

$$\rho_{sa} = \frac{\mu H^{-1}}{4\,\Pi} \quad \frac{H\,\Delta\,H}{g} \quad \text{(Magnetic Contribution)}$$

$$\rho_{effective} = \rho_{material} + \rho_{sa} \quad \text{(Effective Density)}$$

Notes* 1. From Aerospace Structural Metals Handbook, Volume 4 1972 Publication AFML-TR-68-115.
2. AVCO data.

Flotation tests were done at a high magnetic field gradient to maximize the expected density differences. The results in column 10 of Table 38 do not agree well with the calculated data. A possible explanation is that the permeability is a strong function of the magnetic field strength and that data for 200 oe do not apply for 5000 oe. Consequently, the permeabilities of the materials were measured at 5000 oe (column 7, Table 38). These values of permeability were much smaller than handbook values although still not in agreement with calculated effective densities of the materials (columns 8 and 9, Table 38). Empirical observations of the materials in the magnet gap at a field strength of about 5000 oe confirm that the material requiring the highest ferrofluid density to float, INCO 901, is most strongly attracted to the magnet poles. It is likely that the permeability data for 5000 oe is in error.

It appears that INCO 901 may be separable from the other superalloys using the high intensity, laboratory scale magnet. Separation of other superalloys was not successful. Since separation capabilities with this laboratory magnet were, therefore, limited, and the commercial scale magnet would provide a smaller, useful gradient and further reduce separation capabilities, it was decided to terminate further studies of separations between superalloys.

C. FRANKEL COMPANY MOLTEN SALT CHEMICAL PURIFICATION PROCESS

1. Background

Molten salt baths are commonly used in the heat treatment and surface treatment of superalloys. Ordinarily these baths contain metal hydroxides, chlorides and other compounds in various vendor proprietary mixes. A molten salt bath chemical process, co-developed by Frankel and an affiliate company was being investigated for purification of nickel-base superalloy grindings and sludges. The process was evaluated for its potential to reclaim segregated grindings for subsequent melting. The highest potential for recovery of sludges by this process would be a pure multielement master alloy. The molten salt purification process has been demonstrated to be efficient on a small scale but has not been evaluated under full-scale industrial conditions.

In limited experimental work, the Frankel Company had flame-heated superalloy grindings and sludges passing the resultant gases through a molten salt bath to remove combined water, oil and other organic substances. The molten salt directly and catalytically converts carbon and sulfur base compounds without the generation of major polluting gas or liquid residues which would be encountered in other processes. The product effluent from the molten salt system is a dry particulate substance containing metallic and inorganic compounds, especially grinding media. These inorganic particles can be separated from the metallic particles by gravity separations similar to the ones utilized in the ore industry. The thus purified metal particles are sufficiently pure to be remelted in furnaces. In cases of certain metallic sludges where the metallic content (mainly nickel content) is in oxide form, these oxides can be reduced to metallic form in regular smelting furnaces.

The system was originally conceived as an improved method to dispose of common municipal waste products. Conventional town procedures of incineration have always generated undesirable, noxious combustion products. Combined citizen and government concern for ecology and health standards recently resulted in rigid incineration controls which often declare this mode of disposal illegal. Emphasis has been shifted to landfill, land reclamation, ocean dumping and composting.

TABLE 39
TYPICAL PARAMETERS OF TITANIUM MELTING PROCESSES

	Consumable[a] Vacuum Arc	*Nonconsumable[b] Vacuum Rotating Arc*	*Nonconsumable[c] Plasma Arc*	*Nonconsumable[d] Electron Beam*	*Nonconsumable[e] Electron Beam*
Starting Material	75% sponge electrode 25% max scrap and solids	Scrap, sponge, master alloy	Scrap, sponge, master alloy	Scrap chips	Scrap chips, sponge, master alloy
Alloy Control During Melt	Difficult	Function of blending	Possible	Possible	Being determined by spectro
Alloy Element Additions	Difficult	None	Possible	Possible	Possible
Remelts Required	Yes	VAR	None anticipated	Yes	None anticipated
Temperature	—	—	Strong beam concentration	Can superheat surface	Can superheat surface
Vacuum	$<10^{-4}$ torr	5-25 torr argon	10^{-3} to 10^{-1} torr argon	10^{-4} to 5×10^{-3} torr	10^{-4} to 10^{-3} torr
Melt Rate	to 4000 lb/hr	600-1200 lb/hr	800-1200 lb/hr	80 lb/hr	200-550 lb/hr
Melt Energy	<0.5 KWH/lb	0.9 KWH/lb est	3 KWH/lb est	1-3 KWH/lb	0.95 KWH/lb
Interactions:					
Crucible	None	Skull melt	None	None	None
Slag	—	—	—	—	—
Electrode	—	—	—	—	—
Volitalization:					
Impurities	Small	Very small	Some reduction	Yes	Yes
Alloying Elements	Small	Small	Yes	Yes (Al problem)	Yes (Al problem)
Degassing:					
O_2, N_2	Small increase	Small increase	Small increase	O_2 increase	Adequate
H_2	Good reduction	Some reduction	Good reduction	Adequate	Adequate
Ingot:					
Weight	20,000 lb	10,000 lb	4,200 lb	Pellet is 2-4 gms	3,300-10,000 lb
Surface	Adequate	Requires VAR	Approx. continuous cast	—	Excellent
Homogeneity	Adequate	Adequate	Adequate	—	Dependent on feed system
Segregation	No heavy element removal	Partial heavy element removal	Heavy elements removed	Heavy elements removed	Heavy elements removed
Structure	Medium grain size	Refined by remelting	Approx. continuous cast	—	Small/Medium, equiaxed

(a) Conventional furnace
(b) Schlienger-Teledyne Allvac
(c) Schlienger
(d) Leybold-Heraeus-RMI Co
(e) Airco Temescal

The molten salt system offers the advantages of incineration without the production of noxious stack gases. These advantages include:

1. 90% weight reduction to inert, mineral ash
2. Reclamation of metals and glass
3. Reclamation of organic materials by operation as a pyrolysis unit (i.e., nonoxidizing environment)
4. Heat production/reclamation for heating or power generation.

The molten salt bath process also offers the advantage of low capital investment. It was chosen for the program because it is a relatively simple single step operation and was one of the few alternatives to economical processing of grindings and sludges.

An incinerator may be considered a chemical reactor whereby materials are primarily combined with oxygen. As in most reactions, the efficiency can be increased with a catalyst. Conventional catalysts are finely divided solids that need large surface exposures and redundant systems. Since a liquid theoretically has an infinite surface, a liquid catalyst would be more efficient than conventional solid catalysts which have limited life due to surface poisoning. Liquid salt serves as the inexpensive liquid catalyst in this process. The salt is thermodynamically stable and appears to have a relatively unlimited service life.

2. Description of Process

A schematic drawing of the molten salt system is shown in Figure 70. The system consists essentially of a "reaction chamber" inner box floating upon a bed of molten salt that is confined within an outer box. Metal grindings and/or sludges are poured into open trays and the trays placed into the inner box (Figure 70). Incineration is initiated by ignition of the gas burner located at the top of the inner box. Combustible organics, machine oil and waste are converted to flue gas which is ducted into and through the molten salt bed. Flue gas is "scrubbed" clean of noxious elements for safe disposal to the enviornment while metal is accumulated as ash residue in the open trays. Scrubbing of these noxious, incinerated combustion products as they pass through the molten salt satisfies air ecology considerations, while the resulting gas contains mineral products of potential reclamation value.

Heat, generated by preheating and during initial combustion of waste, melts the solidified salt bed whereupon the system becomes self-sustaining. The salt has a high heat capacity, and as additional heat is generated during waste combustion the heat is absorbed by the 1100°F salt. As a result, the use of supplemental fuel to sustain the incineration is negligible. Appropriate air inlet, air bypass and flue outlet manifolds are provided (Figure 70) to maintain process control. Figure 71 shows a photograph of the molten salt incinerator system as evaluated. An incineration cycle processing about 10 ft^3 of grindings or sludge can be performed in 4 hr. The dry metal ash residue, containing only metallic and inorganic particles, is then pulverized in a conventional ball mill. Inorganic particles are then separated from the metallic particles by gravity separations, i.e., Murphy Table, similar to procedures utilized with ores by the extractive metallurgy industry. The resulting purified metal product, although a mixture of unknown metal composition, can be induction-melted to ingot stock. This product should be a useful reclaimed material for semicritical melting operations, i.e., the automotive, steel and stainless industries.

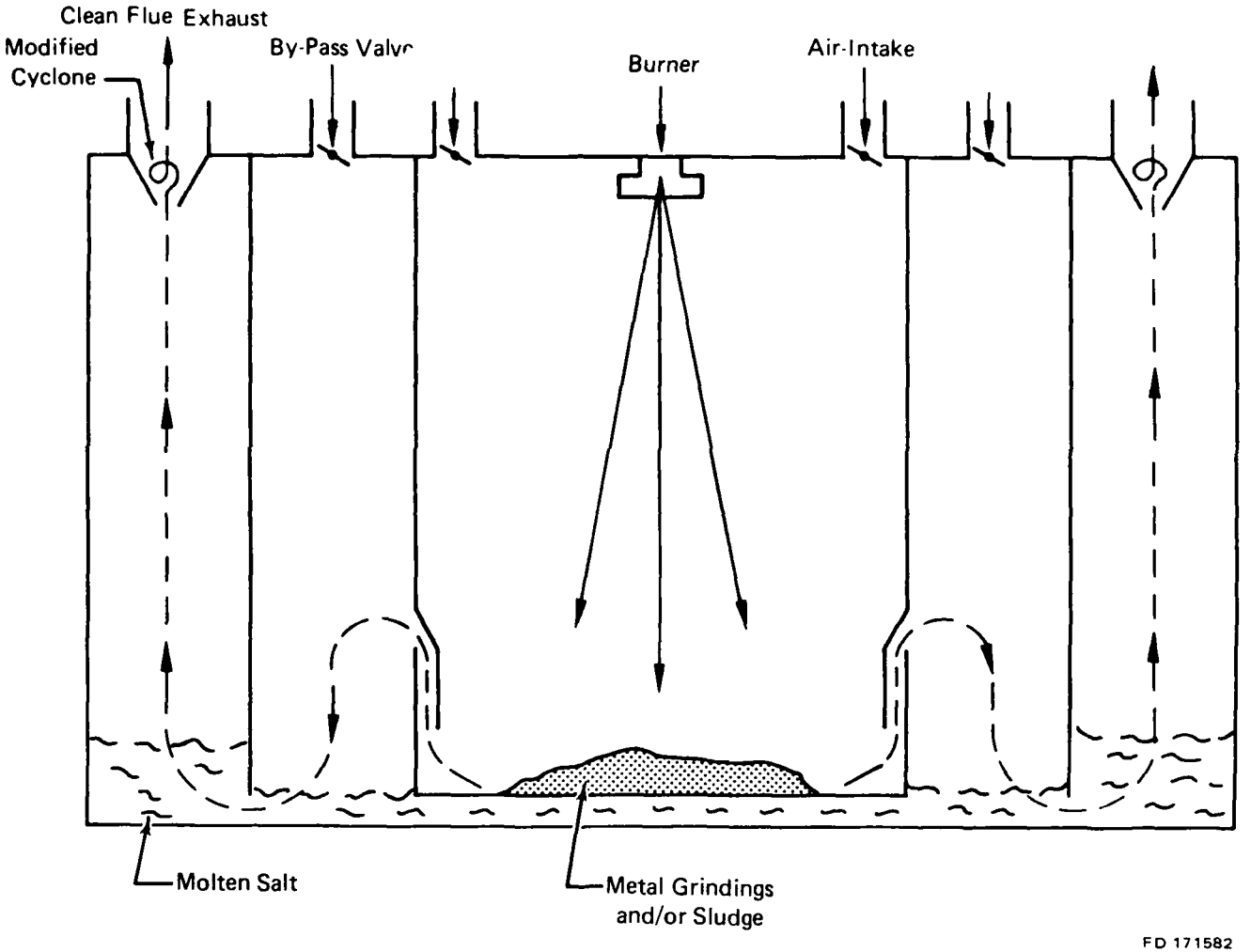


Figure 70. Schematic Drawing of Frankel Molten Salt System

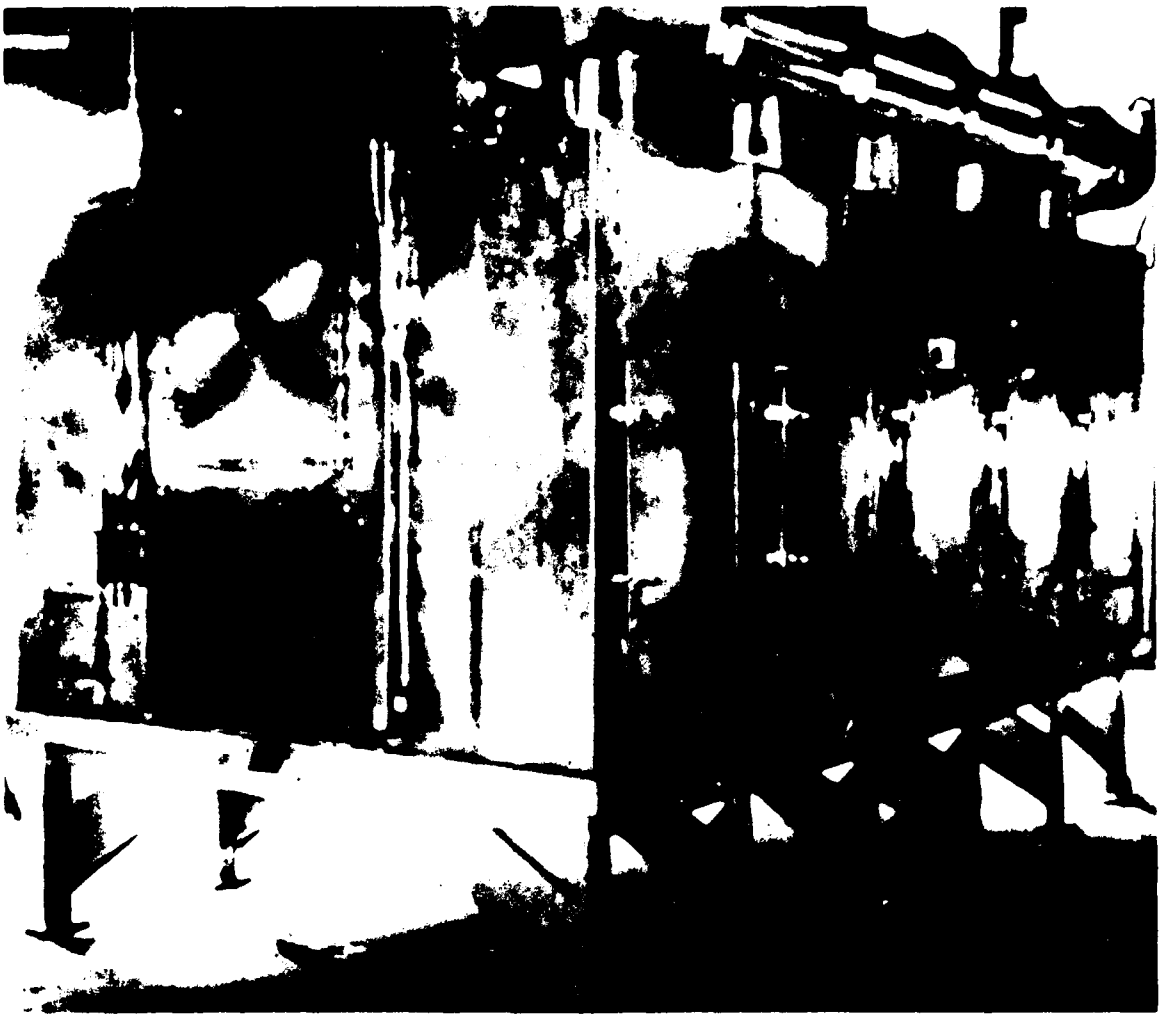


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Figure 71. Frankel Molten Salt System Incinerator

3. Program Plan Details (Phase I)

Molten salt bath experiments planned by Frankel are shown in Table 40. Fourteen lots of 100 lb each were to be processed. Two different types of grindings and sludges were used. The processing steps included molten salt bath purification, particle size reduction, concentration of metallics and melting.

4. Preliminary Test Results

ECM sludge from P&WA was evaluated by Frankel and found to be unsuitable for recovery because the elements present in the sludge are present in compound form but are not in metallic form. Free metal containing sludges was applied to the process.

TABLE 40
FRANKEL SALT BATH PURIFICATION SYSTEM
TEST VARIABLES

Test Run Number	*Material*	*Melting*
1	Grindings A	Air Induction
2	Grindings A	Air induction Blowing
3	Grindings A	Vacuum Induction
4	Grindings B	Air Induction
5	Grindings B	Air Induction Blowing
6*	Grindings A	Air Induction
7	Sludge A	Air Induction
8	Sludge B	Air Induction
9	Sludge A	Air Induction Blowing
10	Sludge B	Air Induction Blowing
11	Sludge A	Method to be Selected
12	Sludge B	Method to be Selected

Runs 13 and 14 are reserved for variation in salt bath composition.

*Concentrations of metallics omitted.

Later, trial runs were conducted by Frankel using subscale molten salt bath metal purification equipment. Test results showed that the subscale equipment was too inefficient, even for the relatively small lot sizes (100 lb) planned for Phase I of the program. The subscale equipment, including a small size waste material container, required several hours to process a single 100 lb lot. On the basis of the trial tests with subscale equipment, plans were adopted to conduct the Phase I runs on full-scale equipment. This machinery was originally scheduled for use in Phase II, but its fabrication was accelerated so that it could be used in Phase I. This change of plans resulted in a delay in this portion of the program.

5. Separation Test Program

a. Description of Effort

Separation experiments were conducted on the two batches (5500 to 1000 lb/batch) of nickel-base grindings (wet and partially wet) and two batches (500 to 1000 lb/batch) of nickel-base sludge (chemically different) that had been processed in the molten salt system. Separations in the Wilfrey Table concentrator were unsuccessful. Retained abrasive contaminants were adherent to the metallic residue and light/heavy separation on this inclined water table was impractical. Attempts to effect separation by air and vacuum induction melting were also unsuccessful. Poor inductive coupling occurred due to the large volume of abrasive contaminant and true melting was not obtained. Use of a steel "starter" pad did not improve induction melting characteristics. Likewise, nonconsumable arc melting was only marginally successful in effecting a separation. Constant slagging of the abrasive contaminant tended to insulate the molten pool and extinguish the arc.

Separation of metallic particles from abrasive compounds, i.e., Al_2O_3, SiC etc., was finally achieved in a Lectromelt No. 0 electric arc furnace at Exemet, Inc., of Greenville, Pa. This furnace, rated at 350 lb maximum load, is of conventional three-phase carbon electrode design.

The furnace lining was initially "cleaned" by arc melting a 300 lb wash-heat of iron sheet clippings. After pouring the iron wash-heat into a sand mold, the furnace was charged with 300 lb of molten salt bath residue from one of the nickel-base sludge batches. This charge was not sufficiently conductive to sustain the furnace arc. Approximately 30 lb of iron sheet clippings were thereupon added to the charge to initiate melting. Minor additions of silicon and fluorspar were also made to the furnace charge to increase fluidity. Separation of the heavier metallic elements from the less dense slag of contaminant abrasives is enhanced with increased fluidity.

Upon completion of melting, plus a 15-min holding period to permit slag/metal separation, the molten metal was poured into a sand mold to yield a pancake-shaped ingot.

The remaining sludge batch (of differing chemical composition) was melted in a similar procedure, and the wet grindings batch was melted in the same way except that this batch was electrically conductive and did not require the addition of iron sheet clippings to initiate melting. An additional quantity of silicon was added to this charge to influence fluidity. Lastly, the dry grindings batch was melted in a similar procedure as all previous batches, with minor additions of silicon and fluorspar. The contaminant slag readily separated from the molten metal in all four melts; detachment of the brittle slag from the solidified ingots was easily attained by hammer impact.

In a final attempt toward upgrading the quality of the recovered metal product from Ti grindings and sludges, a portion of this product was remelted by vacuum induction melting. Specifically, a 15 lb portion of each of the electric arc, air-melted ingots of each of the grinding and sludge batches were vacuum-induction remelted and recast into cast iron ingot molds.

b. *Material Balance — Phase I Molten Salt Process*

Data enabling a materials balance (input versus output) for the Frankel Co. molten salt bath processing of two batches of nickel-base sludge (chemically different) and two batches of nickel-base grindings (wet and dry) are given in Table 41. The molten salt bath facility and a typical tray of as-received sludge, prior to salt bath processing, are shown in Figures 71 and 72 respectively.

Volatiles (moisture) and obnoxious combustibles (oils) were removed from the grindings and sludges to EPA standards of smoke contamination by molten salt bath processing. A high level of nickel recovery (92 to 96%) was determined during this molten salt bath processing.

c. *Material Balance — Phase I Pyrometallurgical (Melting) Process*

A materials balance (input versus output) is presented in Table 42 data for the Exomet, Inc. electric arc melting process that was utilized to convert the molten salt bath product of all four batches of grindings and sludges into cast ingot. Due to the small (350 lb) furnace size, a finite amount of metal was retained in the furnace lining after each melt thereby reducing the accuracy of the recovery measurements. The true recovery would be slightly higher than 86 to 92% as reported in Table 42.

d. *Chemical Analyses — Phase I Processed Grindings and Sludges*

Pertinent chemical analyses (volatiles, combustibles and elementals) were performed during the processing steps of all four batches of grindings and sludges. Specifically, analyses were made of the as-received condition, molten salt bath residue, electric arc melted ingot and vacuum induction remelted ingot. Resultant data are presented in Tables 43 through 46.

e. *Analysis of Phase I Data*

In these four runs, metal recovery was high; an appreciable cost-effectiveness would be realized by the 25 to 45% recovery obtained. However, it may not be prudent to factually analyze these recovery data (Tables 41 through 46) as representative of all grindings and sludges and thereby arrive at firm process conclusions.

TABLE 41
MATERIAL BALANCE OF MOLTEN SALT BATH PROCESS

History				
Run No.	1	2	3	4
Material	Sludge No. 1	Sludge No. 2	Grindings No. 1	Grindings No. 2
Condition	—	—	Wet	Dry
Source	Custom Tool	Custom Tool	Special Metals	Special Metals
Input				
Weight (lb)	600.0	600.0	500.0	500.0
Oil (%)	35.8	7.67	5.77	0.5
Moisture (%)	0.9	1.43	15.33	0.1
Nickel (%)	29.3	45.0	24.8	50.6
Ni Weight (lb)	176.0	270.0	124.0	253.0
Output				
Weight (lb)	360.0	520.0	380.0	480.0
Nickel (%)	44.9	48.3	30.9	50.7
Ni Weight (lb)	162.0	251.0	117.0	243.0
Recovery				
Ni Recovery (%)	92.0	93.0	95.0	96.0

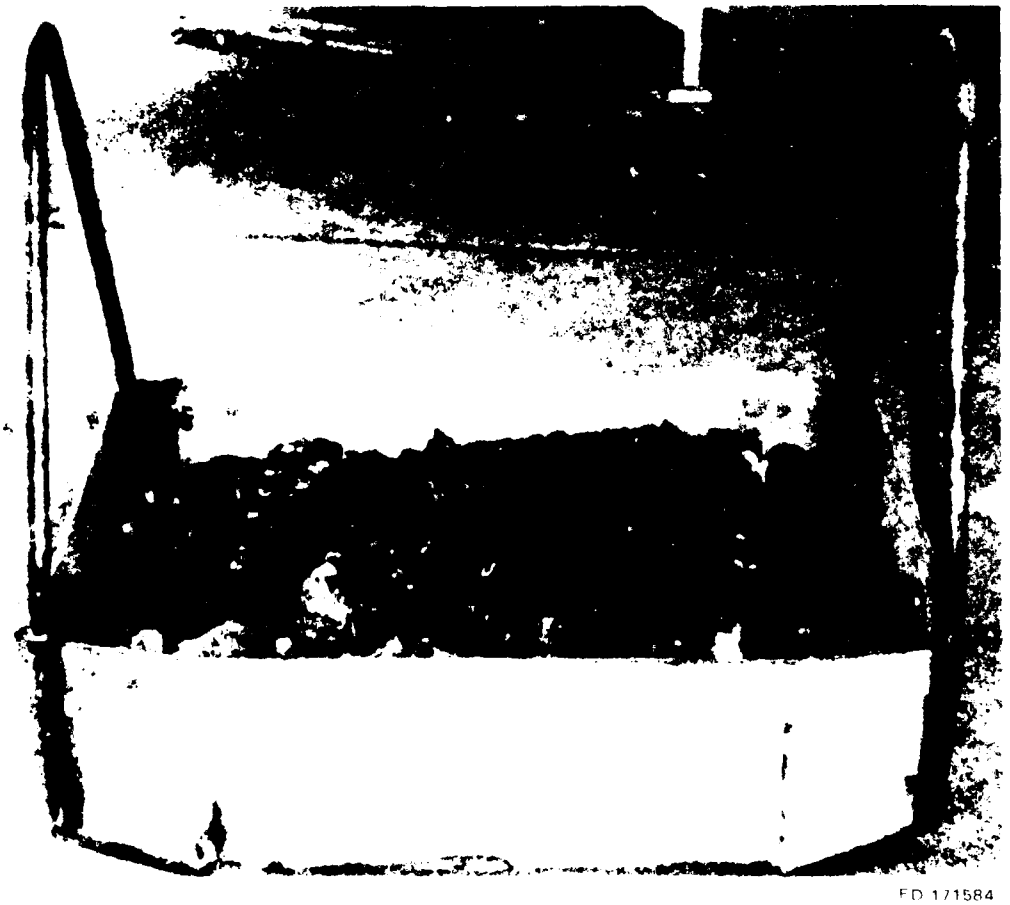

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Figure 72. Typical Tray of As-Received Ni-Base Sludge Prior to Molten Salt Bath Processing

TABLE 42
MATERIAL BALANCE OF ELECTRIC ARC FURNACE MELTING PROCESS

History				
Run No.	1	2	3	4
Material	Sludge No. 1	Sludge No. 2	Grindings No. 1	Grindings No. 2
Condition			Wet	Dry
Source	Custom Tool	Custom Tool	Special Metals	Special Metals
Input				
Weight (lb)	300.0	300.0	300.0	300.0
Nickel (%)	44.9	48.3	30.9	50.7
Ni Weight (lb)	135.0	145.0	93.0	152.0
Output				
Weight (lb)	184.0	192.0	156.0	242.0
Nickel (%)	65.2	66.2	54.0	58.5
Ni Weight (lb)	120.0	127.0	84.0	140.0
Recovery				
Ni Recovery (%)	89.0	86.0	90.0	92.0

TABLE 43
CHEMICAL ANALYSES OF PROCESSED RUN NO. 1, SLUDGE BATCH NO. 1, CUSTOM TOOL

	As-Received Condition	*Molten Salt Bath Residue*	*Electric Arc Melted Ingot*	*Vacuum Induction Remelted Ingot*
Analysis (w/o)				
Oil	35.8			
Moisture	0.9			
Ni	29.3	44.9	65.2	65.8
Co			3.0	3.24
Cr			6.51	6.60
Cu			0.28	0.26
Fe			21.1	21.1
Mn			0.07	0.09
Mo			0.65	0.61
Si			0.29	1.02
Ti			< 0.05	< 0.05
V			< 0.05	< 0.05
W			< 0.1	< 0.1
C			0.54	0.53

TABLE 44
CHEMICAL ANALYSES OF PROCESSED RUN NO. 2, SLUDGE BATCH NO. 2, CUSTOM TOOL

	As-Received Condition	*Molten Salt Bath Residue*	*Electric Arc Melted Ingot*	*Vacuum Induction Remelted Ingot*
Analysis (w/o)				
Oil	7.67			
Moisture	1.43			
Ni	45.0	48.3	66.2	65.0
Co			2.95	1.41
Cr			6.59	9.71
Cu			0.28	0.24
Fe			21.2	18.4
Mn			0.08	0.17
Mo			0.67	0.55
Si			0.38	1.68
Ti			< 0.05	< 0.05
V			< 0.05	< 0.05
W				
C			0.10	0.18

TABLE 45
CHEMICAL ANALYSES OF PROCESSED RUN NO. 3, GRINDINGS BATCH NO. 1, SPECIAL METALS

	As-Received Condition	*Molten Salt Bath Residue*	*Electric Arc Melted Ingot*	*Vacuum Induction Remelted Ingot*
Analysis (w/o)				
Oil	5.77			
Moisture	15.33			
Ni	24.8	30.9	54.0	55.1
Co			13.2	12.1
Cr			9.82	9.72
Cu			0.06	0.08
Fe			4.7	5.9
Mn			0.09	0.10
Mo			3.9	3.9
Si			10.08	9.12
Ti			2.25	2.05
V			< 0.05	< 0.05
W			0.1	0.1
C			1.47	1.74

TABLE 16
CHEMICAL ANALYSES OF PROCESSED RUN NO. 4, GRINDINGS
BATCH NO. 2, SPECIAL METALS

	As Received Condition	*Molten Salt Bath Residue*	*Electric Arc Melted Ingot*	*Vacuum Induction Remelted Ingot*
Analyses (w/o)				
Oil	0.5			
Moisture	0.1			
Ni	50.6	50.7	58.5	56.3
Co			13.5	14.1
Cr			16.7	17.1
Cu			0.04	0.05
Fe			1.95	2.70
Mn			0.03	0.04
Mo			3.90	3.80
Si			2.30	2.55
Ti			0.81	0.85
V			0.03	0.05
W			0.1	0.2
C			0.51	0.49

Two pertinent criteria exist that influence the ultimate cost effectiveness, namely, (a) the compositional grade of the as received grinding/sludge, and (b) the quantity and type of elemental contaminants retained in the melted ingot. These criteria are discussed in the following paragraphs.

(1) Grinding/Sludge Input Composition

These Phase I test runs originated with commercially generated grindings and sludges of little/no economic value. No attempt was made to select the composition or quality of the input materials except that a reasonable level of superalloy content should be present. It will be *difficult, maybe impractical,* to effect a scrap management system on grindings and sludges since industrial production, collecting and handling is far less sophisticated as compared to the area of scrap turnings and solids. As the input composition of grindings and sludges can vary over a wide range, the recovered value of the melted product will vary accordingly. Input material that is "lean" in desirable nickel content will yield a reduced level nickel ingot product and have a corresponding reduction in cost effectiveness. Therefore, process economics will be affected by specific input material compositions and must be treated accordingly.

(2) Retained Contaminants in Output Ingot

The market value of the process output ingot will be influenced by the quantity and type of elemental contaminants retained in the melted ingot. Fe is usually not of concern; however, ingot containing even a minor level of cobalt will not be useful for alloying additions in the stainless steel industry although it may have usage in the cobalt based alloy industry. Appreciable W levels are undesirable; both Co and W are not readily removable. Some elements, when present in objectionable levels, may be reduced by air lancing during melting, i.e., Cb, Al, Ti, Si, C, etc.

f. Phase II Direction — Chemical Purification Process (Molten Salt Process)

Phase I efforts, substantiated by data, realized economic potential from a grinding sludge scrap product that is presently considered relatively worthless. Separation (molten salt and melting) effort was made in Phase I on developmental quantities to establish process feasibility. Additional processing, including quantity scale up was required to establish process economics. On this basis, P&WA pursued Ni base grinding and sludge reclamation during Phase II contract activities as originally scheduled.

D. NONCONSUMABLE MELT PROCESSES

Two nonconsumable melt processes were investigated: the AIRCO-Temescal electron beam (EB) cold hearth melting system, and the Teledyne-Schlienger rotating-electrode system. These systems each have the potential to remove high density contaminants by entrapment within a melt-skull, and each provides a capability for introduction of large quantities of scrap into the melt. The EB cold hearth system was applied successfully to the reclamation of Ti-5Al-2.5Sn scrap for a commercial product not requiring chemistry control. The capability of this process to produce controlled chemistry, aerospace quality ingot from Ti-6Al-4V scrap input material was assessed in Phase I. Evaluation of the Teledyne nonconsumable melt process was based on results of Air Force Contract F33615-72-1126.

1. AIRCO-Temescal Electron Beam Cold Hearth Melting Method

AIRCO-Temescal established a pilot process for reclamation of Ti scrap by EB cold hearth melting. EB hearth melting offers the potential of overcoming many of the limitations encountered in alternative melting processes. The most expedient method for trace element removal lies with better vacuum and higher temperatures. From a technical and economical point of view, an increase in both parameters can be achieved with electron beam hearth refining as compared to other methods. This process can take place as drip melting, continuous hearth refining, zone refining or batch melting, and is applicable to both titanium and superalloys. The application of EB melting in this program was confined to hearth refining, since this is the most economical process for large-scale commercialization.

a. System Technique

AIRCO-Temescal uses an advanced refining technique for scrap metal reclamation. The technique features two sequential operations: (1) electron beam (EB) melting of feedstock in a copper, water-cooled hearth, and (2) pouring and solidification of ingots in a water-cooled mold, both operations are conducted in a hard vacuum atmosphere. In order to melt titanium alloys to AMS specification, a mixture of Ti scrap, sponge and alloying elements is continuously fed to the shallow hearth and melted by a high power electron beam. The resulting molten pool is cradled within a "skull" that forms on the water-cooled hearth.

Purification reactions and homogeneous alloying occur in the molten pool at a pressure of 0.1 to 1μ (10^{-4} to 10^{-3} torr), which is an order of magnitude lower than that of vacuum induction melt processes. When desired, the electron beam process can be operated at a pressure as high as 50μ (50×10^{-3} torr). The purification is achieved by a number of mechanisms, including density separation of heavy compounds (such as tungsten carbide), volatilization of trace/tramp elements, and gas evolution. These reactions are promoted by the shallow inert hearth where a large surface area of the metal is exposed to the high local superheat of the electron beam. When melting involves volatile alloying elements, compensating additions are made to the charge of raw material.

The purified and alloyed metal trickles from the hearth to the water-cooled ingot mold which is exposed to a vacuum and a hot-topping electron beam. The metal is continuously solidified to obtain a homogeneous, controlled grain ingot structure and high ingot yield. The process allows direct observation and independent control of the purification and alloying reactions and the solidification process. The process enables a high percentage of Ti scrap to be conveniently charged, melted, purified, alloyed and solidified in a single melt to produce an ingot with potentially superior structure and overall quality. Selected ingot sizes and shapes (rounds, tube hollows, rectangular) can be cast with potentially attractive economies and properties in finished mill products and forgings.

b. System Hardware

Figure 73 presents a schematic diagram of the electron beam hearth melting furnace. As indicated, the scrap mixture is metered out of the charge hopper, fed to the shallow hearth, melted and purified and then flowed into the ingot chamber. Figure 74 illustrates the incoming scrap mixture (top center), and the hearth pool of molten alloy overflowing into the ingot chamber (bottom center).

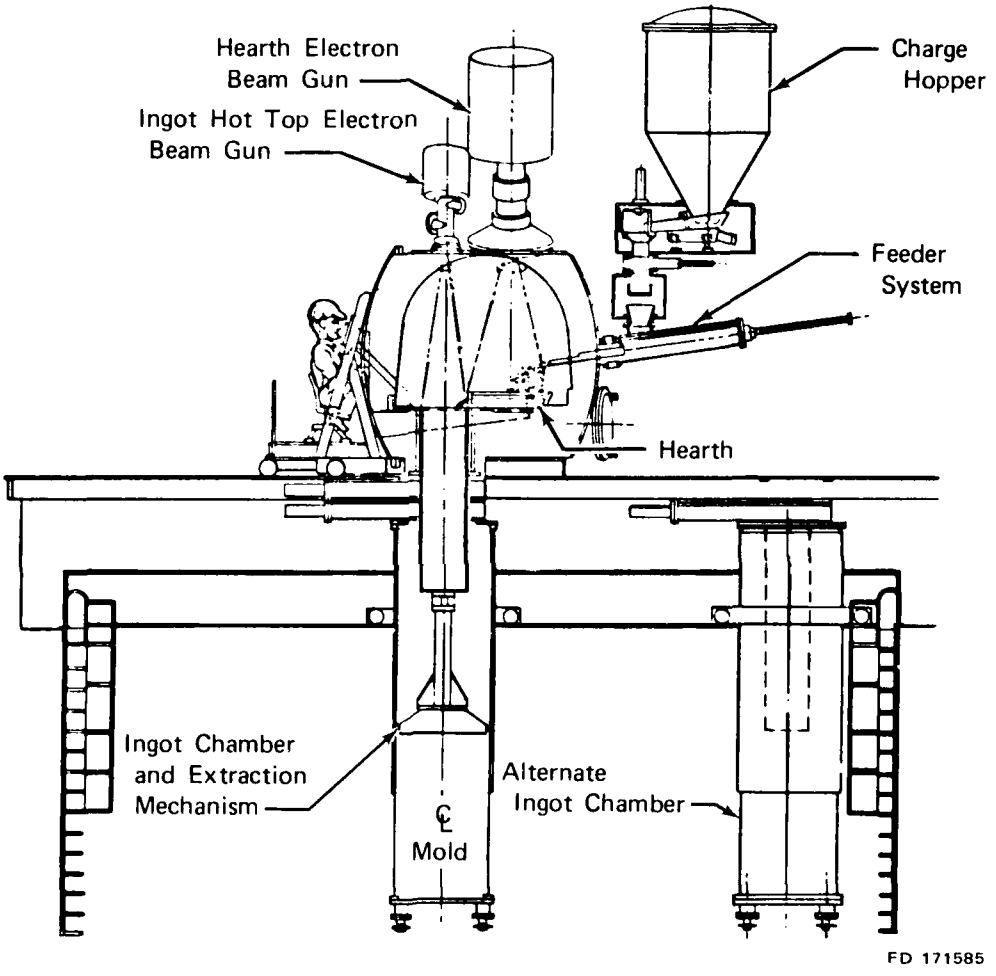

Figure 73. Diagram of AIRCO Electron Beam Cold Hearth Melting Furnace

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Figure 74. AIRCO Electron Beam Cold Hearth Furnace Showing Incoming Scrap Mixture (Upper Center), and Hearth Pool of Molten Alloy Overflowing Into Ingot Mold (Lower Center)

c. Program Plan Details (Phase I)

A mixture of scrap chips of Ti-6Al-4V were blended with virgin metal and master alloy additions and melted in an EB melting furnace. The melt was doped with tungsten carbide chips to ensure contamination. The objective of the process experiment was to demonstrate that EB melting of a Ti scrap charge will achieve removal of heavy metal particles, will meet chemistry specifications for the alloy and demonstrate that aerospace physical properties can be achieved. The ability to measure molten metal chemistry on a continuous basis by advanced techniques was evaluated analytically. Two experimental ingots, nominally 18 in. dia, were melted and cast. Melting of a number of small ingots was required to determine initial large ingot parameters. Conventional chemistry analyses on chill plug samples taken from the melt were used for control and documentation of the chemistry.

Ingot No. 1 was melted to calculated values for feed materials; ingot No. 2 was used for more precise control of composition. Both ingots were seeded with tungsten carbide particles. Ingot chemistry was determined. Ingots were reduced to billet and microstructure was determined on billet series. The billets were forged to nominal 1-in. dia bars. After cropping, chemical and physical tests were performed to certify that the alloy was within specification limits. A preliminary analysis was made to demonstrate that the doped tungsten carbide particles were removed in the melting process. Billet slices and approximately 4000 lb of bar from each ingot were then subjected to chemistry, structural and mechanical property evaluation by P&WA.

d. Test Program

(1) Chip Analysis

Four thousand pounds of Ti-6Al-4V machined chips were received from the Frankel Company. The analysis is shown in Table 47. The chip analysis indicates that the Fe, Cr, Ni and Mo total exceed Specification 4928-C. The chips also appeared to be contaminated with tin. The chemical analysis of the other raw materials used in the melting of Ti-6Al-4V ingots is also summarized in Table 47.

A water condensate was observed on the plastic liner of the barrels of chips. After drying, the chips indicated a weight change of 0.78%. To minimize the introduction of moisture and the attendant increase in oxygen content of the chips, the charge materials were preheated to 250°F before being added to the chip feeder. Frankel, in response to this problem, stated that the chips had been placed into plastic-lined barrels while still hot from the cleaning process and that humid July conditions resulted in condensate retention during cooling of the chips. Frankel stated that most customers do not have critical moisture requirements, and indicated that AIRCO's requirements could be met.

TABLE 47
RAW MATERIAL (CHIPS) ANALYSES — AIRCO

	Frankel	*AIRCO*	*Ti Sponge Lot 154*	*60Al-40V Master Lot 200*	*Al Shot Lot 220*
C(ppm)	159	342	317	890	8
	—	383**	283**	—	—
O(ppm)	2200	2872	446	830	160
	—	2388**	537**	—	—
	—	2766**	680**	—	—
N(ppm)	—	—	19	10	120
	—	—	23**	—	—
	—	—	56**	—	—
Al(%)	6.55	6.10	< 0.02	55.5	99.75
		6.05**	—	—	—
V(%)	3.70	3.82	0.02	43.5	—
Sn(%)	0.09	0.66	0.04	—	< 0.005
		0.65**	—	—	—
Fe(%)	0.38	0.179	0.02	0.33	0.14
	0.11*	—	—	—	—
Cr(%)	0.09*	0.024	0.024	—	< 0.005
Ni(%)	0.30*	0.47	—	—	—
Mg(%)	—	0.05	0.23	—	—
Mo(%)	0.15	0.065	—	—	—
Cb(%)	< 0.05	0.05	—	—	—
Pb(%)	< 0.05	—	—	—	—
Zr(%)	0.07	—	—	—	—
Mn(%)	< 0.05	—	—	—	—

*Aqua regia soluble
**Retest

In order to calibrate the oxygen content of a mixture of Ti scrap and sponge and to evaluate the dilution offset of sponge on aluminum level, two heats of Ti-5Al-2.5Sn scrap with sponge additions of 15% and 60% were made. The feedstock data and chemical analyses of ingots No. 4089 (15% sponge) and No. 4101 (60% sponge) are shown in Table 48.

TABLE 48
FEEDSTOCK DATA AND CHEMICAL ANALYSES OF INGOTS — AIRCO

INGOT NO. 4089

Feedstock

Ti-2.5 Chips	85%
Ti Sponge	15%
Finished Ingot Weight	803 lb

Chemical Analysis

Sample Location (in.)	*Al*	*Sn*	*V*	*Fe*	*Cr*	*C*	*O*	*N*
10.9	2.62	1.74	0.176	0.79	0.056	0.0411	0.3002	0.0120
20.2	2.56	1.72	0.188	0.76	0.055	0.0400	0.2853	0.0130

Typical Scrap Remelt Analysis 3400
O_2 Change by Dilution ~ 400 ppm

INGOT NO. 4101

Feedstock

Ti 5-2.5 Chips	1200 lb	60%
Ti Sponge	800 lb	40%
	2000 lb	100%
Finished Ingot Weight		1676 lb

Chemical Analysis

Sample Location (in.)	*Al*	*Sn*	*V*	*Fe*	*Cr*	*C*	*O*	*N*
11.9	1.21	0.91					0.2216	
							0.2145	
20.6	1.29	0.91					0.2001	
							0.2009	
30.5	1.07	0.84					0.2221	
							0.2200	
40.9	1.15	0.88					0.2126	
							0.2131	
42.0	0.73	0.70					0.1855	
							0.1872	

The results on ingot 4089 showed a nominal 400 ppm drop in oxygen from a nominal 3400 ppm analysis of 100% scrap charge prepared from this lot of scrap. This drop is proportional to the 15% sponge addition. The 60% sponge charge from ingot No. 4101 showed an oxygen analysis somewhat higher than the calculated 180 ppm anticipated from the heat. Only one analysis was within the expected oxygen range.

(2) Ingot Preparation and Melting

Two ingots of composition Ti-5Al-2.5Sn were melted using the CHF 1200 vacuum furnace at AIRCO. The furnace was equipped with vacuum pumps of higher pumping capacity and modified electron beam guns than conventional vacuum furnaces to ensure constant power input atmosphere for control of composition during melting. In addition, feedstock was prepared for Ti-6Al-4V ingots. These test results permitted calculation of the sponge dilution and aluminum addition requirements applicable to Ti-6Al-4V ingot melts.

(3) Test Results

(a) Ingot Manufacture

Mixing trials were conducted with small chip lots to demonstrate the feasibility of feeding a homogeneous four-element mixture, i.e., Ti-6Al-4V chips, Ti sponge, 60Al-40V master alloy and Al shot to the melting hearth. Subsequently, two 1000-lb, 18-in. dia ingots (subscale length) were produced — Nos. 4102 and 4101 with lengths of 18.6 and 26.2 in. respectively. Results concerning ingot No. 4102 are presented in Tables 49 and 50, and in Figures 75 and 76. Ingot No. 4104 results are given in Tables 51 and 52, and in Figures 77 and 78.

Ingot pouring rates (160 to 190 lb/hr) were slower than those established at AIRCO with 100% Ti-5Al-2.5Sn chip feedstock. The chip feed system also required frequent adjustments to maintain a constant pouring rate.

Oxygen levels of 1200 to 5000 ppm were consistent with values calculated prior to the run. The aluminum and vanadium contents were within specification at the mid-length of the ingot but deviated from specification at each end of the billet. The time required to establish steady-state feeding conditions at the beginning of the ingot, and exposure time of the melt to the hot topping gun at the end of the ingot, are likely contributing factors to the observed chemistry variations. An analysis of the melt condensate (volatilized elements), captured by a screen located in proximity to the melt, indicated a chemistry of Al-44.29% Ti-0.71% V.

TABLE 49
AIRCO INGOT NO. 4102 FEED STOCK

Content	*Weight (lb)*	*Weight (%)*
Ti-6Al-4V Frankel Chips	260	26.8
Ti Sponge	578	59.7
60Al-40V Alloy	60	6.2
¼-in. Al Shot	70	7.2
Total	968	99.9
Finished Ingot Weight	680	
Material Difference	288	
Hearth Skull	260	
Hearth Starting Load	52	
Net Hearth Material Inventory	208	
Melt Loss	80	
Melt Efficiency 91.7%		

TABLE 50
AIRCO INGOT NO. 4102 CHEMISTRY

MURRAY HILL LABORATORY

Dist. From Ingot Base (in.)	C (ppm)	O (ppm)	N (ppm)	Al (%)	V (%)	Sn (%)	Fe (%)	Cr (%)	Ni (%)	Mo (%)
Drilled Samples										
5	563	—	—	7.02	4.28	0.067	0.11	0.009	0.091	0.043
	613	—	—							
10	675	—	—							
	724	—	—	5.86	3.68	0.099	0.17	0.008	0.086	0.04
15	760	—	—							
	804	—	—	3.48	2.89	0.097	0.15	0.008	0.078	0.03
Dip Samples										
10	557	1184	45							
	552	1284	49	6.73	4.29	0.07	0.14	0.044	0.11	0.036
								< 0.005	< 0.005	
18.6	508	1511	67							
	515	1474	83	3.2	2.92	0.10	0.12	0.028	0.11	0.034
								< 0.005	< 0.005	

ANAMET LABORATORIES

Dip Samples										
10	—	—	—	6.79	4.31	—	—	—	—	—
18.6	—	—	—	3.77	2.82	—	—	—	—	—
Condens.	—	—	—	55.1	0.71	—	—	—	—	—
Specification (Ti-6Al-4V)										
	1000	2000	500	5.5-6.75	3.5-4.5	—	0.30 max	—	—	—

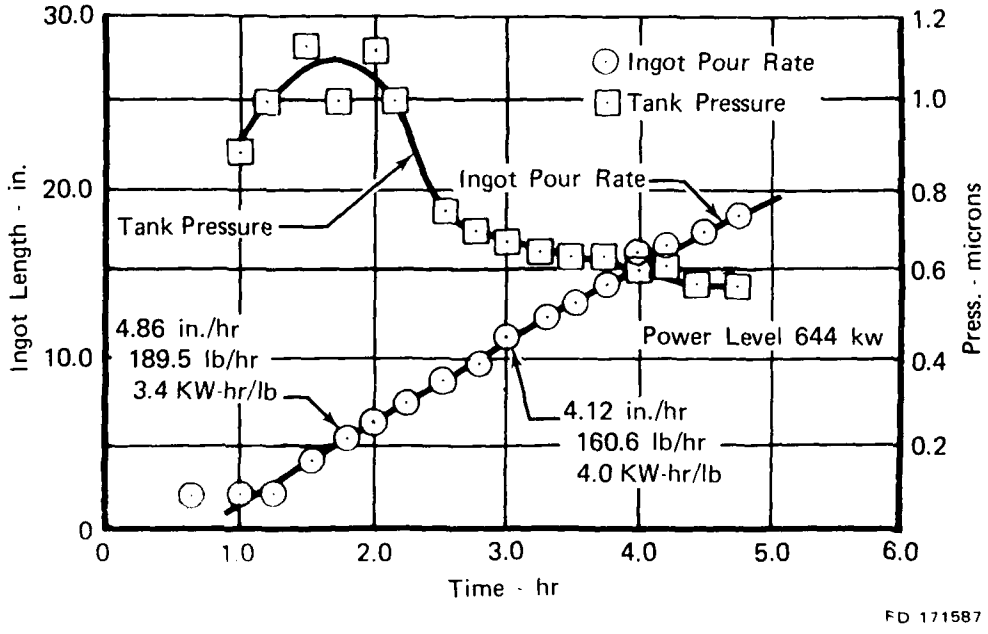

Figure 53. AIRCO Ingot No. 4102 (Ti-6Al-4V) Melting Record

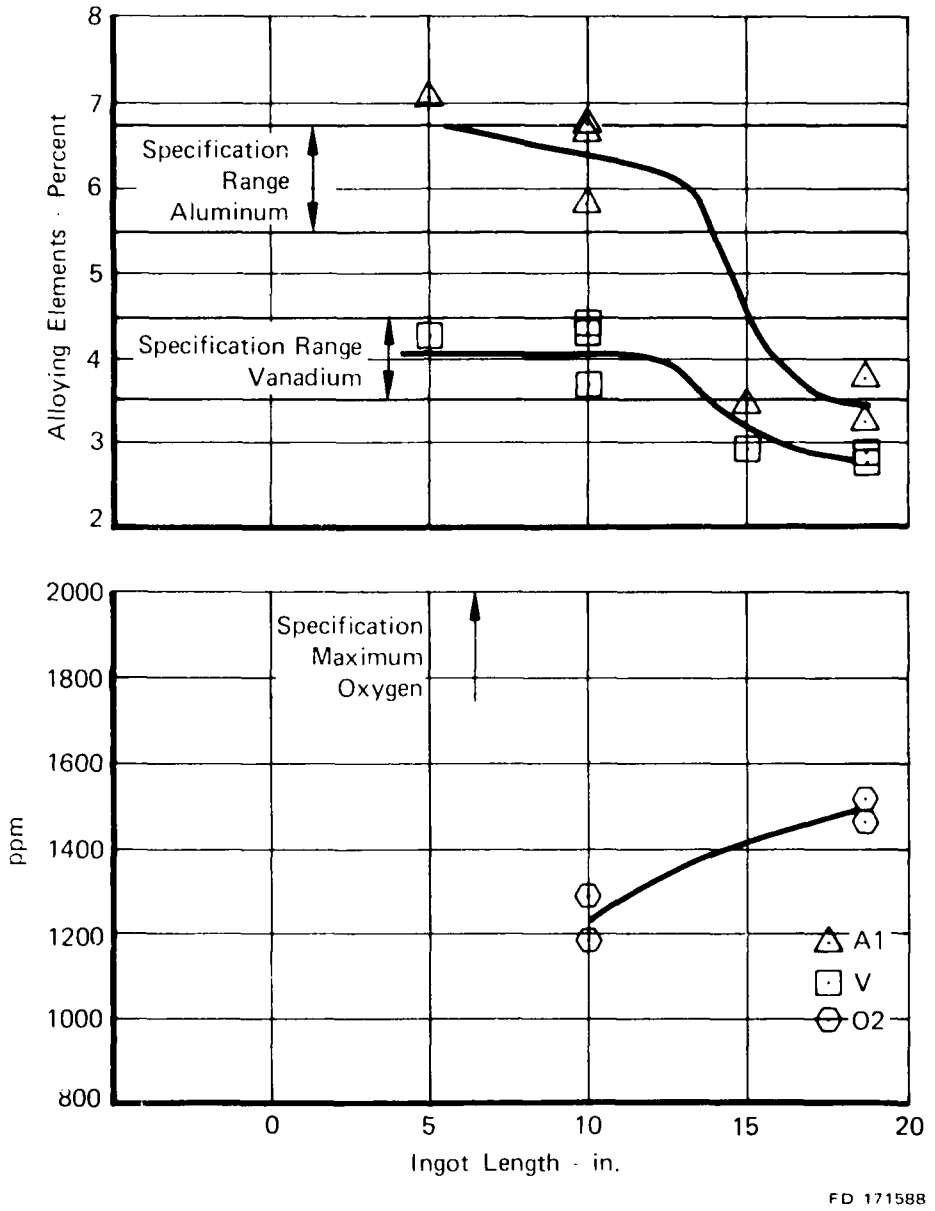

Figure 76. AIRCO Ingot No. 4102 (Ti-6Al-4V) Chemistry

TABLE 51
AIRCO INGOT NO. 4104 FEED STOCK

Content	*Weight (lb)*	*Weight (%)*
Ti-6Al-4 Frankel Chips	432.90	39.3
Ti Sponge	550.02	50.0
60Al-40V Alloy	55.02	5.0
⅛ in. Al Shot	62.10	5.6
Total	1100.04	99.9
Finished Ingot Weight	965.00	
Material Difference	135	
Hearth Skull	256	
Hearth Starting Skull	266	
Loss	(−) 10	
Residue in Feeder	(−) 75	
Melt Loss	70	
Melt Efficiency	93.2%	

TABLE 52
AIRCO INGOT NO. 4104 CHEMISTRY

MURRAY HILL LABORATORY

Dist From Ingot Base (in.)	*C (ppm)*	*O (ppm)*	*N (ppm)*	*Al (%)*	*V (%)*	*Sn (%)*	*Fe (%)*	*Cr (%)*	*Ni (%)*
Dip Samples									
7.6	645	2156	-	3.87	3.94	0.1	0.19	0.019	0.01
	590	2095							
10.6	523	2126	-	5.58	3.85	0.11	0.17	0.014	0.08
	533	2075							
15.6	494	1838	-	6.11	4.12	0.1	0.15	0.015	0.19
	495	1910							
20.2	514	1797		5.5	4.06	0.11	0.16	0.018	0.08
	503	1573							
		1811							
26.2	553	2370	-	3.86	3.59	0.12	0.17	0.019	0.16
	569	2352							

ANAMET LABORATORIES

Drill Samples									
9				3.14	3.76				
14				5.54	3.75				
16				6.17	4.16				
18				5.29	3.93				
20				4.44	3.51				
Dip Samples									
7.6				3.81	3.98	0.05			
10.6				5.18	3.83	0.06			
15				6.07	4.37	0.07			
20.2				5.21	4.13	0.07			
26.2				3.79	3.55	0.09			
Specification (Ti-6Al-4V)									
	1000	2000	500	5.5-6.75	3.5-4.5		0.30 max		

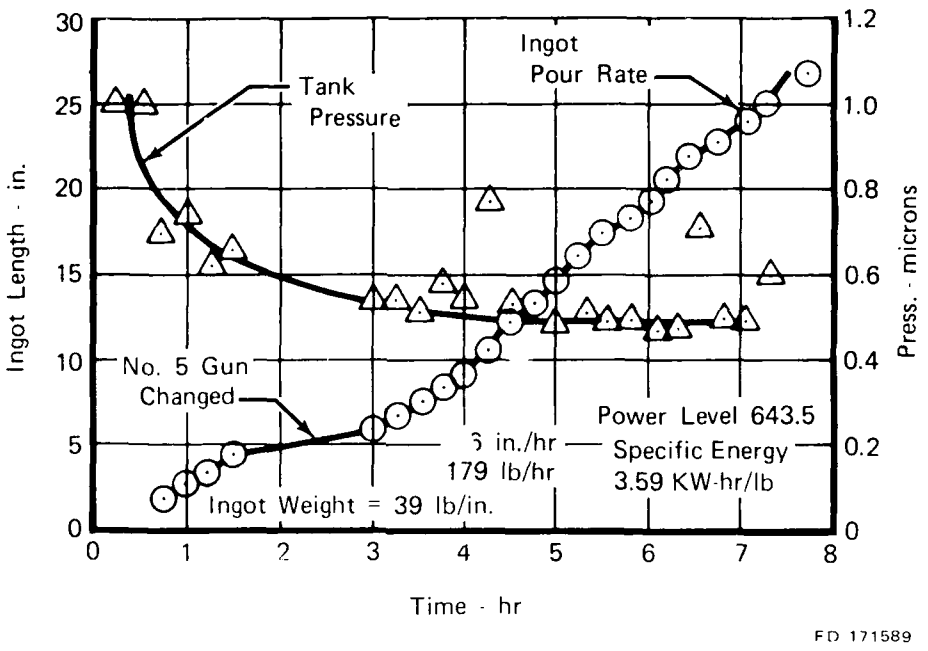

Figure 77. AIRCO Ingot No. 4104 (Ti-6Al-4V) Melting Record

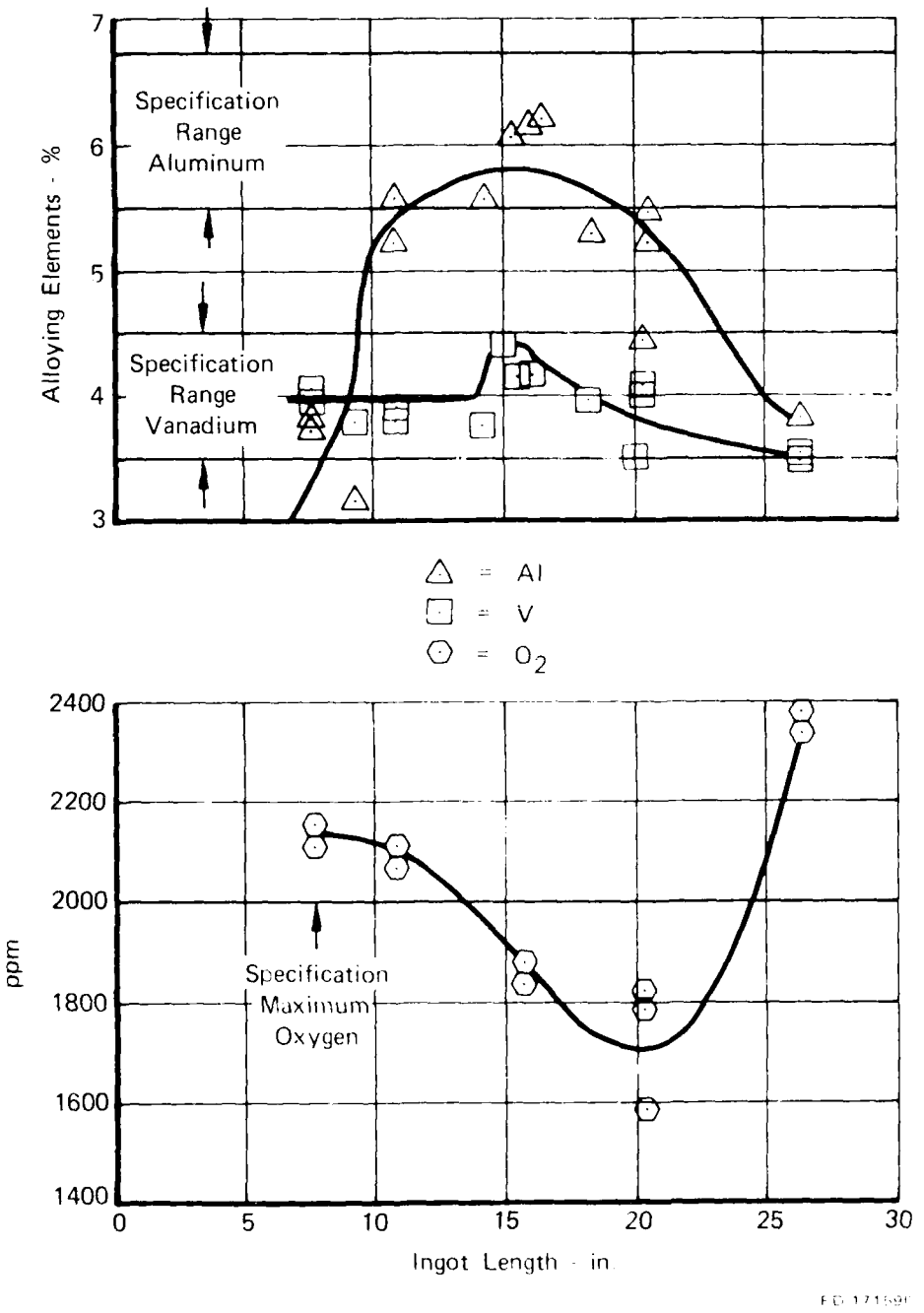

Figure 18. AIRCO Ingot No. 4101 (Ti-6Al-4V) Chemistry

AD-A083 089 PRATT AND WHITNEY AIRCRAFT GROUP WEST PALM BEACH FL G--ETC F/G 13/8
MANUFACTURING METHODS FOR STRATEGIC MATERIALS RECLAMATION.(U)
DEC 79 H K GREGORY, T D TILLMAN F33615-74-C-5019
UNCLASSIFIED PWA-FR-11871 AFML-TR-79-4161 NL

3 OF 4
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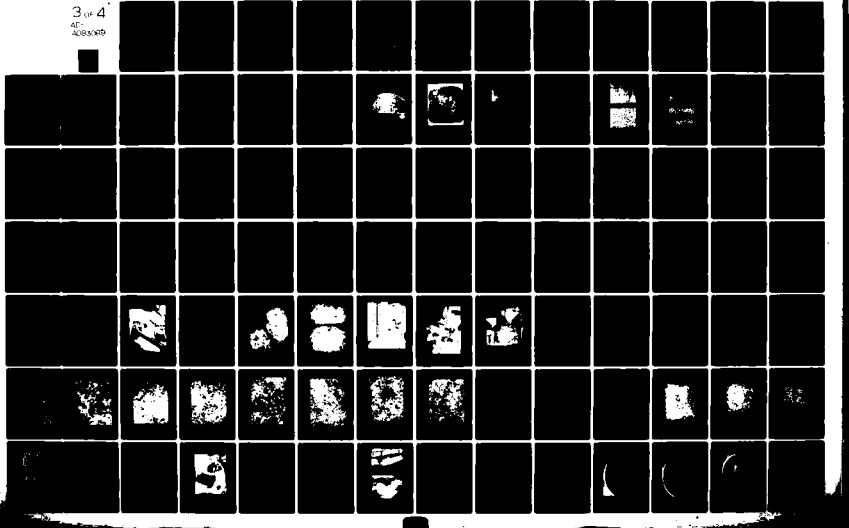


Slow feed characteristics, similar to those experienced with ingot No. 4102, were again noted. Aluminum evaporation rate was high both at the beginning and the end of the run. Delays in the melting rate, incurred during correction of a No. 5 EB gun malfunction contributed to the high evaporation rate at the beginning of the run. Oxygen levels, higher than expected, may also be related to the No. 5 gun problem since the oxygen pickup from the furnace atmosphere increased in proportion to the melt delay. The perturbation of vanadium content was believed related to an inhomogeneity in the feeding of the 60Al-40V master alloy.

In view of the problems encountered with feed rate and compositional homogeneities in these runs, a hopper of chips, sponge and master alloy was fed into boxes, and samples taken every 5 min. Results of this test are presented in Table 53, Figure 79 and Figure 80. It was observed that the feed rate varied from 2.4 to 10 lb/min. The sponge, until depleted, fed at a faster rate than the chips and then at a gradually decreasing rate. The 60 Al-40V master alloy fed at a constant rate and the aluminum shot at an increasing rate.

As a result of the feeding tests and the resultant findings, changes were made to the feeding system of the CHF 1200 furnace to establish the necessary mixing and feeding requirements. The changes feature a vibratory feed system with a capability for constant feed over a range of input rates from 200 to 600 lb/hr. A 500-lb lot was used to test the modified unit: the feedstock mixture (Table 54), was premixed in a rotating drum. Test results are in Figures 81 and 82. The initial five data points are significant; the last two points represent residue. It was concluded that the modifications to the feed system, along with the premix operation effectively reduced the previously experienced variations (Figure 80), however, some separation of components, i.e., sponge feed faster than chips, was indicated by the high concentration of chips in the residue (Figure 82).

AIRCO-Temescal melted two ingots (Nos. 4115 and 4116) required for evaluation under Phase I of the program plan. Ingot No. 4115 was made from the material noted in Table 53, with 30 WC tool bit particles ranging in size from 1/16 to 1/4 in. P&WA received a cross section of the No. 4115 forging billet, and 400 lb of round-corner-square (RCS) barstock forged from the No. 4115 billet, for NDI, chemistry, metallographic and mechanical properties evaluation. Analyses completed by AIRCO-Temsecal indicate that the hearth feed system is a major factor in controlling ingot chemistry.

(b) Feed and Forging Procedures

The feed procedures used for ingot Nos. 4115 and 4116 were essentially identical. Feedstock were premixed into six rotating 55-gallon drums, and each drum load was poured in sequence into the six compartments of the feed hopper. The hopper loading sequence (Table 55), was devised to ensure an even distribution of feedstock. The loading sequence, and a vibratory feed system were adopted as the result of the trial testing noted above.

TABLE 53
MATERIAL USED FOR AIRCO FEED STOCK SYSTEM TEST

Content	*Weight (lb)*	*Weight (%)*
Ti-6-4 Frankel Chips	582.5	38.8
Ti Sponge	750.0	50.0
60Al-40V Alloy	73.0	4.8
1/8 in. Al Shot	94.5	6.3
Total	1500.0	99.9
Minimum Feed	11 lb/5 min	132 lb/hr
Maximum Feed	50 lb/5 min	600 lb/hr

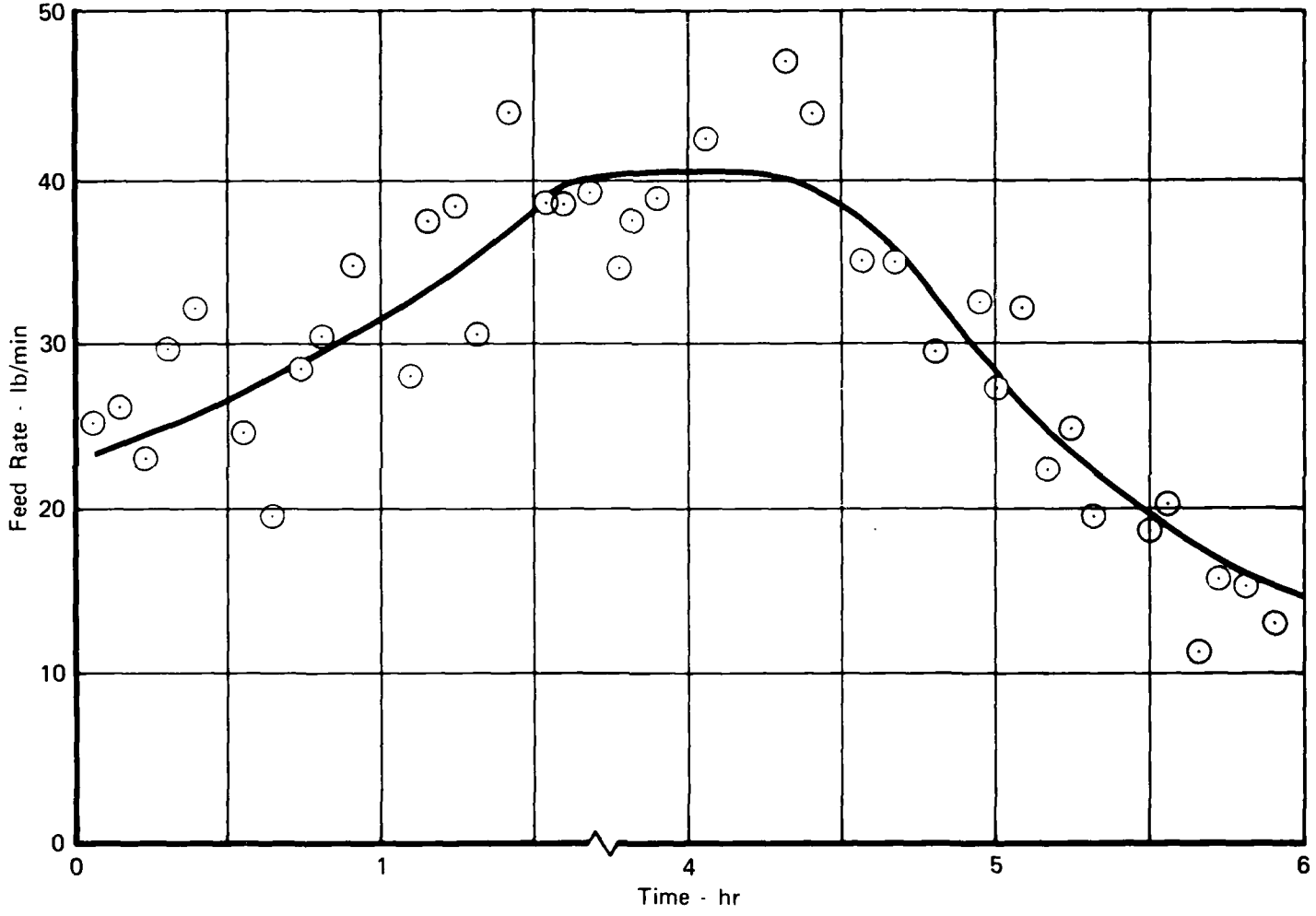


Figure 79. AIRCO Feed System Test Results

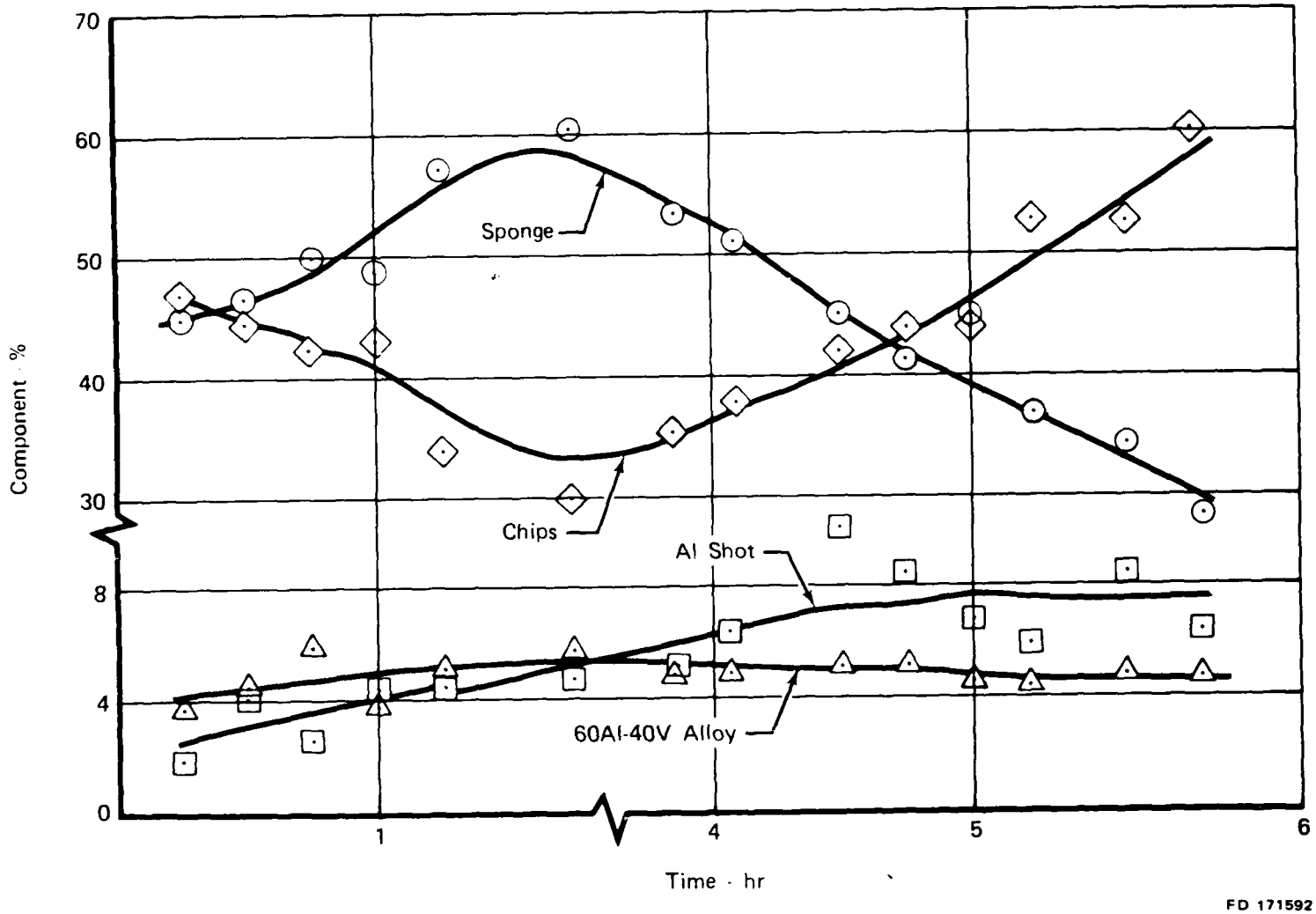


Figure 80. AIRCO Feed System Test — % of Components

TABLE 54
MATERIAL USED FOR AIRCO VIBRATORY FEED SYSTEM TEST

Content	*Weight (lb)*	*Weight (%)*
Ti-6-4 Frankel Chips	250.0	50
Ti Sponge	193.5	38.7
60Al-40V Master Alloy	23.2	4.64
1/8 in. Al Shot	33.1	6.62
Total	499.8	99.98

Minimum Feed	18.5 lb/5 min	222 lb/hr
Maximum Feed	34.0 lb/5 min	408 lb/hr
Typical Feed	33.0 lb/5 min	264 lb/hr

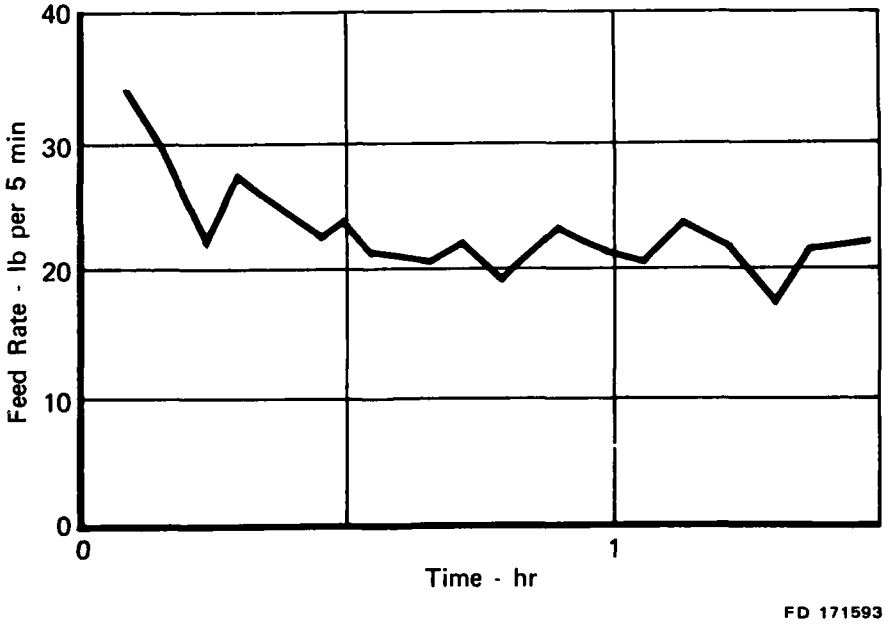


Figure 81. AIRCO Vibratory Feed System Test — Feed Rate Uniformity

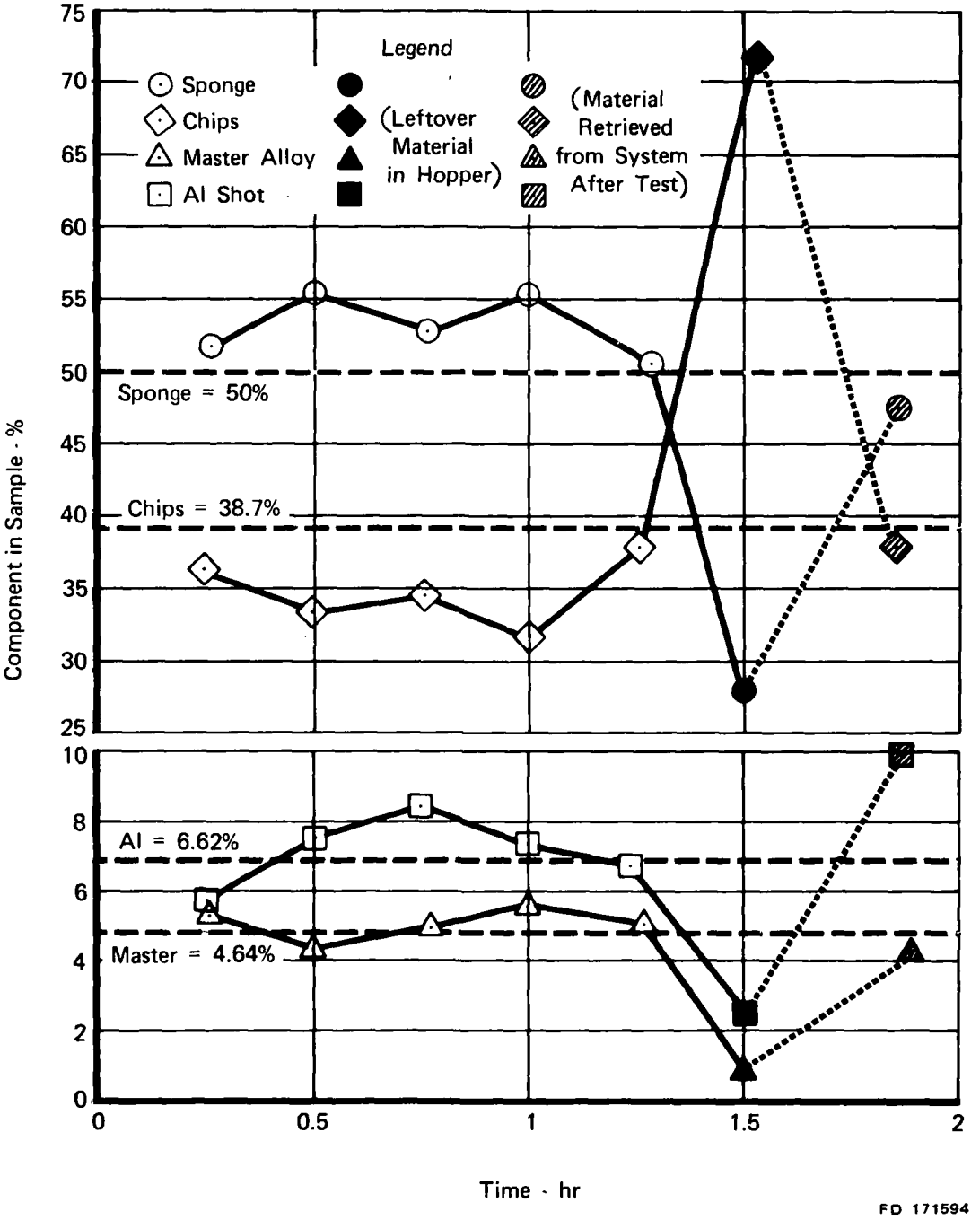


Figure 82. AIRCO Vibratory Feed System Test — Feed Composition Chemistry

TABLE 55
HOPPER LOADING PROCEDURE FOR SIX BARRELS OF PREMIXED Ti-6Al-4V INPUT MATERIAL — AIRCO

	HOPPER SECTION					
	A	*B*	*C*	*D*	*E*	*F*
Loading Sequence	6*	5	6	5	6	5
	4	3	4	3	4	3
	2	1	2	1	2	1
	5	6	5	6	5	6
	3	4	3	4	3	4
	1	2	1	2	1	2

*Each number represents a 1/6 portion of the barrel bearing that number

The EB cold hearth melt parameters, i.e., feed rate and EB power input, were essentially the same for both ingots. A casting profile was determined by measuring the length of the cast ingot every 15 min. Chemical analyses were accomplished by both dip samples removed from the melt at 5 in. increments and drillings at 5 in. increments from the surface of ingot No. 4116 following its complete solidification.

(c) Chemistry and Profile Analysis

Chemical analyses relative to ingot No. 4115 are presented in Table 56, and shown in Figure 83 (chemistry) and Figure 84 (casting profile). The corresponding data for ingot No. 4116 is presented in Table 57 (chemistry) and shown in Figure 85 (chemistry) and Figure 86 (casting profile). Ingot No. 4115 is within chemical specification except for aluminum content within the initial five inches of melting. The initial low aluminum content is believed to be related to dilution of the input feed material by the initial hearth inventory. Further empirical data would provide a basis for compensating for this low aluminum startup level. Ingot No. 4116 shows good control of vanadium along its length, and oxygen is within specification; however, a significant variation in aluminum content is evident in both dip samples and drillings from the ingot surface, with the dip samples showing the more pronounced effect.

The origin of this variation in aluminum content is potentially attributable to the following factors: variable vaporization of aluminum, variable homogenization on the hearth and/or variations in feeding of raw materials. This variation does not appear to be related to the vaporization of aluminum since the casting profile is satisfactory. Homogenization of the charge on the hearth also appears to be satisfactory. The variation appears to be caused by segregation or classification of raw materials during the mixing and the feeding operations.

The results of three "dry run" feeding experiments showed the tendency for sponge to feed faster than chips and for aluminum shot to increase its feed rate during the run. The 60Al-40V master alloy was fed uniformly in all experiments.

TABLE 56
AIRCO INGOT NO. 4115 CHEMISTRY

Dist. From Ingot Base (in.)	*C (ppm)*	*O (ppm)*	*N (ppm)*	*Al (%)*	*V (%)*	*Sn (%)*	*Fe (%)*	*Cr (%)*	*Ni (%)*
Dip Samples									
4.8	—	2103	—	5.16	3.55	0.053	0.17	0.018	0.17
	—	2043							
10.1	673	1839	76	6.11	3.58	0.054	0.17	0.018	0.17
	605	1889	87						
15.0	—	1929	—	6.28	3.59	0.055	0.16	0.016	0.17
	—	1839							
20.6	655	1907	55	5.87	3.63	0.053	0.17	0.018	0.17
	614	1975							
24.5	—	1890	—	6.25	3.66	0.055	0.17	0.016	0.17
	—	1892							
29.7	—	1988	—	5.81	3.6	0.055	0.17	0.016	0.17
	—	2008							
32.6	—	2005	—	5.53	3.57	0.055	0.18	0.017	0.17
	—	2039							
Specification (Ti-6Al-4V)									
	1000	2000	500	5.5/6.75	3.5/4.5		0.30 max		

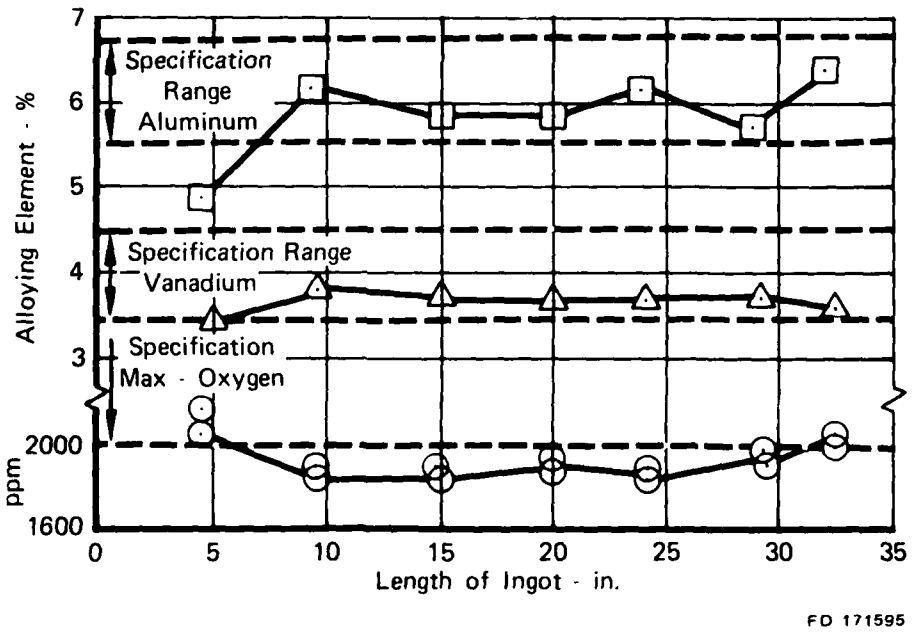


Figure 83. AIRCO Ingot No. 4115 (Ti-6Al-4V) Chemistry

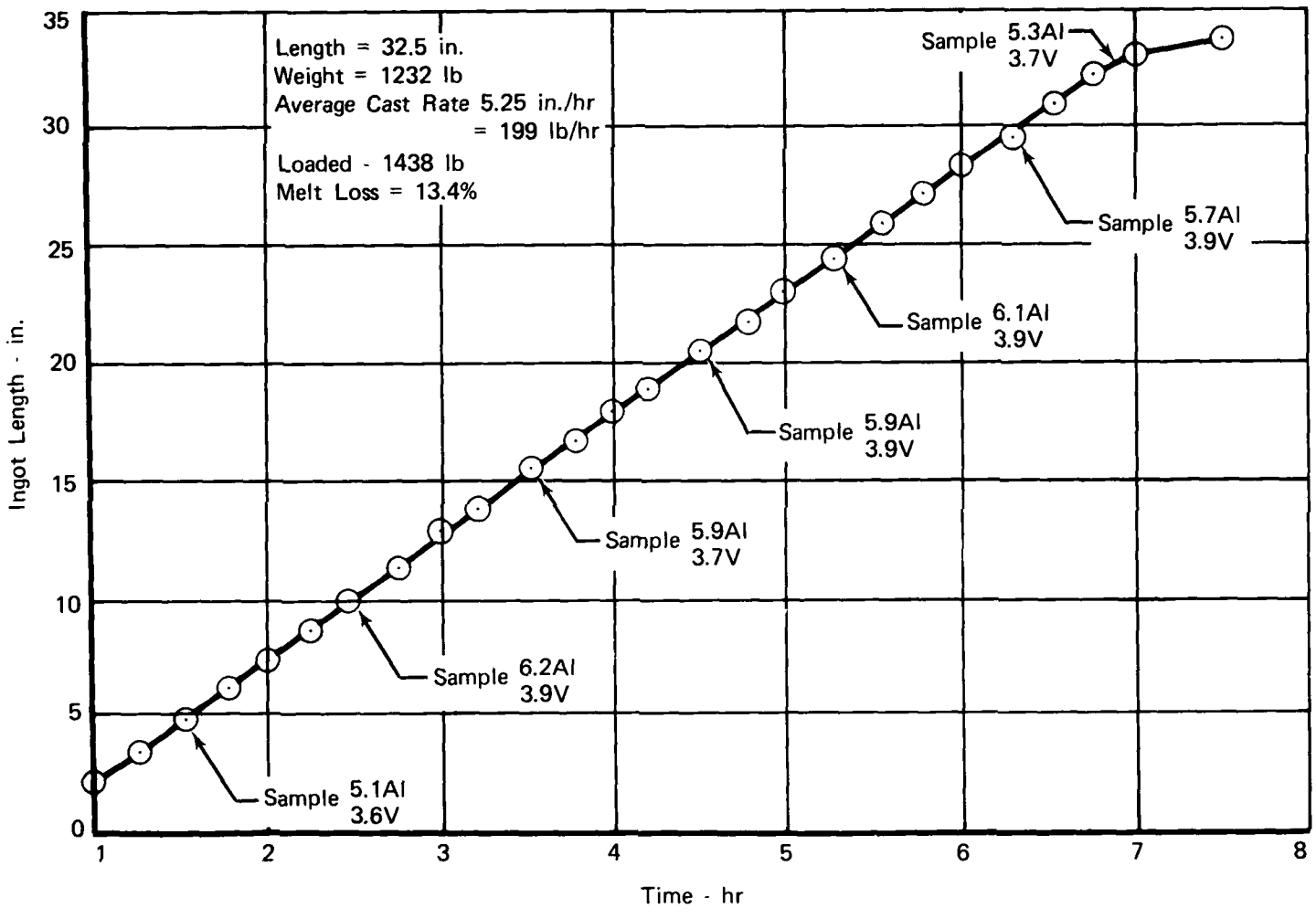


Figure 84. AIRCO Ingot No. 4115 Casting Profile

TABLE 57
AIRCO INGOT NO. 4116 CHEMISTRY

Dist From Ingot Base (in.)	C (ppm)	O (ppm)	N (ppm)	Al (%)	V (%)	Sn (%)	Fe (%)	Cr (%)	Ni (%)
Dip Samples									
6.8				4.04	3.98	0.05	0.16	0.016	0.18
10.2	Chill lost/no sample								
12.9	444	1955	43	5.51	3.80	0.05	0.16	0.021	0.16
	452	1845	24						
		1875	77						
15.7				4.85	3.81	0.05	0.16	0.024	0.24
				5.20					
				5.32					
19.6	411	1840	95	5.95	3.87	0.05	0.16	0.023	0.16
	480	1850	106						
		1837	89						
25.6				7.07	3.38	0.05	0.16	0.024	0.17
				7.26					
				7.35					
30.8				5.60	3.78	0.05	0.16	0.025	0.17
33.3				5.68	3.74	0.05	0.16	0.020	0.16
Drilled samples									
5				4.04	3.72				
10				5.97	3.81				
15				5.25	3.83				
20				6.75	3.70				
25				6.32	3.68				
30				5.89	3.68				
Specification (Ti-6Al-4V)									
	1000	2000	500	5.50 to 6.75	3.50 to 4.50		0.30	Max	

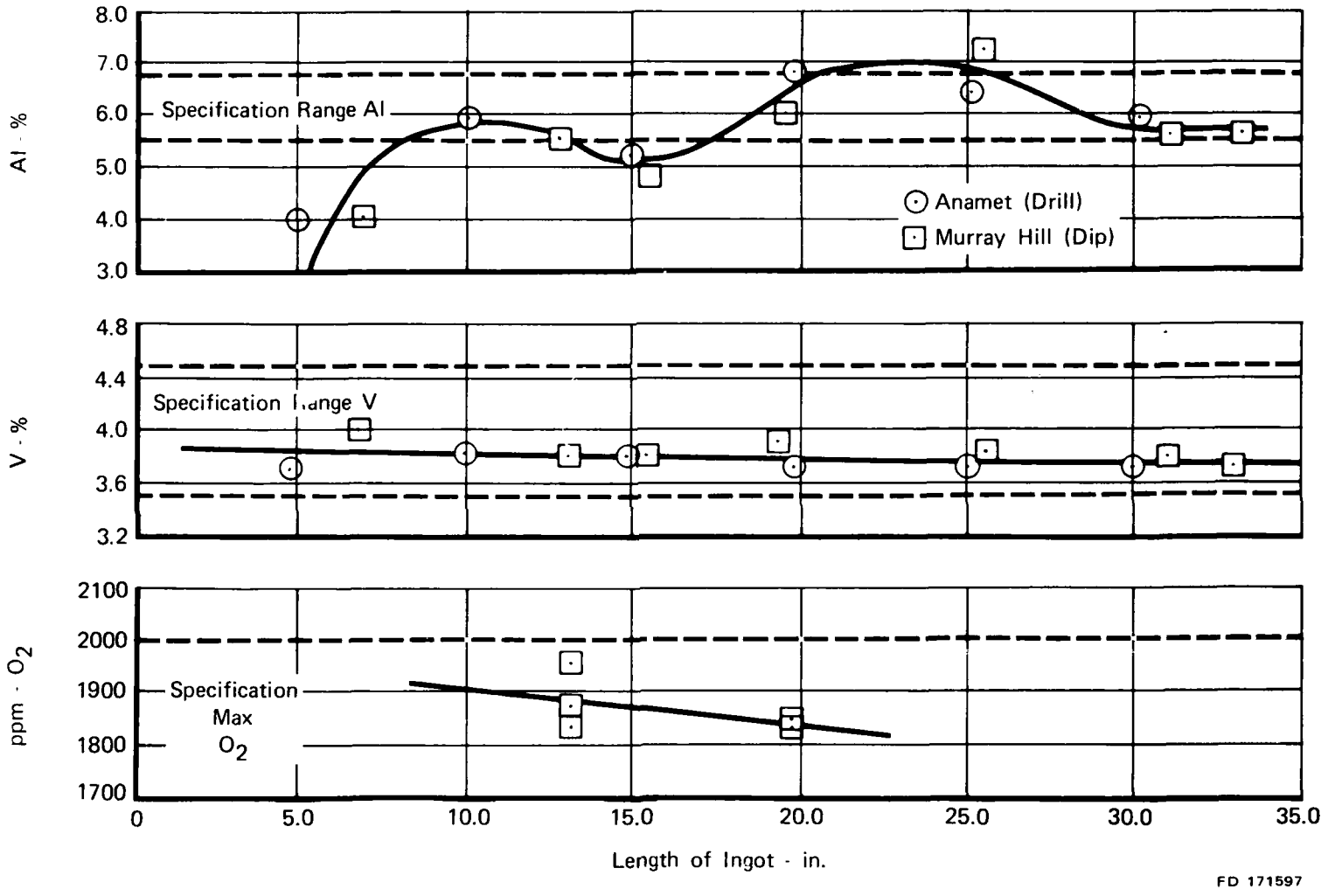


Figure 85. AIRCO Ingot No. 4116 Chemistry

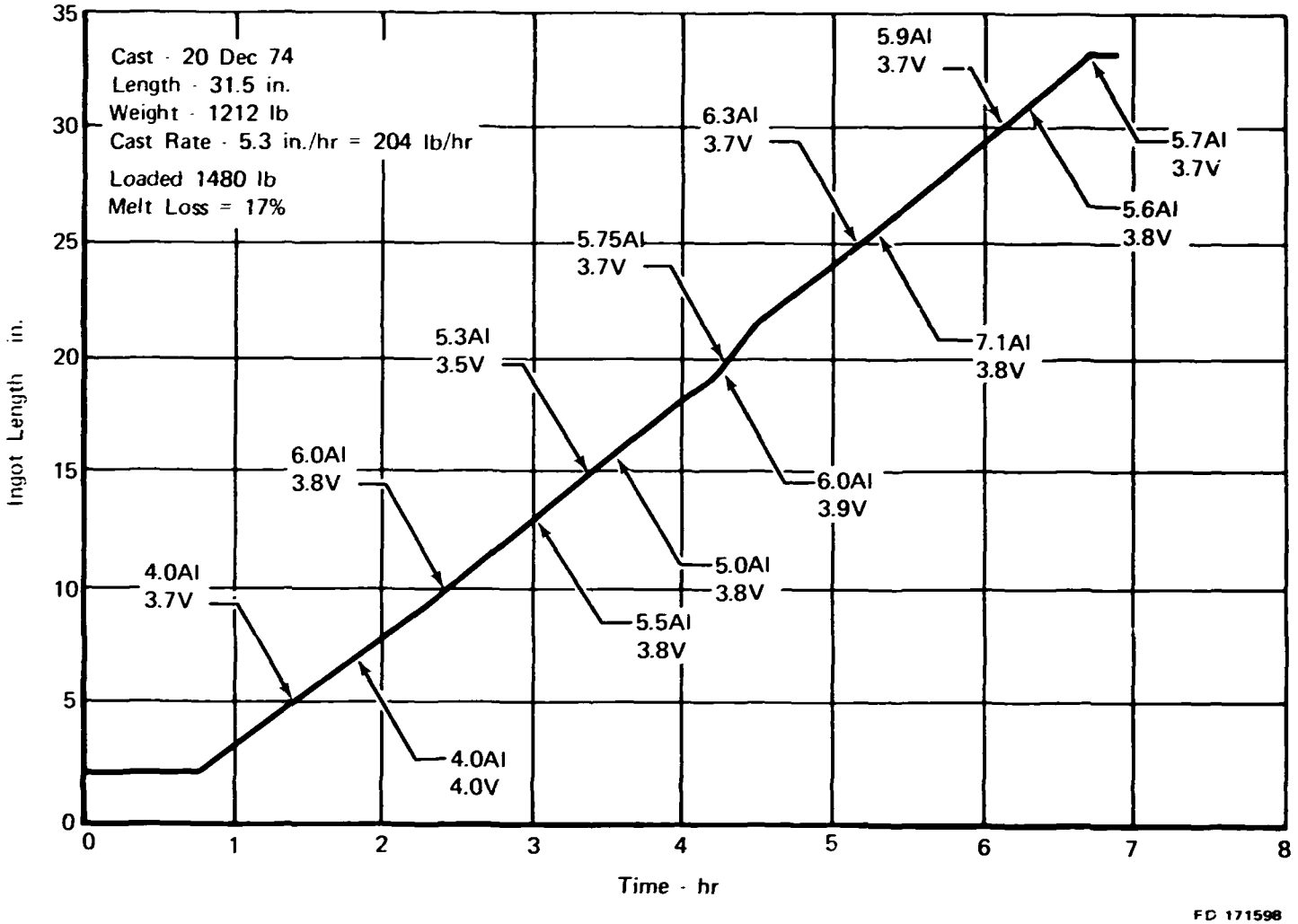


Figure 86. AIRCO Ingot No. 4116 Casting Profile

These results suggest an explanation for the variations* observed in ingot No. 4116. It is reasonable to assume that for vanadium:

$$\frac{V_{chip} + V_{master\ alloy}}{Ti_{chip} + Ti_{sponge}} = \text{constant}$$

and for aluminum,

$$\frac{[Al_{chip} + Al_{vmaster\ alloy}] + Al_{shot}}{Ti_{chip} + Ti_{sponge}} \neq \text{constant}$$

if,

$$[V_{chip} + V_{master\ alloy}] \sim [Al_{chip} + Al_{master\ alloy}]$$

It is therefore reasonable to assume that aluminum shot is the variable component in ingot No. 4116. It is also possible, that as the charge in the feeder is increased from a nominal 1500 lb used for subscale length ingots to the hopper capacity of 3500 lb, the classification of the feed stock into the four components of chips, sponge, master alloy and shot may be expected to increase.

Two options exist for improved feeding of raw materials: (1) selection of feedstock components having more compatible characteristics, and (2) individual feeding of each component in place of the presently used premix technique. Raw material changes to attain better component compatibility might include the substitution of scrap aluminum machined chips for aluminum shot, or the substitution of low oxygen, titanium/aluminum master alloy for aluminum shot. On the basis of the measured constant feed characteristics of the aluminum/vanadium master alloy in both dry run and melting experiments, an improvement may be expected.

Preliminary studies of the potential gains to be obtained by the feeding of individual components of the raw material charge have been made. Several systems appear to be able to contribute a substantial improvement in controlled continuous feed of melt stock to the hearth; however, the development, construction and evaluation of this concept was beyond the scope of Phase I of this contract.

The oxygen levels in both ingot No. 4115 and No. 4116 also appear higher than expected. Prior experience indicates a 250-ppm oxygen pickup in casting a titanium alloy at the same throughput levels with the CHF-1200 furnace, as opposed to the 400-ppm oxygen pickup experienced with ingot No. 4115 and No. 4116. The furnace vacuum system and the leak-up rate were both considered satisfactory; however, it appears possible that the Ti-6Al-4V scrap chips contained more oxygen than AIRCO's in-house cleaned Ti-5Al-2.5Sn alloy.

(d) Conversion Technique

AIRCO converted ingot No. 4115 to billet and finally to 1-in. round-corner square (RCS) barstock. The forging was accomplished in the sequence shown in Figure 87 with the furnace soak/reheat temperature at 2050°F. Billet section samples were removed at the 6-in. RCS size, and selected cross sections (Figure 88) were forwarded to P&WA for evaluation. The remaining billet was then converted to 1-in. RCS barstock, using a hot roll rather than a forging operation. Figure 89 illustrates the hot roll sequence and sample identification, and Table 58 indicates material accountability record for ingot No. 4115.

*Variables refer to the corresponding elemental weight fractions in either the chips, master alloy or sponge.

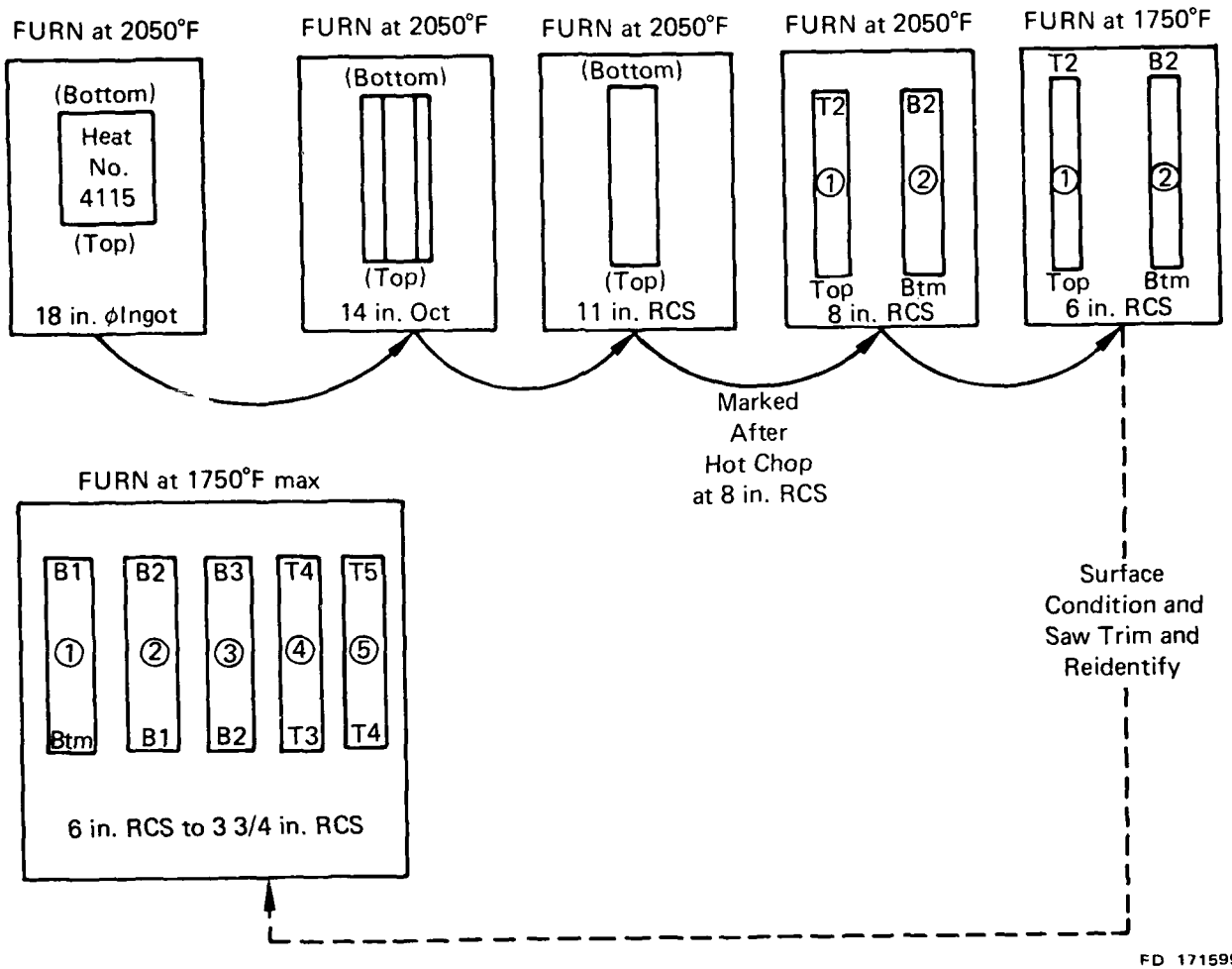


Figure 87. Identification of Conversion of AIRCO Ingot No. 4115 to Bar Stock

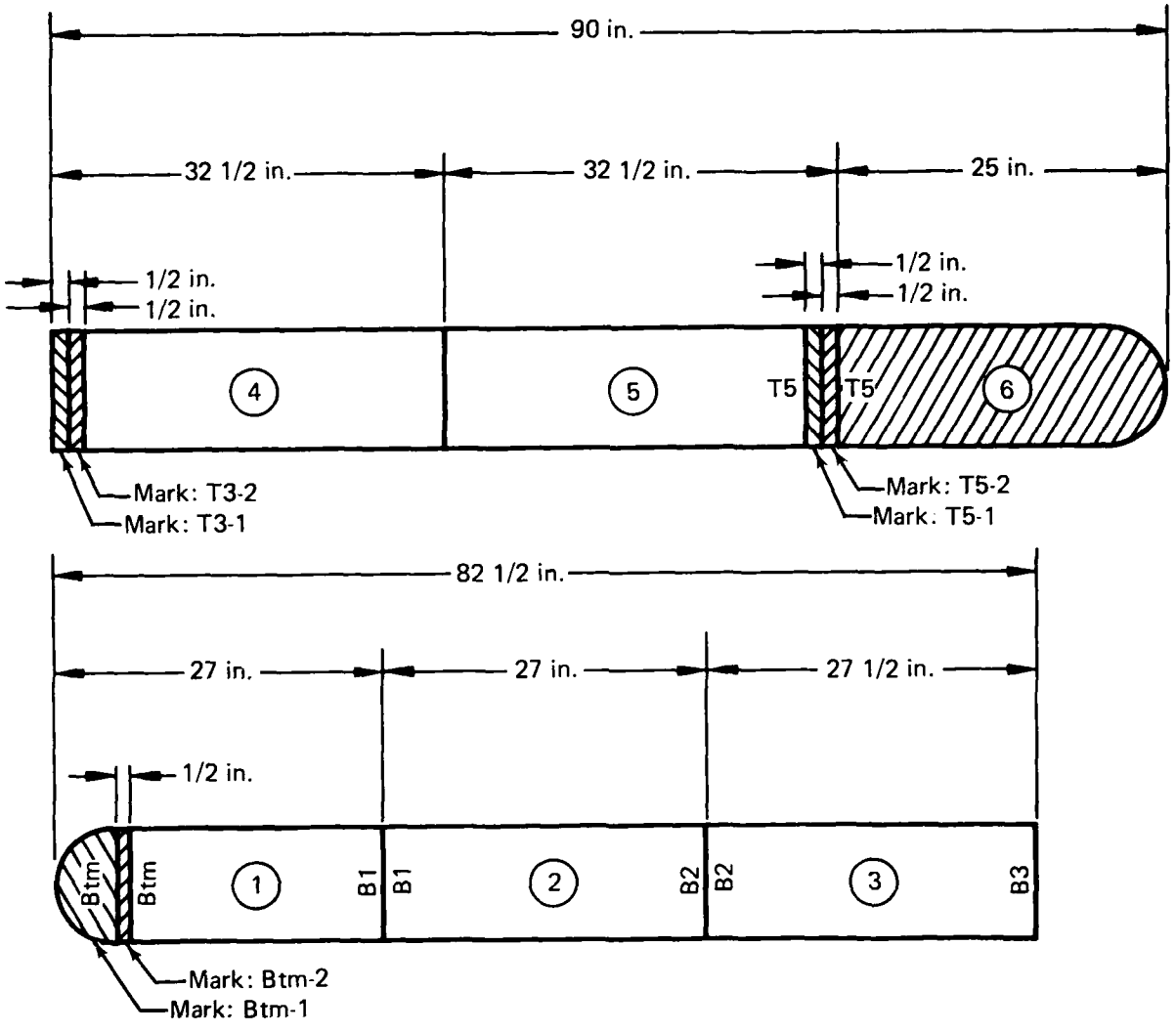


Figure 88. 6-in. RCS' Billet-Cutting Layout for Samples of Ingot No. 4115 Sent to P&WA

TABLE 58
AIRCO INGOT NO. 4115 CONVERSION MATERIAL BALANCE

Ingot No. 4115 — 1232 lb

6 in. RCS Samples	36
— (weight, hot top) —	118 forged to 6 in. RCS
— (weight, bottom) —	240 not forged
— (est. wt. 1 in. slice) —	39 structure study
billet	799
forged to 1 in. bar stock —	654 lb
% of billet to bar yield =	$\frac{654}{799}$ = 82%

Major portion of losses caused by flame cutting

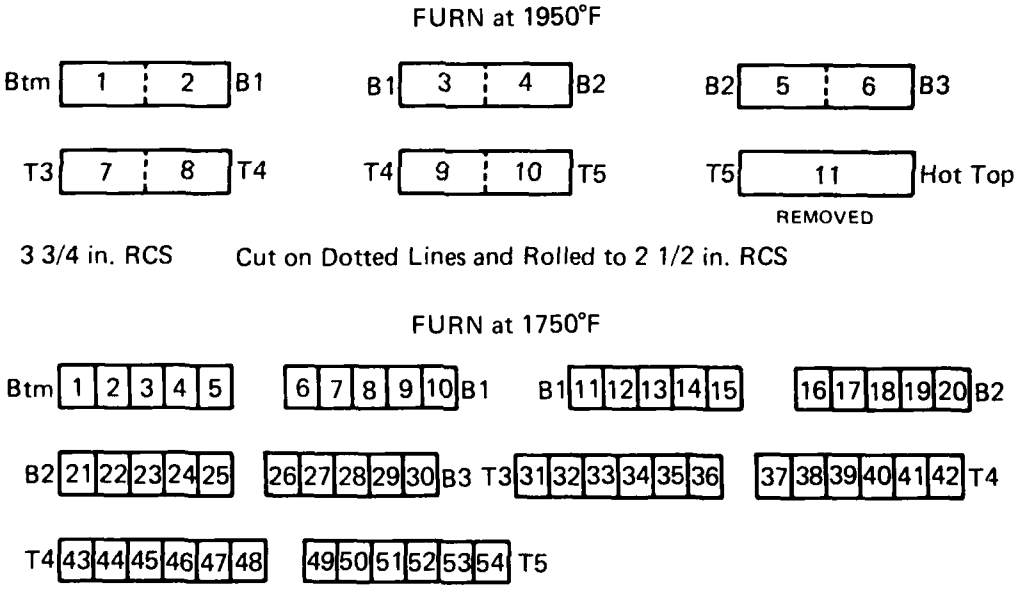


Figure 89. Identification of 1-in. RCS Bar Stock Rolled from AIRCO Ingot No. 4115

The conversion of the ingot and the hot rolling was accomplished in a routine manner, and forgeability was rated excellent. A minimum of surface conditioning was required, and visual inspection indicated no surface cracking. Details of the forging process are described later. Approximately 400 lb of barstock was forwarded to P&WA for further evaluation. The conversion of ingot No. 4116 was cancelled.

(4) Evaluation of AIRCO-Temescal EB Cold Hearth Melting Method

Eighteen-inch-diameter ingot No. 4115 was hot-forged to 3¾-in. bar and hot-rolled to 1-in. round-corner-square (RCS) barstock at AIRCO. NDI, chemistry, metallographic and mechanical properties evaluation was performed by both AIRCO and P&WA.

(a) Conversion Procedure

Ingot No. 4115, 18-in. dia, was hot worked to 1-in. RCS following the sequence shown in Figures 87 and 89. The bottom 6-in. was cut off prior to hot working. A 1-in. thick × 18-in. dia sample was then cut from the ingot for metallographic analysis. This sample was subsequently cut in two, and half delivered to P&WA.

The ingot was then coated with glass to minimize oxygen, nitrogen and carbon pickup, loaded into a furnace at 2050°F and held at temperature for 8 hr and inverted 1 hr before the first breakdown pass. Initial breakdown was made on a 1500 metric ton Loewy cogging press. The initial pass was made not exceeding 1-in. per side (2-in. over the dia). No second pass was permitted over the same surface of the ingot at any stage of the breakdown. The ingot was not forged below 1800°F to ensure forging above the beta transus.

In this manner, the ingot was worked down to 14-in. octagonal. Reheating as required, the ingot was then worked down to an 8-in. RCS, and hot chopped into two equal sections. A final breakdown was made to 6-in. round corner square.

The two 6-in. RCS billets were then track ground, conditioned, cut to appropriate length, and samples cut from the billets (Figure 88). Sample sections were sent to both AIRCO Research and P&WA for evaluation. The top, approximately 150 lb, was not forged further.

The billets were then loaded into a furnace at 1750°F maximum and equalized at temperature. Each billet was then worked down on a 1500-ton HPM forge press to 3¾-in. RCS.

After cooling, the billets were spot conditioned and reheated to 1950°F maximum. After equalizing at temperature to β anneal, the billets were rolled to 2.5-in. RCS. Upon cooling, the bars were flame cut to convenient length and reheated to 1750°F maximum. After equalizing at temperature, the bars were rolled to 1-in. RCS, straightened, air cooled, and sandblasted. Identification of bars was maintained throughout all stages of conversion; approximately 400 lb were sent to P&WA for evaluation. Bar Nos. 6, 30, and 52 were sent to AIRCO Research for preliminary testing.

A material balance of AIRCO EB ingot Nos. 4115 and 4116 is given in Table 59. As ingot No. 4116 was in essence a repeat of ingot No. 4115, it was decided jointly by AIRCO and P&WA that no additional useful information could be gained from ingot No. 4116. Consequently, this ingot was not forged.

TABLE 59
MATERIAL BALANCE OF AIRCO ELECTRON-BEAM-MELTED INGOTS

	AIRCO Ingot No. 4115		AIRCO Ingot No. 4116	
Input	*(lb)*	*(%)*	*(lb)*	*(%)*
Ti-6Al-4V Frankel Chips	558	38.8	573	38.7
Ti Sponge	719	50.0	740	50.0
60Al-40V Alloy	70	4.9	69	4.7
1/8 in. Al Shot	91	6.3	98	6.6
Total Blend	1438	—	1480	—
Hearth Skull	256	—	307	—
	1694	—	1787	—
Left in Hopper	15	—	14	—
Total Input	1679	—	1773	—
Output				
Ingot	1232	—	1212	—
Hearth Skull	307	—	359	—
Total Output	1539	—	1571	—
Melt Loss	140	—	202	—
Melt Efficiency		91.7		88.6

(b) Analyses/Results

Radiographic Inspection

Eighteen rolled "RCS" bars were radiographed at AIRCO; 33 bars were radiographed at P&WA using a sensitivity detection level of 2%. This sensitivity was validated by the radiographic detection of 0.023-in. dia WC particles placed on the 1-in. RCS bar. No high density inclusions were detected in any bars at AIRCO or P&WA.

Chemical Analysis

The 18-in. dia by 32.5-in. long, 1232-lb ingot No. 4115 was cut 7 in. from the bottom and a 1-in. thick section removed. The 6-in. RCS by 180-in. long forged billet was sectioned at the bottom, 82.5 in. from the bottom and 25 in. from the top (ref. Figure 88). These sections were chemically analyzed by AIRCO at center, midradius and edge locations.

Data generated are shown in Table 60. These data indicate consistent uniformity of Fe composition upon traversing from the ingot center-to-OD at each of the four locations examined, an important factor in minimizing undesirable beta segregation. The low propensity for centerline segregation in the EB melted ingot is probably due to the relatively shallow pool depth.

Hydrogen content of the ingot was low, but as expected, some increase in hydrogen level was obtained during the forging operation.

The 1-in. RCS rolled bar Nos. 6, 30, and 52 (representing the bottom, middle and top locations of the original ingot) were also chemically analyzed by AIRCO. P&WA performed similar analysis on bar No. 33. These bar data are shown in Table 61.

A review of all chemistry data shows that specification chemistry was attained except for 5 in. of ingot bottom and 25 in. of billet top, which had been cropped because of non-homogeneous ingot feed problems involving Al additions.

TABLE 60
CHEMISTRY OF AIRCO NO. 4115 INGOT AND BILLET

	Al	V	Fe	C	O	N	H	O_k*
	(wt. %)			(ppm)				
18-in. Ingot Slice (7-in. from Bottom)								
Center	6.72	3.78	0.185	558	1850	45	12.4	2277
Midradius	6.73	3.73	0.18	564	1859	67	3.0	2317
Edge	6.80	3.71	0.175	554	1930	61	1.2	2374
6-in. Billet (25 in. from Top)								
Center	5.77	3.72	0.18	693	2235	61	11.3	2772
Midradius	5.80	3.69	0.18	597	1928	26	23.5	2369
Edge	5.93	3.66	0.19	662	2335	54	39.5	2843
6-in. Billet (90-in. from Top)								
Center	6.11	3.96	0.21	612	1885	52.5	16.1	2358
Midradius	6.18	3.95	0.21	600	1986	54	8.0	2452
Edge	6.22	3.92	0.20	627	2066	66	46.7	2565
6-in. Billet (Bottom)								
Center	6.60	3.97	0.22	562	1855	33	13.5	2271
Midradius	6.61	3.90	0.22	603	1810	24	9.0	2230
Edge	6.71	3.82	0.21	572	1805	50	23.4	2248
Ti-6Al-4V Specification Max	6.75	4.50	0.30	1000	2000	500	125	
Min	5.50	3.50						

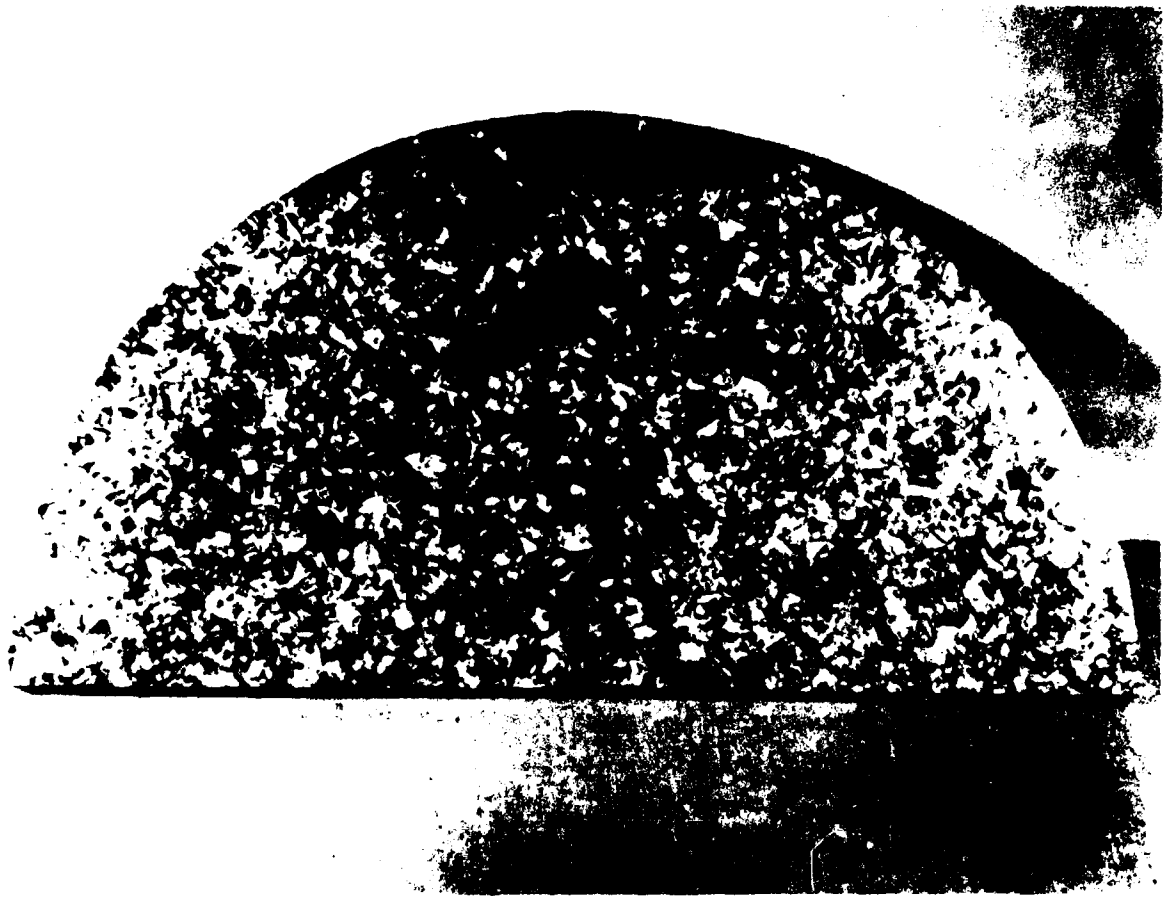
*O_k (Oxygen Equivalent) = O + 1.2 N + 0.67 C

TABLE 61
CHEMISTRY OF ONE-INCH RCS ROLLED BARS FROM AIRCO INGOT NO. 4115

	Ti-6Al-4V Specification		*Rolled RCS Bar No.*			
Element	*Max* (%)	*Min* (%)	*6(AIRCO)* (%)	*30(AIRCO)* (%)	*33(PWA)* (%)	*52(AIRCO)* (%)
Al	6.75	5.50	6.22	6.34	6.0	5.64
V	4.50	3.50	3.93	3.92	3.9	3.90
Si	—	—	—	—	0.05	—
Fe	0.30	—	0.21	0.21	0.16	0.22
O	0.20	—	0.187	0.191	0.19	0.206
C	0.10	—	0.053	0.053	0.06	0.056
N	0.05	—	0.0056	0.0064	0.009	0.0057
H	0.015	—	0.0047	0.0007	0.009	0.0036
B	—	—	—	—	< 0.002	—
Cu	—	—	—	—	—	—
Pb	—	—	—		< 0.001	—
W	—	—	—		< 0.05	—
Cb	—	—	—	—	< 0.05	—
Zr	—	—	—	—	< 0.01	—
Sn	—	—	—	—	< 0.01	—
Ni	—	—	—	—	< 0.1	—
Mo	—	—	—		< 0.05	
Mn	—	—	—	—	< 0.005	—
Cr	—	—	—	—		—

Metallographic Analysis

A transverse section of the 18-in. dia EB-melted ingot, taken 7 in. from the ingot bottom, was macroetched for structural analysis. Figure 90 shows equiaxed, 0.25-in. grain size with no evidence of columnar grains as normally found in arc-cast ingots. Apparently the minimal pool depth of the EB ingot influences the solidification rate and pattern to obtain the aforementioned desirable equiaxed structure.



Etchant: 5% HF

FD 171652

Figure 90. Transverse Macrostructure of AIRCO Electron Beam-Melted, 18-in. Diameter Titanium Ingot

A typical macroetch of one of the sections taken from the 6-in. RCS-forged billet is shown in Figure 91. Grains are equiaxed averaging 1/16 in., and slightly small at the billet surface compared to billet center.

Figure 92 shows typical transverse photomicrographs of the 1-in. RCS rolled bar showing equiaxed alpha structures. Upon heat treating at 1760°F/3 hr/AC, the structure was transformed to a globular primary alpha in a transformed beta matrix as shown in Figure 93. No areas of alpha or beta segregation were found in the 1-in. RCS rolled bar.

Etchant: 5% HF

FD 171653

Figure 91. Transverse Macrostructure of AIRCO 6-in. Round-Corner-Square Forged Titanium Billet

Mag 100X

ED 171654

Figure 92. Transverse Microstructure of AIRCO 1 in Round-Corner-Square As-Rolled Titanium Bar

Annealing at a lower temperature of 1740 F as utilized in mechanical properties testing failed to fully recrystallize the structure as shown by banding in Figure 94. This banding phenomena is not beta segregation as evidenced by microprobe studies which showed a

maximum iron level of 0.22% in one of the suspected regions in accord with an average iron level of 0.2% in the ingot. Banded structures can be eliminated by a higher final annealing temperature, beta annealing during forging and lower finishing temperatures.

Mechanical Properties

Tensile specimens of 0.252-in. dia and 1-in. gage length (AIRCO) and 0.190-in. dia by 1.3-in. gage length (P&WA) were tested according to ASTM specifications. Notched stress rupture specimens were machined according to ASM specification with a notch root diameter of 0.178 in. and a root radius of 0.006 in. (AIRCO and P&WA). A load was applied to produce a stress of 170 ksi. Charpy V-notch impact specimens had dimensions of 0.394 by 0.394 by 2.2 in. with a 45-deg notch angle.

Room temperature tensile data generated by AIRCO and P&WA are given in Table 62. These data all exceed specification minimums; the normal influence of aging temperature on strength and ductility is apparent.

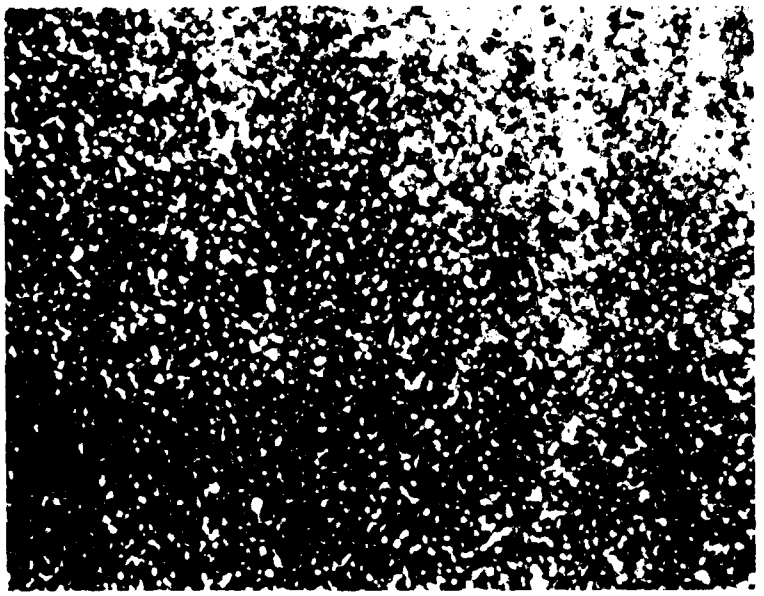
Notched stress rupture testing performed at room temperature exceeded the specification minimum life of 5 hr (with the test then discontinued) for the 1740°F/1 hr/WQ + 1300°F/2 hr/AC (AIRCO) and the 1750°F/1 hr/WQ + 1300°F/2 hr/AC (P&WA) heat treatments.

Charpy impact strength was determined by AIRCO as a function of temperature; data is presented in Figure 95. The typical range of impact data was also plotted in Figure 95 for general information. AIRCO impact data was comparable to the reference data.

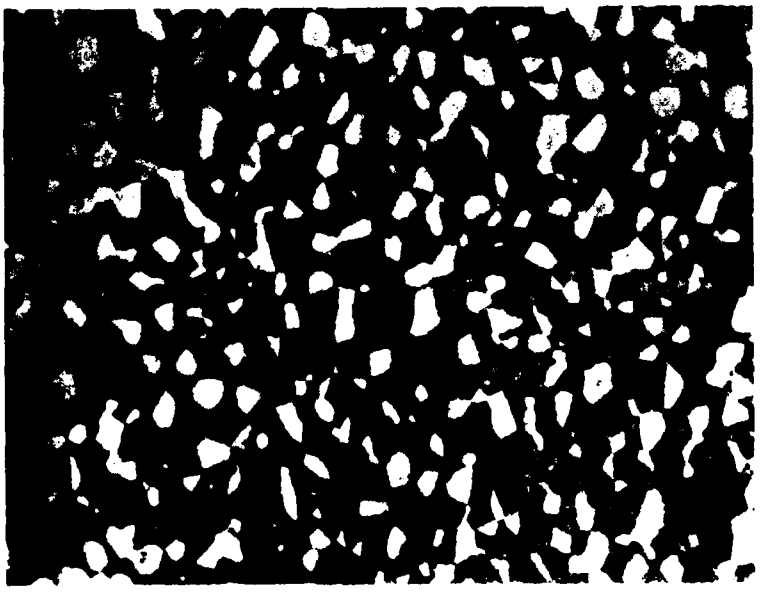
TABLE 62
ROOM TEMPERATURE TENSILE DATA OF ONE-INCH RCS ROLLED BARS FROM AIRCO INGOT NO. 4115

Bar No.	*Heat Treat**	*UTS (ksi)*	*0.2% YS (ksi)*	*El (%)*	*RA (%)*
6 (AIRCO)	A	156	146	21	42
30 (AIRCO)	A	151	143	17	41
52 (AIRCO)	A	151	142	21	43
6 (AIRCO)	B	175	166	16	38
30 (AIRCO)	B	180	172	16	37
52 (AIRCO)	B	172	164	12	34
33 (P&WA)	C	155.2	148.8	11.9	34.6
33 (P&WA)	C	155.2	149.2	10.2	36.2
33 (P&WA)	C	156.8	150.6	11.7	27.6

**Heat Treat* A - 1740°F/1 hr/WQ + 1300°F/2 hr/AC
B - 1740°F/4 hr/WQ + 1000°F/4 hr/AC
C - 1750°F/1 hr/WQ + 1300°F/2 hr/AC



Mag 100X

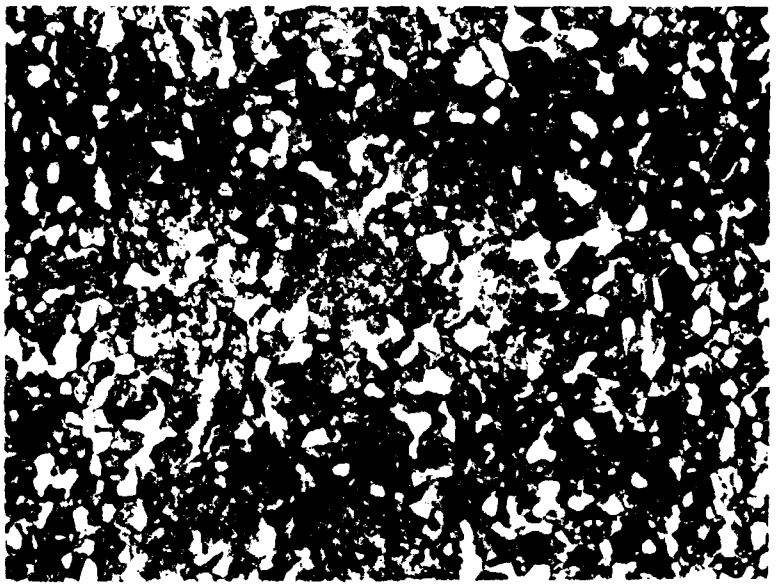


Mag 400X

FD 171655

Figure 93. Longitudinal Photomicrograph of AIRCO 1-in. Round-Corner-Square Rolled Titanium Bar After 1760°F/3-hr/AC Heat Treatment

Mag 100X



Mag 400X

FD 171656

Figure 91 Longitudinal Photomicrographs of AIRCO 1-in. Round-Corner-Square Rolled Titanium Bar Tensile Specimen After 1740°F/1-hr/AC Heat Treatment

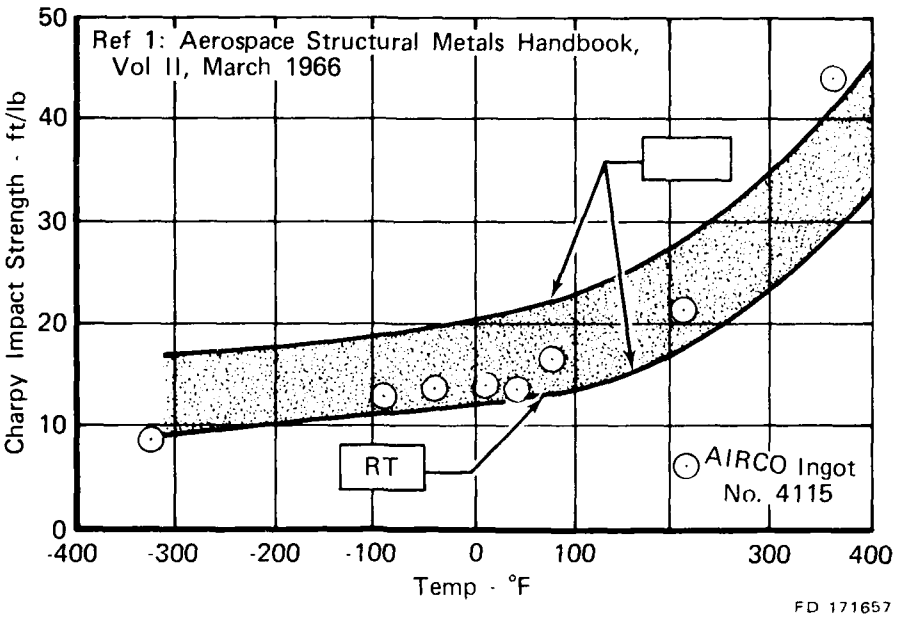


Figure 95. Impact Strength of 1-in. Round-Corner-Square Rolled Titanium Bar from AIRCO Ingot No. 4115

3. Phase I Teledyne Nonconsumable Rotating Electrode Melting System — Conclusions

Teledyne-Allvac has completed the requirements of Air Force Contract F33615-72-C-1126 for evaluation of nonconsumable melted titanium. Results of this program are pertinent to strategic materials reclamation objectives under this contract. The conclusions, as reported in the Teledyne-Allvac testing program of Ti-6Al-4V mill products made from nonconsumable skull melted material has demonstrated the following:

1. Nonconsumable skull melting, followed by either one or two consumable remelts, effectively eliminates nitride type inclusions.

2. All high density inclusions, such as tungsten carbide tool bit particles, are apparently removed provided they enter the molten pool.

3. High ratios of revert, either machining chips or solid scrap, may be used provided foreign alloy contamination and interstitial elements are at acceptable levels.

4. Single consumable remelting of nonconsumable process electrodes results in better chemical homogeneity than double consumable remelting.

5. Microstructural nonhomogeneity (i.e., areas of beta stabilization) increases with residual element content and is more prevalent in double remelted than in single remelted heats.

6. The manufacturing costs for nonconsumable melting exceed those for the conventional process (electrode fabrication plus consumable melt), but overall cost savings result from the ability to use lower cost scrap forms and through elimination of mill product losses for inclusion defects.

An evaluation of engine hardware produced from both single and double remelted Ti-6Al-4V has demonstrated the potential of the process to produce material with acceptable quality and mechanical properties. However, due to the limited scope of LCF testing on forgings with preferred microstructure there was insufficient data to reach a firm conclusion as to the acceptability of nonconsumable melted material for rotating parts in turbine engines.

2. Phase I AIRCO-Temescal EB Nonconsumable Melting Process — Conclusions

Ingots were melted and direct chill-cast by a single pass in an AIRCO EB furnace with a charge comprised of 39% Ti scrap chips. Upon review and analysis of all the aforementioned data, our Phase I conclusions are:

1. Seeded WC tool bit contamination was completely separated to give an uncontaminated ingot.

2. Chemical specification levels were attained with the exception of both ingot ends.

3. Nonhomogeneous ingot-end chemistry (i.e., % Al) must be resolved; feed system alteration is a likely corrective action.

4. Desirable ingot homogeneous macrostructure (equiaxed and relatively fine grain) and bar microstructures were attained.

5. All mechanical property data generated (tensile, stress rupture, impact) exceeded applicable specification minimums.

SECTION VIII
SCRAP MANAGEMENT SYSTEMS

A. SURVEY OF AEROSPACE SCRAP

Precontract research by P&WA revealed inadequate published information concerning the generation and disposition of aerospace titanium- and nickel-base scrap metal; consequently, a survey of aerospace scrap was established as a major element of the Phase I management portion of the contractual effort. Analysis of this type of survey should provide answers to many of the existing questions on scrap generation and usage:

- What constitutes aerospace scrap metal and where does it originate?
- What are the physical forms of this scrap?
- What levels of quality are generated?
- What aerospace scrap is generated at the melters' plants?
- What total quantities are generated and how do the quantities compare with anticipated quantities?
- What is the disposition path for aerospace scrap metal?
- Does export of aerospace metal have a major or minor effect on domestic melting?
- What are the possibilities of recycling ECM sludges and various grindings?
- Can aerospace scrap usage be increased for critical aerospace applications?

The basic objectives of the scrap survey were to determine the quality, quantity and disposition of the titanium- and nickel-base scrap generated by the American aerospace industry. A significant portion of the survey work was subcontracted to Suisman & Blumenthal Inc., a Hartford, Ct. aerospace scrap dealer/processor with extensive experience in plant service scrap removal, processing and marketing of titanium- and nickel-based scrap.

This survey comprises three major sections: (1) background information/industry contacts, (2) aerospace questionnaire/inquiry and results, and (3) potential improvement of aerospace scrap recycling.

1. Aerospace Background Information/Industry Contacts

a. Aerospace Scrap Sources

Scrap metal is generated at all points in the raw material flowpath from ore to final product and finally, upon obsolescence of the final product. The great majority of scrap, however, is generated from three sources, defined as follows:

Source (1) Ingot Production Scrap — Scrap generated by melters during ingot production (i.e., end croppings, collars and turnings); averages 5% of the total melted material (ref. 1).

Source (2) Ingot Conversion to Mill Products Scrap — Scrap generated during conversion of ingot to mill product, i.e., bar and billet ends, sheet and plate trimmings; averages 35-40% of the ingot weight. The ratio of solids to chips for ingot production and conversion scrap is estimated to be 70/30.

Source (3) Mill Products to Manufactured End Products Scrap — Scrap generated by forgers and machine shop/fabricators during manufacture of final end products, i.e., forgings, finished components, averages 70-75% of the mill product weight, and is estimated to have a 30/70 ratio of solids-to-chips. A portion of this scrap is generated in a form which is highly susceptible to physical loss or has such a low recovery potential that it is disposed with no accountability (e.g., fines, sludges, chemical removal products). The percentage of scrap estimated to be in this "material loss" category is 15-20%.

b. Home Scrap Generation and Disposition

Scrap metal generated by Sources (1) and (2) is called "home scrap" because a majority of this scrap is of identifiable composition and of suitable quality for direct in-house recycling to ingot. It is estimated that 75% of this scrap is recycled to ingot, while the remaining 25% is disposed to the open market or is considered as material lost. This scrap accounts for 40-50% of the ingot weight.

As an example, approximately 58 million lb of ingot were actually produced in 1973, of which 50 million lb were consumed (inventory balance = 8 million), while about 30 million lb of mill produce were made. The resultant home scrap, i.e., Sources (1) and (2), was estimated at 20 million lb (40% of the consumed ingot weight), of which 15 million lb (75%) was internally recycled.

It is cost-effective for melters to close-loop-recycle home scrap back to ingot where possible. Principal restraints to remelting of home scrap include the problem of alloy intermixing, unusable scrap forms, and customer limitations on scrap usage.

c. Open Market Scrap Generation and Disposition

Scrap metal generated by Source 3 (forgers and machine shop/fabricators) accounts for the majority of open market scrap, i.e., scrap disposed to scrap dealer/processors. This scrap is potentially upgraded, and made available for competitive purchase by the aerospace, steel and aluminum industries. Open market scrap is the largest category of recyclable scrap.

To illustrate, again using 1973 titanium production data, an estimate may be made of the amount of aerospace scrap that could be expected to be available in the open market. Approximately 30 million lb of titanium mill product were produced in 1973. Aerospace consumption comprised approximately 80-82% of this total. Applying the previously noted scrap generation parameters to this total, including 81% aerospace usage, 73% aerospace scrap generation and 17% material loss, a total of approximately 15 million lb of titanium aerospace scrap could have been expected to reach the open market in 1973, assuming no hoarding by fabricators (a very limited amount of hoarding is believed to occur). This total may be somewhat increased by the limited disposition of nonrecycled home scrap, and by the obsolescent, finished-part open-market scrap.

A similar analysis for nickel-base alloys is more difficult and less precise because of the multiple applications for nickel alloys and the lack of industry statistics. Utilizing an estimate of 45 million lb of mill product consumption by aerospace, and the above scrap generation parameters, a total of 27 million lb of nickel-base scrap could have been expected to reach the open market in 1973.

Open market scrap, generated chiefly from the aerospace manufacturers, is the flexible supply of raw material which the aerospace melter draws upon as required. Open market scrap is almost always treated by the aerospace melter as supplemental to virgin metal and home scrap generation. When the aerospace melter's furnace is operating at high capacity, the melter buys in the open market at a high volume level, being limited chiefly by customer scrap limitations. During periods of low production, open market aerospace scrap purchases are severely curtailed or eliminated. In general, open market scrap is less effectively recycled to aerospace ingot than home scrap due to competitive market demands, increased quality problems, i.e., identification/contamination, and less desirable scrap generation forms. It is estimated, for example, that only 25 to 40% of titanium open market scrap is recycled to aerospace ingot.

During the last two years, competitive demand has tended to reduce the recycle percentage towards the lower end of the range. This is attributable to the high demand of the steel and aluminum industries and is primarily associated with the increased production of a Ti bearing steel grade (AISI 409) for automotive catalytic converters. This trend is likely to continue in the near term and may be further supported by aluminum industry demand associated with the introduction of aluminum automotive blocks. The ratio of steel-to-aluminum demand is currently over 10 to 1. It may be expected that 30-40% of Ti scrap diverted to the steel industry will be exported. It is generally the lower grade fraction which receives this disposition. Further insight on the scrap export situation is provided in a following section.

Thus, the major factors influencing titanium scrap disposition are scrap quality and market demand. Low grade scrap necessarily moves to the steel industry because of its unacceptability for aerospace recycle. The appetite of the steel industry is largely satisfied by low grade scrap. Increases in steel demand and establishment of methods for upgrading scrap will tend to increase the competition for aerospace scrap.

When open market and home scrap recycling is considered, the current disposition of aerospace titanium scrap may be represented as follows:

Titanium Aerospace Recycle	—	< 50%
Steel Production	—	> 50%
Aluminum Production	—	< 5%
Direct use for other titanium products	—	< 5%

The disposition of nickel-base alloy scrap follows a trend similar to titanium with the exception that a higher level of open market scrap is recycled due to less stringent quality requirements. As an example, WC tool bit contamination in nickel-base alloy scrap does not inhibit aerospace recycling. Unfortunately, the multiple industrial applications for nickel-bearing alloys precludes accurate determination of recycle percentage, but it is estimated that as much as 75% of the high nickel aerospace grade open market scrap is recycled back to aerospace ingot via scrap dealer/processors.

For both titanium and nickel scrap, the vast majority of scrap generated by forgers and fabricators of final products is disposed to the open market rather than disposed in a closed-loop aerospace recycle. Although disposition to the open market results in exposure of aerospace scrap to non-aerospace distribution avenues, aerospace companies have shown a clear preference for this approach based on the following factors:

- A reluctance of aerospace companies to expend effort and investment in the handling and upgrading of scrap metal, which is the by-product rather than product of their operation;
- The difficulties in providing guaranties of premium aerospace scrap chemistries and in marketing all of the non-premium scrap grades;

- A reluctance to cope with the unpredictable nature of a scrap market, which may require holding of scrap to achieve a reasonable value.

d. Export of Open Market Scrap

The export status of aerospace scrap is significant to the subject of potential domestic shortages. The preponderance of Free World ingot production, both for titanium and high nickel alloy is located within the continental U.S. The highest use, and normally, the most attractive price for these scrap metals is within America.

In aerospace scrap metals, as in most other scrap metals, the domestic handlers of scrap prefer to ship domestically. Several reasons exist for domestic preference. The shipments are for shorter distances, and therefore, freight rates are lower. Packaging is usually simpler within the domestic market than "for export" packaging. The mechanics of selling, i.e., the language, specifications, contracts, are normally far easier to handle domestically. Financial problems and risks are diminished if domestic sales can be consumated.

Since the major markets are in America and shippers prefer to avoid export shipments, the overwhelming quantities of aerospace metals which can be melted here remain in the continental U.S. However, exports do occur in aerospace scrap metals if a given scrap grade is not competitively required by U.S. melters or (on a temporary basis) the U.S. melters cannot consume all the aerospace scrap metal being generated.

The degree to which titanium or nickel alloy scrap was exported is difficult to ascertain. A tabulation of exported titanium alloys (unwrought waste and scrap) averaged 4,594,000 lb per year between 1966 and 1970 (November):

Titanium Unwrought Waste and Scrap, Exports
(In Thousands of lb)

1966	3465
1967	2858
1968	5506
1969	5604
1970 (November)	5538

One problem is that the designation of this class of export, "Unwrought Waste and Scrap" allows the inclusion of non-scrap items. Determination of which shipments are in reality titanium scrap is very difficult, since designations for the exported item are often ambiguous. A close study of the monthly 1974 U.S. Government figures showed that at least 24% of the weight was clearly non-scrap.

In addition to the lack of accurate export figures, another problem is the type of scrap that is actually being shipped. It would be erroneous to assume that 4,129,000 lb (5,506,000 × 75%) of meltable scrap were exported in 1968. In reality, the majority of the scrap metal exported was in the form of titanium turnings, and as such, was generally not acceptable to U.S. titanium ingot makers.

Similar problems arise in analyzing the export of aerospace scrap nickel alloy scrap. The following tabulation of exported nickel scrap is misleading in that non-aerospace grades and low/medium content nickel grades are included:

Nickel Waste and Scrap, Exports
(In Thousands of lb)

1965	13,466
1966	11,752
1967	27,783
1968	33,485
1969	36,056
1970	19,093
1971	12,879
1972	16,505
1973	12,524

As in titanium, much if not the majority of this nickel alloy scrap could not find melting application, at competitive prices, in the U.S. Some nickel alloy scrap has cobalt or copper inclusions which preempt or severely limit its use here; other grades, especially turnings, are composed of several intermixed grades which U.S. alloy melters generally decline to buy.

To summarize, net export shipments of aerospace scrap titanium and nickel alloys are less than the totals shown in government statistics and they represent, overwhelmingly, metal which cannot be readily absorbed into the U.S. aerospace melting systems at the present time. To the degree that these exports support the U.S. policy of free trade and improve the U.S. balance of payments, they serve a national purpose. These exports also serve to keep open the channels of imports; and, there are occasions when imported aerospace scrap metals may be of crucial importance to domestic melters.

Participants in the Strategic Material Reclamation Seminar, (see Section IV), agreed that these open channels of trade should be maintained. The titanium panel, when discussing future trends, stated that there was a general concern "over the imposition of market controls on an international commodity such as scrap." Similarly, the superalloy (nickel) panel recommended that "restrictive legislation of the scrap market should be discouraged."

e. *Grindings and Sludges Generation and Disposition*

Industry contacts on grindings and sludges offered disposition comments such as "hauled away for nothing," "disposed," "landfill," and "no value." As discussed later in Survey Results, only an insignificant quantity of these wastes were reported sold, apparently for some type of recycling. At the Strategic Materials Reclamation Seminar, the superalloy panel stated that "the second category includes unsegregated low grade wastes, such as ECM and EDM sludges and pickle liquors and metallurgical smokes, which would have to be treated chemically to recover their values."

The answer to the potential for recycling lies, unfortunately, not in the desirability of reclamation but rather in the basic nature of these sludges and grindings. These wastes are very often intermixtures of a number of alloys of titanium or of nickel, and sometimes both. Almost always, the wastes have nonmetallics intermixed to a significant degree; in the case of grindings, for example, nonmetallic abrasives are broadly interspersed through the waste. Oil and water are frequently used in the manufacturers' processing and high moisture contents are characteristic of ECM and EDM sludges. The actual metallic content may be remarkably low, less than 20% of the weight since some of these wastes are oxidized.

Currently, none of this material, or very little of it, finds its way to airmelt or even refinery remelting today. The heterogeneous nature of sludges and grindings and their frequently low metallic content implies that remelting will not be cost-effective; to state the reverse, there are many alternative, less expensive primary and secondary melt materials available for remelting at this time.

The reclamation of grindings and sludges as applied to aerospace recycling seems distant. Reclamation processes are being considered, and technological advances and/or national metal shortages may develop which would elevate these sludges and grindings for refinery or airmelt use, but their present use for aerospace recycling is not apparent.

As a contrast to the wastes which are generated at manufacturing plants, those at melters facilities are in a number of instances recycled. At the melter's plant, nickel and titanium are not normally processed together, the range of alloys is limited and the moisture is low if present at all. Remelting of identifiable grindings is utilized for the melting of lower-grade stainless steels, etc. In summary, grindings and sludges have some disposition to the less-critical steel/stainless remelting sources but have not attained any reclamation disposal to the aerospace industry.

2. Aerospace Scrap Questionnaire/Inquiry

The key element of the survey was a questionnaire/inquiry directed by the prime contractor to a large segment of the aerospace industry. The questionnaire/inquiry for the open market survey (Figure 96) was developed jointly by P&WA and the scrap processor subcontractor Suisman & Blumenthal, Inc. The inquiry was directed towards a determination of scrap quantities disposed to the "open market." The survey also invited information on in-house recycled scrap in order to enable overall raw material accountability. It is known that melters and casters effectively recycle in-house scrap; however, actual quantities recycled are frequently considered proprietary by these industries, and therefore an incomplete response to this portion of the survey appeared likely. The year 1973 was chosen as a baseline because that year appeared to be a typical year for the fluctuating titanium industry, with ingot production comparable to that of the previous peak in 1969, yet less than the 1974 output, as indicated by the following data:

Millions of lb

Year	*Ingot Output*	*Mill Products*
1974	73.32 est.	35.40 est.
1973	57.97	29.50
1972	40.53	25.25
1971	36.76	22.48
1970	49.46	29.02
1969	56.85	31.88
1968	36.65	23.80

The nomenclature used in the questionnaire was based on definitions provided by the scrap industry. The physical forms of scrap are classified broadly as "solids," "turnings," "grindings," and "sludges." Solid forms are of varied shape and include material such as bar ends, sheet trimmings, castings, forgings, rings, etc. Turnings are the shavings from various machining operations; the term "turnings" is generally interchangeable with the term "chips," although a narrower interpretation given to chips is crushed turnings. "Grindings" are the product of grinding operations which produce a relatively dry by-product. "Sludges" covers the relatively wet by-product of a variety of operations including EDM, ECM, grinding and pickling baths.

Pratt & Whitney Aircraft DIVISION OF UNITED AIRCRAFT CORPORATION

TO:

SUBJECT: SCRAP RECLAMATION

We seek your participation in a program which offers potentially great benefits for your company, the aerospace industry, and the defense procurement system.

The problems of raw material availability and cost are becoming increasingly evident. Material recycling can play an important role in alleviating these problems. In recognition of this, the Air Force Materials Laboratory recently initiated a program, "Strategic Materials Reclamation", which is being managed by Pratt & Whitney Aircraft. The purpose of the program is to identify and evaluate methods for increased recycling of aerospace scrap within the industry. Towards this objective, a Strategic Materials Reclamation Seminar was held May 14-15, 1974 in Hartford, Connecticut. A copy of the Seminar report, which includes a description of the overall program, is enclosed (or has been previously forwarded) for your use.

One conclusion of the Seminar was that sufficient information is not currently available on the type, quantity and disposition of aerospace scrap to enable a thorough assessment of potential reclamation methods. We seek your assistance in accumulating this necessary information, and have accordingly attached a scrap survey questionnaire. Information obtained by this questionnaire will be combined with scrap dealer and government input to produce a complete survey. Your information is requested by February 28, 1975 in order to allow timely completion of the overall survey.

The survey seeks information on scrap which is disposed to the open market. Information on scrap which is internally recycled is of interest to provide accountability, but not essential to the survey. No information on individual company input will be published or made public.

The Air Force Materials Laboratory joins us in urging your timely attention to this request. It is noteworthy that both nickel and titanium, the basic aerospace defense materials, come predominately from foreign sources, so that any greater utilization of American Aerospace scrap metals tends to lessen our dependence on the uncertainties of world trade. AFML will provide, upon request, interim reports on the overall program, including final survey results, to responders.

J. E. Flynn

J. E. Flynn
Materials Engineering and Research Laboratory
Pratt & Whitney Aircraft

EAST HARTFORD CONNECTICUT 06108

INFORMATION ON COMPLETING SURVEY

1. The survey covers the calendar year 1973 only.
2. Figures are requested for the total of all individual plants in your company.
3. Survey forms should be returned by February 28, 1975 to:

   J. E. Flynn
   Materials Engineering and Research Laboratory
   Pratt & Whitney Aircraft
   400 Main Street
   East Hartford, Connecticut 06108
   (203) 565-4488

4. DEFINITIONS

Scrap Type		
Solids	–	Bars, sheet, forgings, wire, castings, finished and semi-finished parts, etc.
Turnings	–	Chips, turnings, shavings
Scrap Quantity	–	Metal weight in pounds (lbs.)
Scrap Disposition		
Open Market	–	Scrap disposed to scrap dealers or other bidders.
Internally recycled	–	Scrap which is recycled in-house or transferred to another aerospace firm for recycling.

Figure 96. Open-Market Scrap Reclamation Questionnaire/Inquiry

FD 171658

SURVEY

I. Locations

Plants Included

II. Solids/Turning

Titanium

	Solids, (lbs.)	Turnings, (lbs.)	Disposition
Mixed/ Contaminated			
Segregated: 6/4			
C. P			
5-2.5			
6-2-4-2			
8-1-1			
6-6-2			

If a significant portion of your purchased titanium is machined by vendors, what is the estimated total of this out-of-house scrap in pounds (lbs.) of titanium or what is the estimated % of your machining done out-of-house?

Nickel

	Solids, (lbs.)	Turnings, (lbs.)	Disposition
Mixed/ Contaminated			
Segregated: Inconel and Inconel X			
Inco 901			
A-286			
Hastelloy X			
Inco 718			
Rene 41			
Waspaloy			
IN-100			
Udimet 700			
B-1900			
Inco 713			
Rene 80			
Inco 738			

If a significant portion of your purchased nickel-base alloys are machined by vendors, what is the estimated total of this out-of-house scrap in pounds (lbs.) of nickel alloy or what is the estimated % of your machining done out-of-house?

FD 170371

Figure 96. Open-Market Scrap Reclamation Questionnaire/Inquiry (Continued)

The major source of generation of aerospace metals, in the open market, is the "aerospace manufacturing plant." The aerospace manufacturing plants fall into five major categories: Airframe, Engine, Aerospace Equipment, Forgers and Machine Shop/Fabrications.

A limited amount of scrap is disposed to the open market, i.e., to aerospace scrap processors, by melters, casting houses and final product users. This scrap is generally of a poor grade for recycling and therefore was not considered in the main body of the survey. Survey data received from these sources was anticipated to be relatively incomplete due to the reduced interest in maintaining records of this scrap grade. However, a summary of responses received from these sources is given as supplementary survey information. Published literature and industrial contacts, particularly in the scrap processing and melting industries, were utilized to provide further information.

P&WA directed the scrap questionnaire-inquiry to 83 aerospace-associated companies in various industrial categories. These companies represented, collectively, over 60% of the scrap generation for their particular industrial group (Figure 97). It was necessary to make extrapolations of reported data to obtain complete industry data for subsequent analysis. A few sources of aerospace scrap, such as the metal powder producers, were excluded from the survey because of the small quantities involved.

Following the original survey inquiry, a supplemental survey inquiry was made to 11 aerospace companies in pertinent industrial categories to determine the 1973 production quantity of titanium and nickel-base grindings and sludges. The supplemental questionnaire inquiry for grinding and sludge scrap is shown in Figure 98, recipients and respondents in Figure 99.

3. Aerospace Scrap Survey Results

The scrap generation data obtained by survey was separated into three distinct groups for purposes of analysis and summary:

Summary I groups the information on scrap solids and turnings submitted by the Airframe, Engine, Aerospace Equipment, Forgers and Machine Shop Fabrication Industries. This group accounts for all the aerospace scrap generated from the processing of mill products. This scrap is the mainbody of open market, potentially-recyclable scrap.

Summary II groups the information on open market scrap solids and turnings submitted by the Melting, Casting and Final Product User Industries. This scrap quantity data is presented as a separate summary because it represents a significantly lower quality type and, as such, has questionable reclamation potential. It has been previously noted that melters and casters efficiently recycle their home scrap and, therefore, the fraction of this scrap released to the open market is likely of low quality. Used-part scrap, although variable in quality, i.e., dependent on processing and service history, has generally been treated as a high risk category for recycling to aerospace.

No information on in-house recycled scrap from melters and casters has been presented because of the very incomplete input.

Summary III groups the information on grinding and sludges submitted by all of the above industry categories with the exception of Final Product Users. This scrap represents a specialized category with questionable reclamation potential.

SCRAP SURVEY RECIPIENT AND RESPONDENT LISTING

TITANIUM MELTERS	NICKEL MELTERS	FORGERS	CASTERS	MACH./FAB. VENDORS
*Timet	*Special Metals	*Wyman Gordon	*Howmet	*Rohr
*RMI	*Teledyne Allvac	*Reisner	*TRW	*Excello
Oremet	*Carpenter Tech.	Ladish	*Arwood	*TRW
*Crucible	Latrobe	*Cameron	*Precision Foundry	
*Teledyne	*Crucible	*Taylor	*Jetshapes	
*Howmet	*Stellite	Alcoa	Hitchiner	
Martin	Universal-Cyclops	*TRW	REM	
	*Cameron	*American Welding	*PCC	
Ti-West	*Huntington	*Monroe	*Ti-Tech	
	*Allegheny Ludlum	*Edgewater		
	Cannon Muskegon	King Fifth		
	*Babcock Wilcox	*Carlton		
	Simonds Steel	Airco Viking		
		Schlosser		
		*McWilliams		
		*SIFCO		

USERS			END USERS		
ENGINE	AIRFRAME	AEROSPACE	AIRLINES	MILITARY	INDEPENDENT O/H CENTERS
*P&WA	*Grumman	*Rockwell Int.	*American	AFLC	Cooper Airmotive
GE	*McDonnell	*Bendix	*United	*Okla. City	*Pacific Airmotive
*Allison	*Boeing	*Hamilton	*TWA	*San Antonio	
*Lycoming	*Lockheed		Pan Am	NAS Norfolk	
*AiResearch	*Northup		*Eastern	NAS Pensacola	
Teledyne CAE	*General Dynamics		*Delta	NAS Jacksonville	
	Republic			NAS Alameda	
	*Sikorsky			NAS San Diego	
	*Ryan				
	*Bell			Navy ASO	
				*Defense Supply Agency	

*Response received.

FD 171659

Figure 97. Scrap Survey Recipient and Respondent Listing

GRINDINGS AND SLUDGES SCRAP RECLAMATION QUESTIONNAIRE/INQUIRY

Pratt & Whitney Aircraft DIVISION OF UNITED AIRCRAFT CORPORATION

TO

SUBJECT SCRAP RECLAMATION

The receipt of your information on scrap metal generation and disposition is gratefully acknowledged. Your input is being combined with that from other sources to complete a major portion of the aerospace scrap survey. The completed survey will be documented in reports to the Air Force Materials Laboratory. You are reminded that these interim reports on the Strategic Materials Reclamation program are available, upon request, from

Kenneth Love, AFML/LTM
Wright-Patterson AFB, Ohio 45433

To complete the aerospace scrap survey, we need information on an additional category of scrap material, i.e. grindings and sludges. This information was not requested in the initial survey because these forms of scrap are generated by only a limited number of companies. Accordingly, we have attached a supplemental scrap survey questionnaire. Your information is needed by 31 May. Again, thank you for your attention to our initial survey, and for your consideration of this supplemental request.

Pratt & Whitney Aircraft

J. E. Flynn
Program Manager

EAST HARTFORD, CONNECTICUT 06108

INFORMATION ON COMPLETING SURVEY

1 The survey covers calendar year 1973 (use 1974 if 1973 data is not available.)

2 Figures are requested for the total of all individual plants in your company.

3 Survey forms should be returned by to

J. E. Flynn
Materials Engineering, J. Bldg.
Pratt & Whitney Aircraft
400 Main St.
East Hartford, Conn. 06108
(203) 565-4488

4 DEFINITIONS

Scrap Type		
Grindings		Product of either a dry or wet grinding operation
Sludges		Product of ECM, EDM or other operations which produce a wet product
Scrap Amount	–	Weight in pounds (dry grindings), or amount in gallons (wet grinding, sludges). Indicate % of nickel or titanium contained, if known.
Scrap Source	–	Indicate the fabrication operation which generates the scrap.
Scrap Disposition		
Disposed	–	Scrap disposed with no compensation
Open Market	–	Scrap sold to scrap dealers or other bidders
Internally Recycled	–	Scrap which is recycled in-house

FD 171660

Figure 98. Grindings and Sludges Scrap Reclamation Questionnaire/Inquiry

SURVEY

I. NICKEL CONTAINING GRINDINGS

	Amount (lbs or gals.)	Source	Disposition
Mixed Alloy			
Segregate Alloy			

II. NICKEL CONTAINING SLUDGES

Amount (gals.)	Source	Disposition

III. TITANIUM BASE GRINDINGS

	Amount (lbs or gals.)	Source	Disposition
Mixed Alloy			
Segregated Alloy			

IV. TITANIUM BASE SLUDGES

Amount (gals.)	Source	Disposition

FD 170372

Figure 98. Grindings and Sludges Scrap Reclamation Questionnaire/Inquiry (Continued)

Titanium Melters	Nickel Melters	Forgers	Casters
Timet	*Special Metals	*Wyman Gordon	*Howmet
RMI	Teledyne Allvac	*Reisner	*TRW
*Crucible	*Carpenter Tech.	*Cameron	*Arwood
*Teledyne	Crucible	Taylor	*Precision Foundry
Howmet	Stellite	*TRW	*Jetshapes
	*Cameron	*American Welding	*PCC
	*Huntington	*Monroe	*Ti-Tech
	*Allegheny Ludlum	*Edgewater	
		*Carlton	
		McWilliams	
		*SIFCO	

Mach./Fab. Vendors	Users		
	Engine	Airframe	Aerospace
*Rohr	*P&WA	*Grumman	*Rockwell Int.
*Excello	*Allison	*McDonnell	
*TRW	*Lycoming	Boeing	
	AiResearch	Lockheed	
		*Northrup	
		*General Dynamics	
		*Sikorsky	
		*Bell	

*Response received.

FD 171661

Figure 99. Sludge and Grinding Survey Recipient and Respondent Listing

a. *Principal Titanium (Ti) and Nickel (Ni) Open Market Scrap Data (Summary I)*

Summary I information is presented in Tables 63 through 74. Tables 63 and 64 present the overall quantity information for titanium and nickel, respectively, while Tables 65 through 74 present a breakdown by alloy type and character. The survey goal of determining hard facts on the quantity of open market scrap was largely achieved by the Summary I information.

A precise determination of these scrap quantities was precluded, however, by the failure to receive a few responses from large scrap generators. Included among companies which declined to provide data were a large engine manufacturer and two large forgers. Nonetheless, the estimated industry totals indicated in Tables 63 and 64 are believed to be reasonably accurate. Estimates for companies which did not respond were made by applying a ratio of product output in pounds for the unknown source to a known source and applying this ratio to the scrap quantity of the known source. For example, the total weight of engines produced in 1973 by the nonresponding engine maker were compared to the total weight of engines produced by P&WA in 1973, and this ratio was applied to the total quantity of P&WA scrap to estimate their generated scrap. Airframe estimates were similarly based on data for delivered aircraft. Forging scrap estimates were based on production ratio data for both titanium and nickel alloys. Aerospace equipment estimates were based primarily on data for the percent consumption of mill products by aerospace relative to Engine and Airframe Manufacturers. Estimates for Machine Shop/Fabricators were based on the premise that 55% of Airframe/Engine/Aerospace material is machined/fabricated by out-of-house vendors. The utilization of the 55% figure was based on survey responses to the question on out-of-house machining.

The overall titanium quantity survey results (Table 63) of 13,278,000 lb compares reasonably well with the 15 million lb total titanium scrap deduced (ref., Open Market Scrap Quantities, para. A-1c). The largest potential inaccuracy in the estimated data would be in the Machine Shop/Fabricator totals, while the Engine and Forger totals would be subject to some error due to nonrespondents. The major potential error in the deduced figures would be in the assumed material loss percentage and the fact that some 1973 scrap was generated from 1972 mill products; 1972 was a lower production year than the 1973 production year utilized.

The results for the nickel-base scrap quantity follow a pattern similar to titanium. The potential for inaccuracy, however, is considered to be substantially greater for the nickel-base alloy scrap because of the multiple avenues of generation and disposition, and the large number of alloy types. A comparison of the estimated industry total vs a deduced total is also not as meaningful for nickel because of a lower degree of confidence in each of these figures. A significant contributory factor to the greater difference noted between deduced and estimated nickel totals is that the largest generator of nickel forging scrap has a captive melt shop and hence none of this scrap enters the open market.

Tables 65 through 74 give further definition to scrap character as a function of the industry generating the scrap. Four industries produce relatively equivalent quantities of the total Ti scrap: (30% Machine Shop/Fabricators; 22-23% each for Airframe, Engine and Forgers). Three industries produce the major quantities of total Ni scrap (39% Engine, 30% Machine Shop/Fabricators, 27% Forgers). Upon applying these major scrap producing industries to their surveyed record of segregation/contamination, it may be noted that only the Machine Shop/Fabricator industry appears to have a relatively high level (40%) of mixing/contamination for Ti. A range of 10-31% mixing/contamination was found for the remaining major scrap producing industries when applied to Ti and Ni. This implies that industry segregation is active, yet there is ample room for improvement in all industries surveyed.

Using a similar analysis of the same industries that produce the major quantities of scrap, it is noted that three of the industries generating the major quantity of titanium scrap (Machine Shop/Fabricators, Airframe and Engine) produce mostly turnings, while the fourth industry (Forger) produces mostly solid scrap. Upon consideration of the product line of each industry, this result would be anticipated.

TABLE 63
QUANTITIES OF TITANIUM ALLOY OPEN MARKET SCRAP VS INDUSTRY CATEGORY

Type of Industry	*Number Surveyed*	*Survey Responses*	*Scrap (Ti) Quantity Reported (lb × 1000)*	*Total Industry Scrap Not Reported* % (Est)*	*Total of Open Market Industry Scrap (lb × 1000)*
Airframe	10	8	2644	10	2937
Engine	6	4	1909	33	2870
Aerospace Equipment	3	3	182	65	520
Forgers	16	11	1831	39	3031
Machine/ Fabricators	3	3	1788	54	3920
Totals	38	29	8354		13278

*Includes scrap generated by companies not responding to the survey or not surveyed.

TABLE 64
QUANTITIES OF NICKEL ALLOY OPEN MARKET SCRAP VS INDUSTRY CATEGORY

Type of Industry	*Number Surveyed*	*Survey Responses*	*Scrap (Ti) Quantity Reported (lb × 1000)*	*Total Industry Scrap Not Reported* % (Est)*	*Total of Open Market Industry Scrap (lb × 1000)*
Airframe	10	8	91	10	101
Engine	6	4	4270	33	6348
Aerospace Equipment	3	3	183	65	521
Forgers	16	11	3371	23	4370**
Machine/ Fabricators	3	3	714	85	4834
Totals	38	29	8628	—	16174

* Includes scrap generated by companies not responding to the survey or not surveyed.

** A substantial quantity of nickel-base alloy scrap internally recycled by aerospace forgers (presumably to captive melt shops) is not included in these figures.

TABLE 65
AIRFRAME INDUSTRY TITANIUM ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids (lb)*	*Turns (lb)*	*Total (lb)*
6/4	78756	778670	857426
C.P.	30416	45456	75872
5-2.5	0	0	0
6-2-4-2	0	0	0
8-1-1	0	0	0
6-6-2	67274	928959	996233
Other	4381	3247	7628
Mixed/Contaminated	299854	407802	707656
Totals/% of Total	480681/18%	2164132/82%	2644815*

*Contains 27% unsegregated/mix.

TABLE 66
ENGINE INDUSTRY TITANIUM ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids* (lb)*	*Turns* (lb)*	*Total* (lb)*
6/4	74358	859094	933452
C.P.	11434	47718	59152
5-2.5	45145	940884	986029
6-2-4-2	0	0	0
8-1-1	3121	5399	8520
6-6-2	0	0	0
Other	0	0	0
Mixed/Contaminated	162190	494109	656299
Totals/% of Total	296248/11%	2347204/89%	2643452**

*Includes estimate of scrap generated by nonrespondents
**Contains 25% unsegregated/mix.

TABLE 67
AEROSPACE INDUSTRY TITANIUM ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids (lb)*	*Turns (lb)*	*Total (lb)*
6/4	3,267	0	3,267
C.P.	1,058	0	1,058
5-2.5	0	0	0
6-2-4-2	0	0	0
8-1-1	0	0	0
6-6-2	0	0	0
Other	0	0	0
Mixed/Contaminated	40,162	137,939	178,101
Totals/% of Total	44,487/24%	137,939/76%	183,426*

*Total contains 98% unsegregated/mix.

TABLE 68
FORGING INDUSTRY TITANIUM ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids (lb)*	*Turns (lb)*	*Total (lb)*
6/4	780,750	342,190	1,122,940
C.P.	16,932	13,348	30,280
5-2.5	68,905	113,174	182,079
6-2-4-2	73,825	34,110	107,935
8-1-1	18,653	1,400	20,053
6-6-2	102,570	0	102,570
Other	6,300	0	6,300
Mixed/Contaminated	62,500	197,120	259,620
Totals/% of Total	*1,130,435/62%*	*701,342/38%*	*1,831,777**

*Contains 14% unsegregated/mix.

TABLE 69
MACHINE SHOP/FABRICATION INDUSTRY TITANIUM ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids (lb)*	*Turns (lb)*	*Total (lb)*
6/4	479,732	407,813	887,545
C.P.	76,713	1,318	78,031
5-2.5	3,305	44,006	47,311
6-2-4-2	561	3,718	4,279
8-1-1	7,503	31,974	39,477
6-6-2	18,145	0	18,145
Other	0	0	0
Mixed/Contaminated	46,066	668,035	714,101
Totals/% of Total	632,025/35%	1,156,864/65%	1,788,889*

*Contains 40% unsegregated/mix.

TABLE 70
AIRFRAME INDUSTRY NICKEL ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids (lb)*	*Turns (lb)*	*Total (lb)*
Inconel and Inconel X	740	0	740
Inco 901	0	0	0
A-286	25	0	25
Hastelloy X	21	0	21
Inco 718	0	0	0
Rene 41	0	0	0
Waspaloy	0	0	0
IN-100	0	0	0
Udimet-700	0	0	0
B-1900	0	0	0
Inco 713	0	0	0
Rene 80	0	0	0
Inco 738	0	0	0
Other	43,552	6,999	50,551
Mixed/Contaminated	10,350	30,009	40,360
Totals/% of Total	54,693/60%	36,999/40%	91,697*

*Contains 44% unsegregated/mix.

TABLE 71
ENGINE INDUSTRY NICKEL ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids* (lb)*	*Turns* (lb)*	*Total* (lb)*
Inconel and Inconel X	88,260	237,880	326,140
Inco 901	47,841	1,118,413	1,166,254
A-286	87,395	504,636	592,031
Hastelloy X	673,492	107,680	781,172
Inco 718	90,795	547,918	638,713
Rene 41	0	0	0
Waspaloy	41,504	395,842	437,346
IN-100	0	0	0
Udimet-700	0	0	0
B-1900	14,145	0	14,145
Inco 713	19,732	0	19,732
Rene 80	0	0	0
Inco 738	0	0	0
Other	126,972	27,552	154,524
Mixed/Contaminated	817,552	1,032,264	1,849,816
Totals/% of Total	2,007,688/34%	3,972,185/66%	5,979,873**

*Includes estimate of scrap generated by nonrespondents
**Contains 31% unsegregated/mix.

TABLE 72
AEROSPACE EQUIPMENT INDUSTRY NICKEL ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids (lb)*	*Turns (lb)*	*Total (lb)*
Inconel and Inconel X	33,866	0	33,866
Inco 901	0	0	0
A-286	383	0	383
Hastelloy X	2,026	2,026	2,026
Inco 718	3,094	1,800	4,894
Rene 41	0	0	0
Waspaloy	60,373	0	60,373
IN-100	0	0	0
Udimet-700	0	0	0
B-1900	0	0	0
Inco 713	0	0	0
Rene 80	0	0	0
Inco 738	0	0	0
Other	0	14310	14,310
Mixed/Contaminated	67,297	0	67,297
Totals/% of Total	167,039/91%	16,110/9%	183,149*

*Contains 37% unsegregated/mix.

TABLE 73
FORGING INDUSTRY NICKEL ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids (lb)*	*Turns (lb)*	*Total (lb)*
Inconel and Inconel X	248,379	481,642	730,021
Inco 901	148,682	345,048	496,750
A-286	138,509	247,328	385,831
Hastelloy X	32,756	151,557	184,413
Inco 718	396,514	361,652	758,166
Rene 41	17,796	3,519	21,315
Waspaloy	158,764	233,468	392,232
IN-100	0	1,430	1,430
Udimet-700	3,530	3,800	7,330
B-1900	0	0	0
Inco 713	0	0	0
Rene 80	0	0	0
Inco 738	0	0	0
Other	0	0	0
Mixed/Contaminated	39,761	353,344	393,605
Totals/% of Total	1,184,691/35%	2,186,388/65%	3,371,079*

*Contains 12% unsegregated/mix.

TABLE 74
MACHINE SHOP/FABRICATION INDUSTRY NICKEL ALLOY SCRAP QUANTITY AND QUALITY

Alloy Spec.	*Solids (lb)*	*Turns (lb)*	*Total (lb)*
Inconel and Inconel X	1,705	20,598	22,303
Inco 901	6,143	75,989	82,132
A-286	23,087	26,362	49,449
Hastelloy X	46,016	34,873	80,839
Inco 718	130,347	7,032	137,379
Rene 41	700	0	700
Waspaloy	7,274	100,746	108,020
IN-100	164	0	164
Udimet-700	0	0	0
B-1900	0	0	0
Inco 713	0	0	0
Rene 80	0	0	0
Inco 738	0	0	0
Other	58,706	106,309	165,015
Mixed/Contaminated	18,370	49,158	68,528
Totals/% of Total	293,512/42%	421,067/58%	714,579*

*Contains 10% unsegregated/mix.

b. *Melters, Casters, and Users/Ti and Ni Open Market Scrap Data (Summary II)*

Summary II information is presented in Table 75. These survey data are supplemental results of a limited amount of scrap generated by the Melters, Casters and Final Product Users that is disposed of in the open market. Final Product Users are defined here as airlines, military and independent overhaul centers. This scrap (Table 75) is usually of low grade and not conducive to aerospace reclaiming. Melters tend to recycle all scrap possible in their internal closed loop, with contamination and/or customer requirements being their principal limitations. Therefore, scrap being disposed of by Melters is overly contaminated and useless for aerospace reclamation. Likewise, scrap being disposed of by the aforementioned Final Products Users is often from coated, welded and contaminated engine and airframe components. This scrap also is of poor quality for aerospace reclamation.

TABLE 75
SUPPLEMENTAL SURVEY QUANTITIES OF OPEN MARKET SCRAP FROM CASTING, MELTING AND FINAL PRODUCT USER INDUSTRIES

Type of Industry	*Number Surveyed*	*Survey Response*	*Alloy Type*	*Quantity Reported (lb × 1000)*
Melters	8	5	Ti	2429
	13	9	Ni	833
Casters	9	7	Ti	106
	9	7	Ni	76
Final Product Users[a]	18	9	Ti	70
	18	9	Ni	268

[a]Includes airlines, military, and independent overhaul centers.

c. *Ti and Ni Grindings and Sludges Data (Summary III)*

Summary III information is presented in Table 76. These data are results of a supplemental survey conducted with 44 companies to assess their scrap generation activities in the field of grindings and sludges. It was shown clearly by some of the 19 respondees that minor resale value is obtained from Ni grindings and EDM sludges; no resale value is associated with Ti grindings or ECM sludge products from Ni and Ti. No information on the relative metal content of this type scrap was received; therefore, an accurate analysis of the survey to estimate metallic quantity condition was not possible.

Ten of the nineteen respondees reported 10,207,000 lb and 207 gal. of sludges and grindings; the remaining nine respondees reported none or unknown quantities. These data must be viewed cautiously since 89% of the total reported was from one source. In addition, a number of respondents implied that careful records, as maintained for scrap metals, are not kept since wastes and sludges are regarded as "rubbish."

This lack of survey information must be given consideration in recognizing the survey incompleteness of Table 76. Based upon discussions with survey sources and others, it can be expected that the Ni and ECM sludge (Ti, Ni estimates) is several orders of magnitude low.

4. Potential Improvement of Aerospace Scrap Recycling

During the course of this survey, observations were made and constructive criticism and suggestions were offered to facilitate additional recycling of aerospace scrap. Many of these areas are semicriticisms of present-day controls applied by manufacturers and users regarding the reuse of scrap for aerospace applications.

TABLE 76
QUANTITIES OF TITANIUM AND NICKEL BASE ALLOY GRINDINGS AND SLUDGES SCRAP

Industry Code Machine/ Fabricators		*Per Year*	*Disposition*
A	Nickel Containing Grindings	24,000	Hauled away - No recompense
B	Nickel Containing Grindings	1,800	"Vendor Recycle"(?)
Forgers			
A	—	None	—
B	Nickel Containing Grindings	25,000	Hauled away - No recompense
	Nickel Containing Grindings	Data not available	—
	Titanium Containing Grindings	18,000	Hauled away - No recompense
	Titanium Containing Sludges	Data not available	—
C	—	None	—
D	—	None	—
E	—	None which can be sold	—
F	—	None	—
G	Nickel Containing Grindings	25,000	"Disposed"
	Nickel Containing Sludges	17,000	"Disposed"
	Titanium Containing Grindings	3,000	"Disposed"
	Titanium Containing Sludges	0	"Disposed"
H	Nickel Containing Grindings	185,000	"Sell"
Engine			
A	"Sludges, Grindings"	Unknown	Not Sold
B	Nickel Hydroxide, ECM	330,000	Open Market
	Titanium Oxide, ECM	9,100,000	Land Fill
C	Nickel Containing Grindings, Wet	260,000	"Disposed"
	Nickel Containing Sludge, ECM	Unknown	"Disposed"
	Titanium Containing Grindings	Small Amount	"Disposed"
Airframe			
A	Nickel Containing Grindings, 0.3% Ni	53,000	Disposal company
B	Titanium Base Grindings	5,000	Burned by vendor
	Titanium Base Sludges	207,500	"Disposed"
C	Grindings and Sludges	Unknown	"Disposed without Comp."
D	Grindings and Sludges	None	
E	Titanium Base Grindings (Wet)	Small Amount	"Discarded"
F	Grindings and Sludges	None	
Aerospace Equipment			
A	Grindings and Sludges	None	

It is suggested that an objective assessment be made of present controls and procedures in view of the cost-effective potential of reclamation. It is recognized that prudence is justified and confidence and acceptance of unproven procedures is attained with repetitive success. However, the following potential areas are of a sensible, progressive nature with minimal degrees of user offensiveness; consideration should be given to these areas for improved aerospace scrap recycling:

- *Segregated Titanium Turnings* — Any step which eliminates the presence of dense inclusions will tend to increase the use of titanium turnings. The increased use of nonconsumable and EB furnaces should also increase aerospace scrap titanium turnings recycling.

- *Cast Superalloys* — A major portion of scrap from cast nickel alloys, often blades and vanes, is not recycled for aerospace scrap use. Efforts to find ways to include these valuable alloys in the aerospace recycling program should be fostered.

- *All Aerospace Metals* — Efforts should be made to increase the allowable scrap used in both rotating and nonrotating parts. Major improvements in processor's abilities to provide guaranteed chemistry should be considered when considering liberalized specifications.

- *All Aerospace Scrap Metals* — Efforts can and should be made to increase the quality of aerospace scrap metals generated at aerospace manufacturing plants. Methods for accomplishing this are spelled out in the Model System program, another facet of this Strategic Material Reclamation Program.

- *All Aerospace Scrap Metals* — Consideration should be given, where applicable, to relax restrictions that require alloys to be produced only from the same alloy composition. In addition, all critical material specifications should include the maximum allowable tramp element content, so that melters may know the limits for those major alloying elements not included in a particular grade. Both of these suggestions were supported at the Strategic Material Reclamation Seminar, May 1974.

B. SCRAP CONTROLS REVIEW

1. Background

P&WA thoroughly reviewed in-house scrap utilization controls and thereupon conducted informal surveys with representative airframe and engine fabricators and raw material suppliers to establish general industrial controls. The sensitive and frequently proprietary nature of these controls precludes specific detailed accounting and source identification. However, analyses of these data did enable definition of the controls presently in industrial vogue for the utilization of scrap.

The review confirmed that the controls applied by raw material suppliers by themselves, and/or controls imposed by users on these raw material suppliers, represent a significant restraint to the increased utilization of scrap although a trend towards control reevaluation is evident.

As might be expected, the raw material supplier, recognizing his financial investment in attaining an acceptable product, exercises initial judgment on utilization of various types and grades of scrap. This judgment reflects the cost effectiveness of scrap usage vs the inherent risk of a rejected heat due to material chemical and mechanical property requirements.

A broad spectrum of additional scrap controls may then be applied by the user which ranges from severely restrictive to mildly controlled. It was clearly obvious that the maximum levels of applied controls are the direct result of the user's concern with critically stressed components. As the criticality of the component application diminishes, so does the degree of control applied in scrap utilization.

2. Concept of Maximum Control

Maximum control implies that the user is genuinely concerned with accurately establishing the "lineage" of the raw material to assure supplier repeatability of an acceptable raw material, and reduce suspect areas if unforeseen problems or trends develop. Raw material "lineage" is established and controlled by various combinations of material specifications, source approvals and user/supplier agreements.

a. Raw Material Specifications

Aerospace Material Specifications (AMS) are used to specify raw material form, composition, condition (finish and heat treatment), mechanical properties, structure, quality assurance (sampling, testing and identification procedures), reporting and rejection criteria, etc. These specifications are often supplemented with similar User Material Specifications when the user specifies additional requirements to the AMS specification.

b. Source Approvals and/or User/Supplier Agreements

When a user is genuinely concerned with establishing, approving and solidifying a raw material process, i.e., deviations require user approval, the application of source approval and/or user/supplier agreements is generally followed. These actions can limit the type and quantity of scrap usage that is applicable to specific melters. The scrap source (dealer) may be designated via source approval based on approved procedures for processing, identification and quality control.

3. Concept of Reduced Control

Reduced control implies that the user is primarily concerned with obtaining raw material that consistently attains all material specifications as given in the AMS and/or User specification requirements. Control is not normally applied on the melter's operations or the utilization of scrap by the melter. In this area of reduced control, the melter assumes full responsibility for the choice and usage of scrap within the guidelines of producing an acceptable, saleable product.

4. Controls Applied to Titanium and Nickel Alloy Scrap

Survey results of controls applied to titanium and nickel alloy scrap, using a reasonable interpretation of control from maximum scope to reduced (minimal) scope, and as a function of scrap form, are summarized in Tables 77 and 78, respectively.

TABLE 77
CONTROLS APPLIED TO TITANIUM ALLOY SCRAP

Scrap Form	*Applied Control*	*Applied Maximum Control (+ Specification)*	*Applied Reduced (Minimal) Control + Specification*
Solids	Source	In house scrap admissible.	
		Purchased scrap admissible upon dealer source approval.	
		Source approval includes identification, controls and processing.	
		Identification by approved spot checking is adequate.	
	Melter	Aborted heats unacceptable.	Aborted heats unacceptable.
		Sheet clippings and cobbles unacceptable.	-
		Flame cuttings unacceptable unless hydride processed.	Flame cuttings unacceptable.
		Melter is source approved.	Melter is source approved.
Turnings	Source	In-house scrap admissible if magnetic tool bits were used.	
		Purchased scrap admissible upon dealer source approval.	
		Source approval includes identification, controls and processing.	
	Melter	Percent scrap addition *self-limiting due to* O_2 spec and electrode green strength	
		Melter is source approved.	Melter is source approved
Castings	Caster	In-house mold-contaminated castings unacceptable.	Mold-contaminated castings unacceptable
		In-house aborted casting heats unacceptable.	Aborted casting heats unacceptable
		Purchased castings not presently under consideration.	
		Scrap additions in final melt unacceptable.	
		Caster is source approved.	Caster is source approved.
Dust	Source	Dust and grindings unacceptable.	Dust and grindings unacceptable.

TABLE 78
CONTROLS APPLIED TO NICKEL ALLOY SCRAP

Scrap Form	*Applied Control*	*Applied Maximum Control (+ Specification)*	*Applied Reduced (Minimal) Control + Specification*
Solids	Source	In-house scrap admissible.	
		Purchased scrap admissible upon dealer source approval.	
		Identification by approved spot checking is adequate.	
	Melter	Liberal allowances granted for purchased scrap.	
		Percent scrap addition is established and approved.	
		Melter is source approved.	Melter is source approved
Turnings	Source	In-house scrap admissible.	
		Purchased scrap admissible upon dealer source approval.	
	Melter	Melter is source approved	Melter is source approved.
Castings	Source	In-house castings admissible	
		Purchased castings admissible upon dealer source approval.	
	Caster	Revert must be same composition (Some exceptions)	
		Revert of gates, risers, sprue cups admissible.	
		Revert of hot tops (i.e. flash powder) unacceptable.	Revert of hot tops (i.e. flash powder) unacceptable.
		Percent revert addition is established and approved.	
		Revert addition in final melt unacceptable	
		Caster is source approved.	Caster is source approved

5. Summary

In general, controls applied to scrap usage for nickel alloys are considerably less severe than those applied to titanium alloys. Ni alloys are more tolerant of common contaminant sources such as frictional or heat treat scaling (i.e., — interstitial contaminants) and fractured tool bits compared to Ti alloys. Also, melt pool sampling with adjustment of Ni alloy melting composition may be made during the initial induction melting step to accommodate minor compositional problems due to scrap additions. This adjustment is not feasible during the initial arc or EB melting associated with Ti alloys.

A very considerable number of raw material user rely upon the experience and procedures of the principal users with regards to their application of maximum controls upon raw material suppliers. These users express the general opinion that reasonable procedures and controls have been established and are in daily usage. Since these users often are not staffed adequately to maintain constant vigilance regarding raw material controls, it is advantageous for them to utilize existing industrial procedures. This enables them to include specific controls for their own usage while maintaining minimal surveillance. This "pseudo-universal" feature of several users adopting the basic maximum raw materials controls of the principal users can be beneficial to the suppliers since standardization of procedures is always attractive.

PHASE II

SECTION IX

RAW MATERIAL CHEMISTRY

A. ANALYSIS OF TITANIUM CHIPS

Analyses of raw material Ti alloy chips utilized in this program were performed by participating subcontractors and by P&WA. Subcontractor analyses were made after representative sampling; P&WA analyses were made after chips were consolidated by nonconsumable arc melting into cast buttons.

Table 30 summarizes data generated on raw material Ti alloy chips of medium, high and low density grades. Consistent analytic agreement was attained between individual subcontractors and P&WA. As indicated (Table 30), oxygen level is consistently about 50% above the specification level of 0.2% maximum, regardless of chip density level. This oxygen contamination is reasonable: (1) Ti billets are usually heat-treated in air and the external contaminated layer is removed by machining; (2) chips may be inadvertently contaminated by excessive frictional heating during preliminary machining operations. Table 30 also shows that the Fe level was often 25 to 100% above the maximum specification level of 0.3%. Minor increases in Ni and Cr levels were also detected. However, previous study indicated that 0.4% contaminant was removed from raw chips by magnetic separation. This implies that the Fe content in the titanium could be attributed to contamination by nonmagnetic austenitic stainless steel (AISI 300 series) or an age-hardenable iron base alloy, e.g., A-286 Tinidur. The only discrepancy in Table 30 data appears to be one analysis of 0.66% Sn for the medium density material. This analysis was later repeated in duplicate by P&WA with <0.01% Sn detected.

B. ANALYSIS OF WASPALOY CHIPS

Table 79 compares data generated on raw material Waspaloy chips. Bi content was determined to exceed specification maximum along with minor presence of Pb and Sn in one analysis; this result was not substantiated in a re-analysis at P&WA. Possibly a trace of Cerrobend (Pb-Sn-Bi) potting alloy was present to influence the Bi level. No other evidence of deviation from Waspaloy specification chemistry was detected in the raw material chips analyzed.

TABLE 79
ANALYSES OF WASPALOY CHIP RAW MATERIALS

		Raw Material Chips	
	PWA 1007 Spec.	*Frankel*	*PWA*
Cr	18 to 21%	19.35	18.9
Co	12 to 15	13.7	13.0
Mo	3.5 to 5.0	3.95	3.9
Ti	2.75 to 3.25	3.00	2.8
Al	1.2 to 1.6	1.25	1.2
Zr	0.02 to 0.12	< 0.07	0.06
B	0.003 to 0.010	0.004	0.005
C	0.02 to 0.10	0.068	0.04
Mn	0.75 max	0.06	0.006
Si	0.75 max	< 0.05	0.13
S	0.020 max	0.003	0.007
Fe	2.00 max	1.07	0.64
Cu	0.10 max	< 0.05	< 0.05
Bi	0.00005 max	0.00055	< 0.00003
Pb	0.0010 max	< 0.0001	< 0.0001
Se	—	—	< 0.0001
Te	—	—	< 0.00002
Tl	—	—	< 0.0001
P	—	—	—
Ta	—	< 0.05	—
W	—	< 0.1	—
V	—	< 0.1	—
Sn	—	< 0.05	—
Cb	—	< 0.1	—
O	—	—	—
N	—	—	—
Ni	R*	R	R

R* = Remainder

SECTION X

RECLAMATION TECHNOLOGY

A. AEROSPACE INDUSTRY REQUIREMENTS FOR RECLAMATION OF TITANIUM AND SUPERALLOY SCRAP RECLAMATION

1. Analysis of Aerospace Scrap Survey

An analysis of the Aerospace Scrap Survey was deemed necessary to define aerospace industry requirements for separation of titanium (Ti) and nickel (Ni) superalloy scrap. The surveyed quantities of open-market scrap disposed to scrap dealers/processors showed slightly more Ni scrap was generated as compared to Ti scrap during the 1973 survey year. Specifically, about 16.1×10^6 total lb of Ni alloy vs 13.3×10^6 total lb of Ti scrap was determined.

An analysis of the industrial controls applied to usage of Ti and Ni scrap was then made. It was determined that controls applied to scrap usage for Ni alloys are considerably less severe than those applied to Ti alloys. Superalloys are more tolerant of common contaminant sources such as fractured WC tool bits and frictional or heat treat scaling, i.e., interstitial contaminants, compared to Ti alloys. Also, melt pool sampling with adjustment of superalloy melting composition may be made during the initial induction melting step to accommodate minor compositional problems due to scrap additions. This adjustment is not feasible during the primary melting (arc or EB) associated with Ti alloys.

This implication of increased aerospace consumption of open-market Ni scrap, as compared to Ti scrap, was reinforced by industrial contacts. A high level of open-market Ni scrap is recycled due to less-stringent quality requirements. Unfortunately, the multiple industrial applications for superalloys precluded accurate determination of recycle percentage, but it was estimated that as much as 75% of the high nickel, aerospace grade open-market scrap is recycled back to aerospace ingot via scrap dealer/processors. As a result, it became increasingly obvious that the aerospace industry has a predominant need for a cost-effective, reliable separation of Ti scrap, i.e., machine turnings and chips, as compared to their need for Ni scrap separation.

2. Scrap Material Selection for Phase II

On the basis of the above analyses of the aerospace scrap survey, the Phase II program effort was redirected to separation of Ti scrap turnings. Separation work on Ni scrap turnings was terminated; however, a need does exist for recovery of Ni grindings and sludges which presently attain little or no attention. The recovery of this material was addressed in Phases II and III using a two-step process, i.e., a chemical (molten salt bath) plus pyrometallurgical (melting) procedure.

B. TITANIUM DENSITY SEPARATION PROCESS (FRANKEL FLUIDIZED BED) PHASE II

1. Background Information

The Frankel Co. fluidized bed density separation process was selected by P&WA for further development under Phase II on the basis that the process was being used in commercial production with reasonably established operating parameters and operating costs. P&WA directed Frankel to process all Phase II titanium turnings in accordance with optimized process parameters developed during Phase I, and further, to maintain complete material balance records of pertinent process operations.

The Frankel density separation process consists of eleven sequential operations: (1) wet-ring crushing of the turnings into chips, (2) chip sampling for chemical analyses, (3) chip cleaning in an alkali solution, (4) water-spray rinsing, (5) drying of chips in a centrifuge, (6) hot air drying, (7) screen separation of fine particles, (8) magnetic separation, (9) fluidized bed density separation, (10) chemical analyses of product samples and (11) packaging of the final product.

2. Frankel Fluidized Bed Process Operations (Phase II)

a. Preparation of Raw Material

Approximately 10,000 lb of titanium (Ti-6Al-4V) scrap chips and turnings were procured from Ladish Co., a Cudahy, Wisconsin forging company with extensive machine shop operations, and delivered to the Frankel Co. in Detroit, Michigan. The as-received weight breakdown of the raw material is noted in Table 80a.

In the initial operation, Frankel wet-crushed the titanium turning into chips of approximately 0.5-in. length. The operation was accomplished in a ring crusher fitted with inlet feed and output conveyors, Figure 100. The crusher processing rate amounted to approximately 1500 lb/hr. The total crushed chip quantity weighed 11,117 lb (Table 80b).

TABLE 80
MATERIAL BALANCE OF PHASE II TITANIUM PROCESSING

As-Received Raw Material				
Ti Turnings Received	10,043 lb			
Retained Oil + Moisture	214 lb			
Dry Turnings Received (Calculated)	9,829 lb			
Wet Ring Crushing				
Ti Chips After Wet Crushing	11,117 lb			
Division Into Two Lots	Lot A (Unseeded) Wt.(lb)	%	Lot B (Seeded) Wt.(lb)	%
Ti Chips After Wet Crushing	5,412		5,705	
Addition of Contaminants				
Seeded Contaminants Added (Dry)	0		51	
Total Weight to be Processed	5,412	100.0	5,756	100.0
Cleaning and Drying				
Oil + Moisture Removed	628	11.6	662	11.5
Dry Chips (Calculated)	4,784	88.4	5,096	88.5
	5,412	100.0	5,756	100.0
Screening + Magnetic Separation				
10 Mesh Fines Removed	517	10.8	539	10.6
Magnetic Rejects Removed	28	0.6	74	1.4
Clean, Demagnetized Chips	4,102	85.7	4,321	84.8
Unaccounted Matl.*(Calculated)	137	2.9	162	3.2
	4,784	100.0	5,096	100.0
Fluidized Bed Separation				
Total Fluidized Bed Rejects	245	6.0	295	6.8
Total Processed, Acceptable Chips	3,823	93.2	3,984	92.2
Unaccounted Matl.**(Calculated)	34	0.8	42	1.0
	4,102	100.0	4,321	100.0

* Includes production loss portion of the "nonmetallics" (paper, wood, etc.) that was removed in cleaning process and possible slight discrepancy between actual and calculated weight of chips.
** Includes "dust" blown out of chips which is mostly nonmetallic.

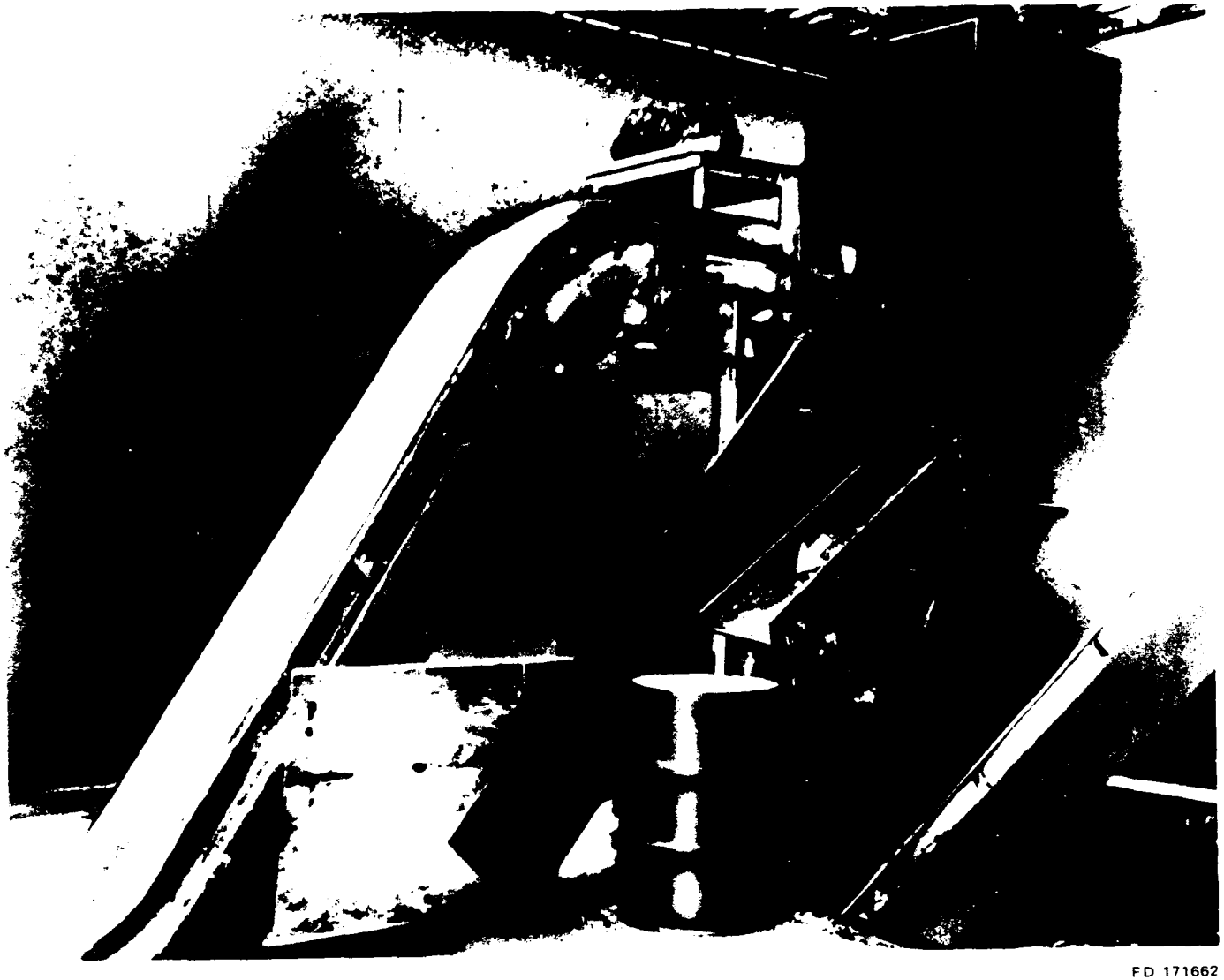


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Figure 100. Wet Ring Crusher (Converts Scrap Turnings to Chips)

b. *Chip Lot Contamination*

In order to dispel any concern that the purchased turnings may be of "pedigreed stock" and thereby not contain representative machine-shop contaminants, the crushed chips were divided into two nearly-equivalent weight quantities (Table 80c), designated as Lots A (unseeded) and B (seeded). Both lots were subjected to separate treatment in all subsequent process operations.

The "seed" added to the 5705-lb quantity of Lot B titanium chips consisted of: (1) high density contaminants — crushed tungsten carbide (WC) tool bit fragments; (2) nonmagnetic contaminant chips — A-286 Modified Tinidur; and (3) magnetic contaminant chips — steel and grade 400 stainless steel.

Figure 101 shows the total quantity of high density contaminant added to Lot B, and Figure 102 illustrates a representative combination of the magnetic and nonmagnetic contaminants added to Lot B. Table 81 summarizes the seeded contaminants added to Lot B.

TABLE 81
SUMMARY OF SEEDED CONTAMINANTS ADDED TO "LOT B"

	High Density Inclusions (Crushed WC Tool Bits)	*Nonmagnetic Chips (A-286 Mod. Tinidur)*	*Magnetic Chips (Grade 400 S/S)*
Total Weight	533 gm	25 lb	25 lb
Particle Range			
Large (+ ½ in.)	287 gm (45 pcs)		
Medium (− ½ in. + 10 Mesh)	240 gm (300 pcs)		
Small (−10 Mesh + 30 Mesh)	6 gm (150 pcs)		
	533 gm (Total)		

c. *Chip Processing*

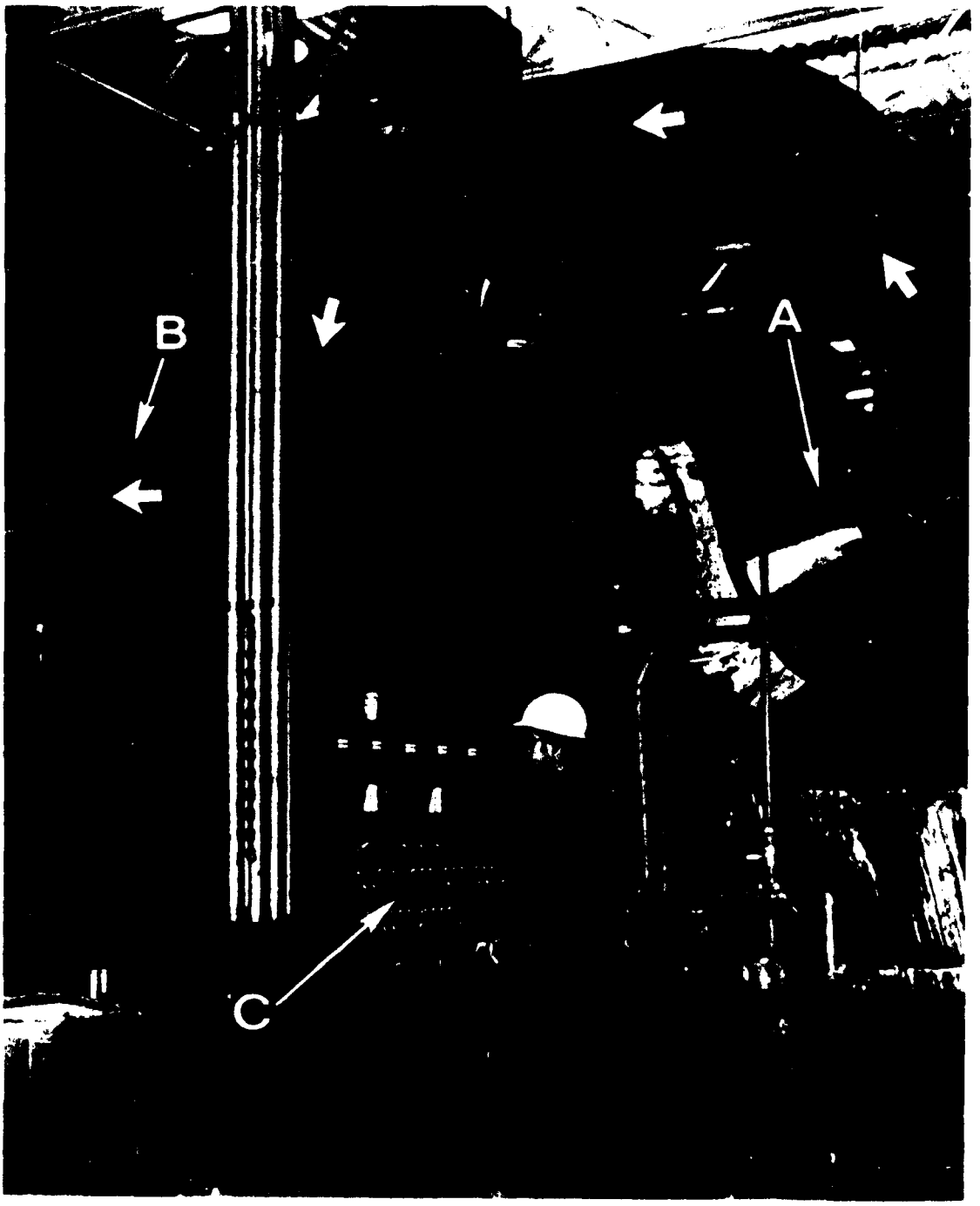
Chip cleaning and drying operations were accomplished by Frankel with the semi-automated equipment shown in Figure 103. Under operator control from a central console, the chip lots were exposed to alkali cleaning, water spray rinse and centrifuge and hot air (400°F) drying. The chip lot weight loss due to removal of oil and moisture content is tabulated in Table 80d.

Screening and magnetic separation of both chip lots were conducted in a tandem operation with the equipment shown in Figure 104. The screening process consisted basically of the removal of fine particles by passing the chip lots over a No. 10 mesh screen (0.078-in. opening). This operation is required because of potential interstitial contamination that is inherent with "fines." The chips were then transported through a cross-belt permanent magnet separator for removal of the strongly-magnetic-attracted contaminants, such as tramp iron and steel, and then through a high-intensity DC electromagnetic separator for removal of the weakly-magnetic-attracted contaminants. Material balance data for both the screening and magnetic separation operations are tabulated in Table 80e.

Fluidized bed separation was then performed on the two titanium chip Lots A and B, using equipment shown in Figure 105. The separation process, described in detail in Section VII.B.2, is shown schematically in Figure 41. Optimal fluidized bed process parameters had been chosen earlier during Phase I data analysis; these Phase I parameters are repeated here in Table 82 for informational purposes. The (approximate) 5% rejection rate for Phase II fluidized bed processing was maintained constant during all three passes with no further rejection rate adjustments made during process passes. Material balance data generated in the fluidized bed operation are tabulated in Table 80f.

Figure 101. Broken and Crushed Tungsten Carbide Tool Bits Used to Contaminate Phase II Titanium Chip Lot B

Figure 102 Magnetic and Nonmagnetic Contaminants Added to Phase II Titanium Chip Lot B Upper View — Steel and Grade 400 Stainless Steel (Magnetic) Lower View — A-286 Modified Tinidur (Nonmagnetic)



FD 171665

Figure 103. Automated Ti Chip Cleaning and Drying Station: (A) Rotary Alkali Cleaner and Spray Water Rinser, (B) Rotary Hot Air Dryer, and (C) Centralized Control Console

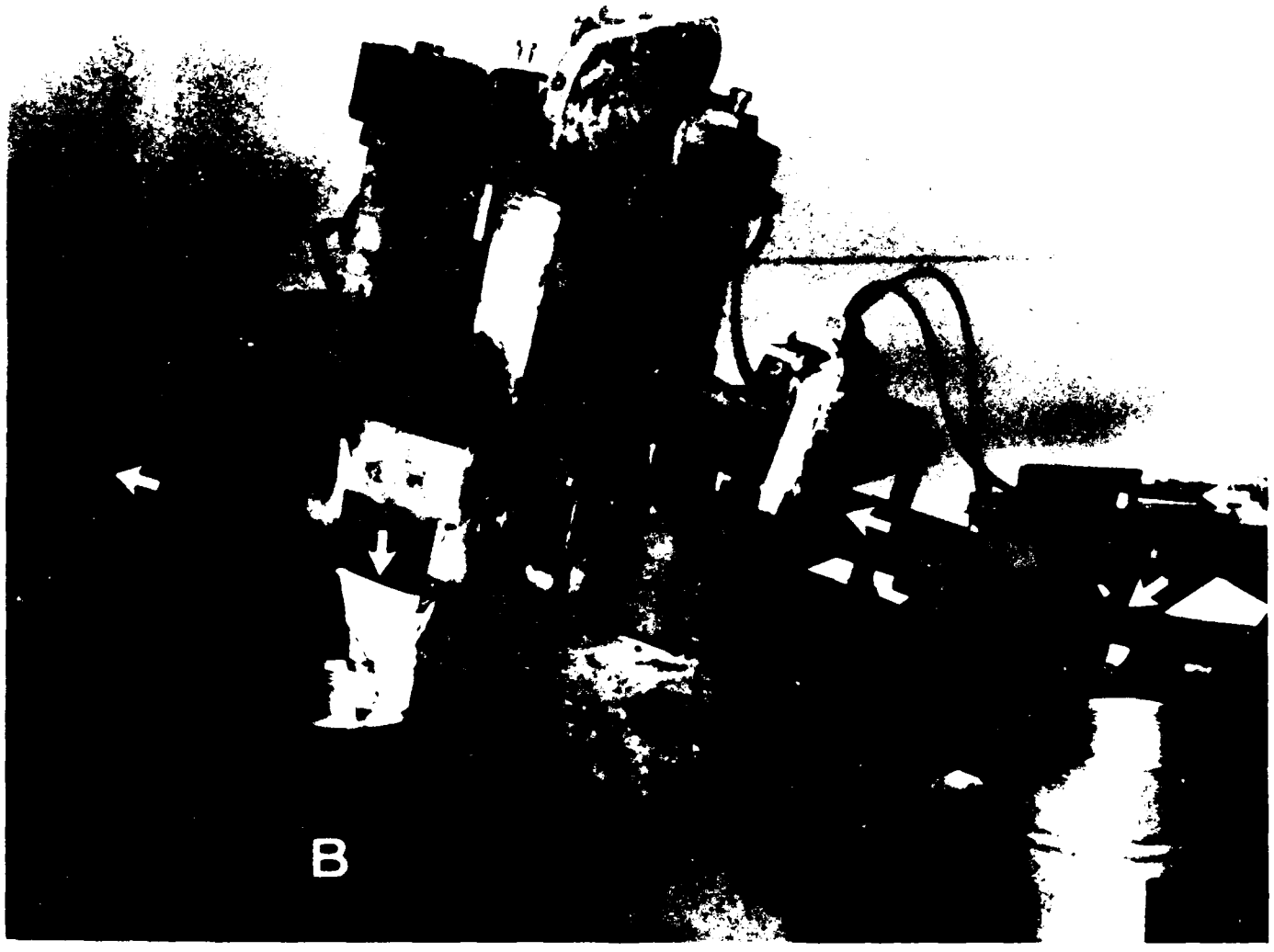
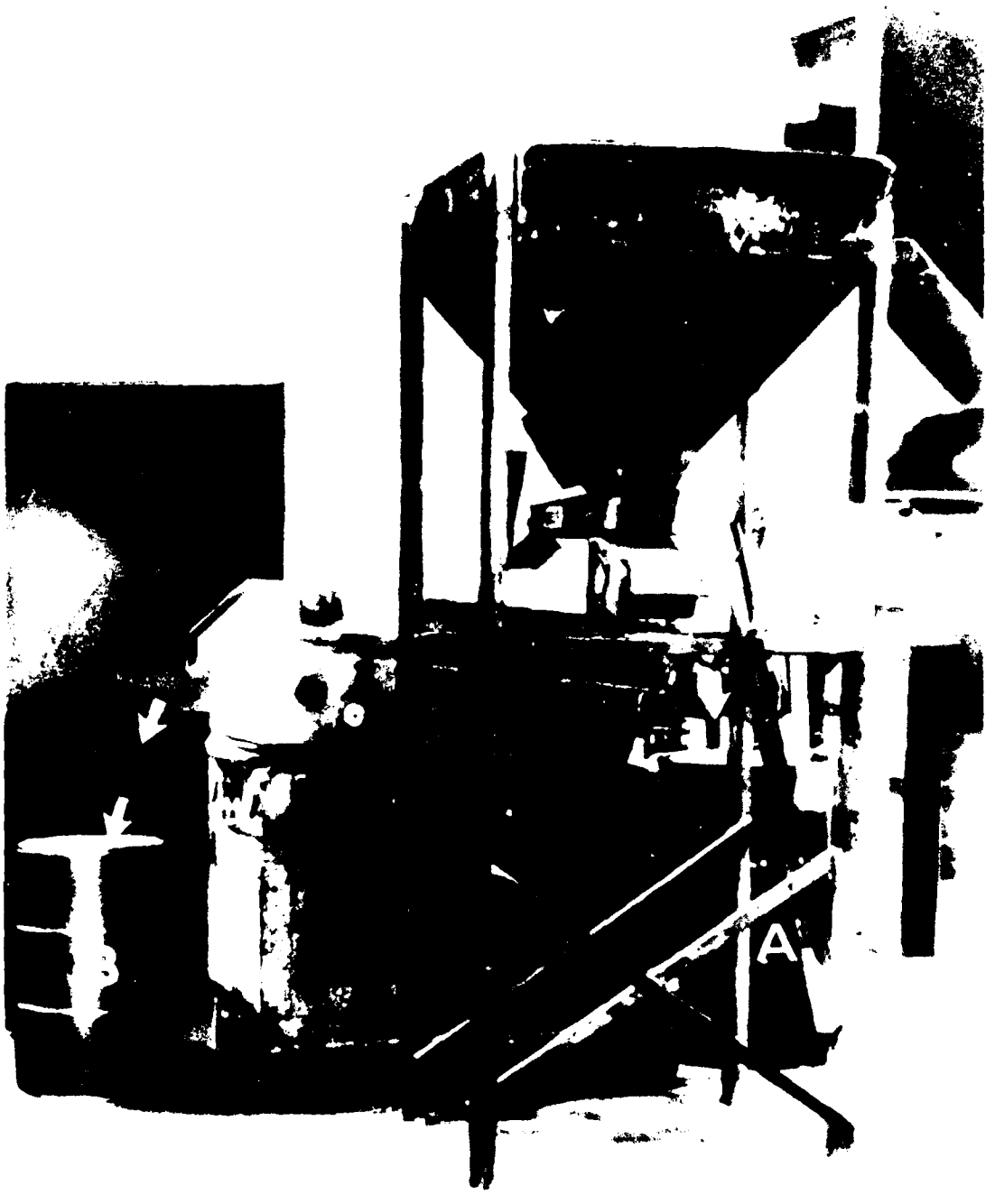


Figure 104. Screening and Magnetic Separation Station: (A) Screen Reject Material, (B) Magnet Reject Material



FD 171667

Figure 105. Fluidized Bed Density Separation Station: (A) Separated Chips, (B) Reject Material

TABLE 82
FRANKEL CO. OPTIMIZED PARAMETERS FOR FLUIDIZED BED TITANIUM DENSITY SEPARATION STATION

PHASE I PARAMETERS:

Run No.	Alloy Comp. (w/o)	Bulk Density (lb/ft³)	Chip Size Range	No. of Passes	Preset Reject Rate (%)	Feed Rate (lb/min)
2	6Al-4V	25	+10 Mesh (+0.078 in.) -¼ in.	3	5	5

PHASE I RESULTS:

Run No.	Input (lb)	Reject Quantity Pass 1 (lb)	Pass 2 (lb)	Pass 3 (lb)	Retained On Bed (lb)	Acceptable Product (lb)
2	100	5.0	1.5	1.0	5	85

3. Post-Process Analyses

a. Material-Balance Control

Table 80 contains all data relevant to the maintenance of material balances during all processes associated with the Phase II fluidized bed operation. In summary, these data show that 10,043 lb of as-received titanium turnings were processed to yield 7,807 lb (3,823 lb of Lot A, and 3,984 lb of Lot B) of titanium chips that are considered to be adequately separated for nonconsumable melting. Based on these figures, the Phase II recovery rate of 78% is a higher efficiency level would be expected with increased volume.

b. Visual Inspection of Segregated and Reject Material

Visual inspection of both segregated chip lots indicated that the material was clean with no foreign matter detected.

Visual inspection of the rejected material indicated that a considerable quantity of extraneous material, other than dense particles and heavy metal chips, had been rejected due to shape. These rejects included stones and slag particles, which, if not rejected, could be related to low-density inclusions.

c. Chemical Analysis of Segregated and Reject Material

(1) Segregated Chip Chemistry

Representative samples of segregated titanium chips from Lots A and B were subjected to chemical analysis. Results are presented in Table 83. The relatively high iron (Fe) level is associated with the Fe retained in the titanium alloy turnings, and is not free Fe from stainless or A-286 Tinidur contaminants. A comparison of the overall chip product chemistry with that of the AMS 4928 specification chemistry indicates that acceptable chemistry will be maintained with subsequent melting operations.

(2) Rejected Chip Chemistry

Samples of material rejected during each of the Lots A and B separation operations (screening, magnetic, and each fluidized bed run) were subjected to chemical analysis to determine the relative effectiveness of each separation. Relevant data is presented in Table 84 and discussed in the following paragraphs.

TABLE 83
CHEMICAL ANALYSES — SEGREGATED TITANIUM CHIPS

	Lot A (Unseeded) (w/o)	*Lot B (Contaminant Seeded) (w/o)*	*AMS 4928G*	
			Spec Min (w/o)	*Spec Max (w/o)*
Al	6.55	6.50	5.50	6.75
V	4.00	4.18	3.50	4.50
Fe	0.27	0.27		0.30
O				0.20
C	0.02	0.03		0.10
N				0.05
H				0.0125
Others				0.40
Oil + Moisture	< 0.01	< 0.01		
Ni (free)	< 0.05	< 0.05		
Cr (free)	< 0.05	< 0.05		
Fe (free)	< 0.05	< 0.05		
Al (free)	< 0.05	< 0.05		
Pb	< 0.05	< 0.05		
Mo	< 0.05	< 0.05		
Cb	< 0.05	< 0.05		
Zr	< 0.05	< 0.05		
Mn	< 0.05	< 0.05		
Sn	< 0.05	< 0.05		

The high (4 w/o)V content of the reject fines from the screening separation is normal and can be attributed to the Ti-6Al-4V fines included in this material. The high level of magnetic material and free Fe, Cr and Ni in the reject material of the magnetic separator from both unseeded Lot A and contaminant seeded Lot B (Table 84) is worthy of special discussion. Relatively high concentrations of these free elements, unrelated to titanium alloy composition, would be expected for the seeded Lot B that contained contaminant of steel/Fe-Cr grade 400 stainless steel/Fe-NiCr A-286 modified Tinidur. The equivalent free elemental concentrations in the unseeded Lot A is conclusive evidence that the raw material titanium turnings contained ample quantity of contaminants and that the Lot B seeding was unnecessary.

In a similar analysis, a very high concentration (7-8 w/o) of high density WC tool bits was determined in the aforementioned reject material of the unseeded and the contaminant-seeded lots of the magnetic separator. To put this into perspective, the quantity of WC high density inclusions removed in this unseeded lot was about double the quantity added intentionally in the seeded lot. Again, contaminant seeding was unnecessary. This amplifies the evidence that standard titanium scrap turnings have an inherent, unintended degree of contamination which is further justification for improvements in scrap management and handling at the scrap generator.

The chemical analysis of the fluidized bed reject materials, as a function of fluidized bed pass number, is also contained in Table 84. The relatively high level (0.3 to 1.3 w/o) of nonmetallics, such as stone and iron cinders in the reject material of the fluidized bed first pass, is of significance in avoiding low-density inclusions. Nonmetallics were not detected in analysis of the final (third) pass reject material. Also, the diminishing level of free elements (Table 84), as a function of fluidized bed pass number, confirms that such contamination is predominantly removed in the first pass, with decreasing quantities in subsequent passes.

TABLE 84
CHEMICAL ANALYSES OF PROCESS REJECT MATERIALS

		Lot A (Unseeded) (w/o)	*Lot B (Contaminant Seeded)* (w/o)
SCREENING SEPARATION			
Magnetic Material		0.5	2.1
Ni (free)		0.14	0.80
Cr (free)		0.05	0.23
Fe (free)		0.15	0.52
Al (free)		0.07	0.10
Pb		< 0.05	0.10
Mo		0.08	0.11
Cb		< 0.05	< 0.05
Zr		< 0.05	< 0.05
V		4.02	3.86
Mn		< 0.05	< 0.05
Sn		0.13	< 0.05
MAGNETIC SEPARATION			
Magnetic Material		41.2	83.2
Ni (free)		1.10	3.60
Cr (free)		2.0	5.45
Fe (free)		40.2	47.0
Al (free)		< 0.05	< 0.05
WC tool bits		7.2	8.2
FLUIDIZED BED SEPARATION			
Nonmetallics	Pass 1	1.3	0.3
	Pass 2	0.3	< 0.1
	Pass 3	< 0.1	< 0.1
Ni (free)	Pass 1	0.95	1.30
	Pass 2	0.40	0.77
	Pass 3	< 0.05	0.70
Cr (free)	Pass 1	0.55	0.95
	Pass 2	0.16	0.44
	Pass 3	0.08	0.31
Fe (free)	Pass 1	1.10	1.62
	Pass 2	0.29	0.90
	Pass 3	0.29	0.55
Al (free)	Pass 1	< 0.05	0.07
	Pass 2	< 0.05	< 0.05
	Pass 3	< 0.05	< 0.05

d. Radiographic Inspection of Process Reject Material

Radiographic inspection of representative quantities of the reject material from pertinent separation operations was performed to further define the effectiveness of these separations. Specifically, samples of reject material from each separation operation (screening, magnetic and fluidized bed) were subjected to radiographic exposure. The technique, as in Phase I, consisted of placing about a one-inch thickness of reject chip material randomly within a 14- by 17-in. tray, and radiographing on a double-film cassette. The double-film technique avoids misinterpretation of any film defect which may appear as a dense particle. Typical radiographs of reject chip material for both unseeded Lot A and contaminant-seeded Lot B are included herein as follows: (1) screening separation, Figures 106 and 107; (2) magnetic separation, Figures 108 and 109; and, (3) fluidized bed separation, Figures 110 through 115.

ED 171668

Figure 106. Radiograph Showing Typical Material Rejected by Screening Process from Phase II Titanium Chip Lot A. High-Density WC Tool Bit Fragments Visible Among Screened Fines

Scale: 1:1

FD 171669

Figure 107. Radiograph Showing Typical Material Rejected by Screening Process from Phase II Titanium Chip Lot B. High-Density (WC Tool Bit) Fragments Visible Among Screened Fines

SCALE 1:1

FD 171670

Figure 108. Ra[illegible] M[illegible] Removed by Magnetic Separation [illegible] P[illegible] Lot A. Considerable [illegible]

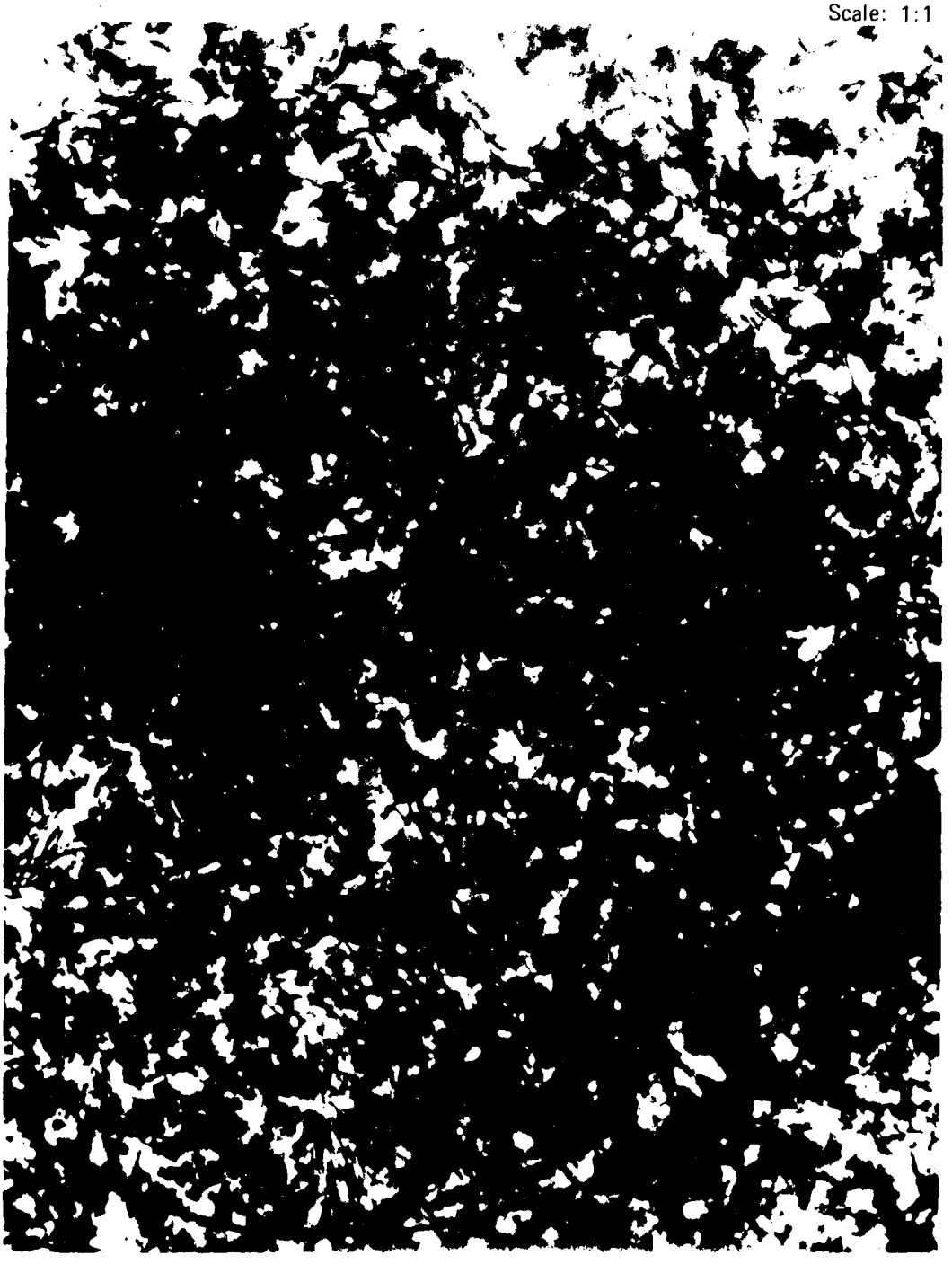
Figure 109. Radiograph Showing Typical Material Rejected by Magnetic Separation Process from Phase II Titanium Chip Lot B. Numerous High-Density Particles Detected

Scale: 1:1

ED 171672

Figure 110. Radiograph Showing Typical Material Rejected by Fluidized Bed Separation Process During Pass No. 1 of Phase II Titanium Chip Lot A. Some High Density (WC Tool Bit) Fragments Apparent

Scale: 1:1



FD 171673

Figure 111 Radiograph Showing Typical Material Rejected by Fluidized Bed Separation Process During Pass No. [illegible] Depleted Uranium Chip Lot [illegible]

Scale: 1:1

FD 171674

Figure 112. Radiograph Showing Typical Material Rejected by Fluidized Bed Separation Process During Pass No. 3 of Phase II Titanium Chip Lot A. No High-Density Particles Detected

ED 171675

Figure 113. Radiograph Showing Typical Material Rejected by Fluidized Bed Separation Process During Pass No. 2 of Phase II Titanium Chip Lot B. Numerous High-Density (WC Tool Bit) Particles Detected

Scale 1:1

FD 171676

Figure 111 Radiograph of Typical Material Rejected by Fluidized Bed Separation Process During Pass No. 2 of Phase II Titanium Chip Lot B. Note Presence of (A) High-Density (W or Ta) Wire "Hairpin," and (B) Particle With High-Density Characteristics.

Scale: 1:1

FD 171677

Figure 115. Radiograph of Typical Material Rejected by Fluidized Bed Separation Process During Pass No. 3 of Phase II Titanium Chip Lot B. One Particle (Encircled) Displays High-Density Characteristics

Analyses of these radiographs indicates that highly efficient removal of the high-density inclusions (WC tool bits) occurred in the first pass. No evidence of inclusions were detected in the second and third passes of Lot A. A small "U-shaped," dense wire was detected in the second pass of Lot B (Figure 114), and one suspicious particle as noted in both the second and the third pass of Lot B. These 1/8- to 3/16-in. length particles are of tapered cross section and therefore not of wire origin. Although unconfirmed, it is likely that these particles are titanium chips that are randomly oriented "on-end," thereby presenting a misleading denser appearance to radiographic inspection.

4. Summary of Phase II Fluidized Bed Processing Results

- Machined titanium turnings contained a high concentration of unintended high (WC and steel) and low (stones, etc.) density contaminants. The need for improvements in scrap management and handling at each generator is obvious to reduce/eliminate this contaminant situation.

- The aforementioned separation procedure, including screening, magnetic and fluidized bed separation appears very effective in removal of these contaminants.

- Almost all detectable contaminants were removed after the first pass of the fluidized bed separation as defined by representative inspection of separation reject material.

- The application of the two additional fluidized bed separation passes, followed by a nonconsumable skull melting separation, should ensure removal of these contaminants from the recovered titanium chips.

C. TITANIUM NONCONSUMABLE MELTING PROCESS (TELEDYNE-ALLVAC) PHASE II

1. Background Information

The Teledyne-ALLVAC nonconsumable, rotating electrode, arc skull-melting process was chosen for Phase II titanium melting on the basis of the established procedures applied and the acceptable data generated in an earlier AFML program. (Ref. AFML IR-160-2)

2. Phase II Development

The unseeded and the contaminant-seeded lots (nominally 5000-lb each) of titanium chips that completed the triple-pass density separation process (Frankel Co. — fluidized bed separation) were received by the nonconsumable melter (Teledyne-ALLVAC).

Representative samples of the separated chips were analyzed for elemental and interstitital content to determine the additions of virgin titanium sponge, master alloy and iron, necessary to attain AMS 4928 chemistry specification. The elemental sponge will dilute the interstitial oxygen level of the chips; the master alloy and iron additions will thereupon adjust the elemental sponge addition alloy chemistry.

Several trial melt runs were made, using surplus chips in the feeder system of the nonconsumable melting system. During these operations, it was observed that an occasional, stray feed chip would miss the skull melt and be deposited directly within the ingot mold cavity. Further observation indicated that a chip could tumble from the lip of the feed hopper and fall directly into the mold cavity, be propelled into the cavity after contact with the rotating electrode and/or fall into the unmelted rim of the skull and drop into the mold cavity during subsequent

tip-pouring operations. In each possibility, the situation would tend to nullify the function of the skull melt which is to separate dense inclusions from the melt, i.e., heavy WC tool bit contaminants will sink and be retained in the solidified skull, thereby producing a noncontaminated, nonconsumably-cast ingot. In summary, stray chips cannot enter the ingot mold cavity if complete melting separation is to be assured.

Teledyne-ALLVAC responded to the problem with modifications to both the furnace and process operation procedures. A movable cover plate and an additional sight port were added to the tunnel to shield the ingot mold cavity during all feeding and melting operations. This will preclude the possibility of direct chip impingement into the ingot mold cavity. The stray chip possibility on the unmelted skull rim is being avoided by a modified process procedure (interrupted melt plus reversed skull tip) followed by a feed hopper redesign. Nonconsumable melting of the aforementioned lots of separated titanium chips was then scheduled.

3. Input Material Inspection

a. Chemistry

Upon receipt of the Frankel Company fluidized bed chips, chemistry samples were taken from both lots and analyzed so that accurate charge makeups could be determined. Sampling was accomplished by retrieving and thoroughly mixing 0.5-lb grab samples from the top of three random drums from each lot. This practice was repeated for an additional three drums from the same lot to produce two chemistry samples per lot. A portion of each sample was then quartered out and distributed at Teledyne-ALLVAC and to an external source for the manufacture of buttons for complete chemistry. Extra analyses for O_2 were performed due to the concern over accurately diluting this major chip contaminate.

Complete results of all chemical analyses are summarized in Table 85. Data were reasonably consistent, with the exception of some N_2 values, and agreed very well with Frankel Company results. These chemistries indicated it would be possible to formulate heats with a 50/50 chips/virgin blend and meet all chemistry requirements of AMS 4928G.

TABLE 85
CHEMICAL ANALYSIS OF Ti-6Al-4V CHIPS AS-RECEIVED FROM FRANKEL COMPANY, LOT A

Element	*Spec AMS 4928 Min*	*Max*	*ALLVAC Button ALLVAC Analysis Drums No. 1,2,3*	*Wah Chang Button Wah Chang Analysis Drums No. 1,2,3*	*Wah Chang Button ALLVAC Analysis Drums No. 1,2,3*	*ALLVAC Button ALLVAC Analysis Drums No. 4,5,6*	*Wah Chang Button Wah Chang Analysis Drums No. 4,5,6*	*Wah Chang Button ALLVAC Analysis Drums No. 4,5,6*
C		0.10			0.026	0.038		0.028
Mo			0.01			0.05		
Sn			0.03			0.05		
Cr			0.01					
Ni			0.03					
Fe		0.30	0.19			0.20		
Cu		0.10	<0.01					
Zr			0.01			0.03		
Mn								
V	3.50	4.50	4.05			4.06		
Al	5.50	6.75	6.45			6.45		
Si		0.10	0.02			0.02		
Y		0.005	<0.005			<0.005		
H_2		0.0125		0.0059			0.0064	
N_2*		0.05	0.037	0.0180	0.0125	0.1620	0.0180	0.0170
O_2*		0.20	0.250	0.240	0.237	0.287	0.240	0.222
B		0.003	<0.001			<0.001		

*Average of Multiple Runs

b. High Density Particles

As chemistry samples were being analyzed, additional sampling was performed in an effort to locate tungsten carbide particles in the chips. Assuming that any such particles would tend to settle, 30 lb of chips from the bottom of two random barrels of each lot were dumped into clean poly bags and taken to an outside source for X-ray examination.

Chips from each sample were placed 4 in. deep in a plastic container 11 in. wide by 13 in. long. Three tungsten carbide sensitivity indicators 10, 15, and 20 mils thick were taped to the tray nearest the source to serve as standards. They were situated in such a manner that they would run diagonally across the developed film. To differentiate a carbide standard from a particle which might be present in the chips, small lead marker arrows were placed near the carbide indicators, pointing directly at them. X-rays were taken at a source input of 220 kilovolts and 3 milliamps. A source to film distance of 58 in. was used with an exposure time of 15 min. Kodak type M-54 film was used with a development time of 8 min at 68°F. Film was double loaded to confirm any questionable sighting which might be the result of imperfections on the film. Two sets of photos were taken for each sample set representing alternate halves of the container due to film size limitation.

X-ray results revealed the presence of a variety of high density in both lots of chips. The carbide-like indications were seen in three of the six sample trays examined and appeared to be in the range of 10 to 30 mils when compared to the sensitivity standards. Figures 116, 117 and 118 show the suspicious areas indicated with a white arrow.

Chips from the trays shown to contain high density particles by X-ray were hand screened through 16 by 18 mesh window screen. The resulting fines were sorted magnetically, and the magnetic fraction was pickled in hot dilute sulfuric acid. A few of the most suspicious looking particles remaining were sorted out by hand and sent to Micro Met Laboratories to determine if any were tungsten carbide. Examination by a scanning electron microscope (SEM) equipped with an X-ray energy dispersive analyzer proved negative.

A second attempt was made to locate high density particles by machine screening an entire 50-gal drum selected at random from each lot of chips. The same 16 by 18 mesh window screen was used as before, and the fines were sorted and pickled in the same manner. The end product of this effort was a very small quantity of particles (less than one gram) from each of lots A and B. The entire quantity of material represented was again sent to Micro Met Laboratories.

This time the presence of WC particles was confirmed by SEM on both lots A and B (Figure 119). It was reported for the sample examined that the WC particles were more numerous and larger (10 to 25 mils) in lot B than in lot A (5 to 10 mils).

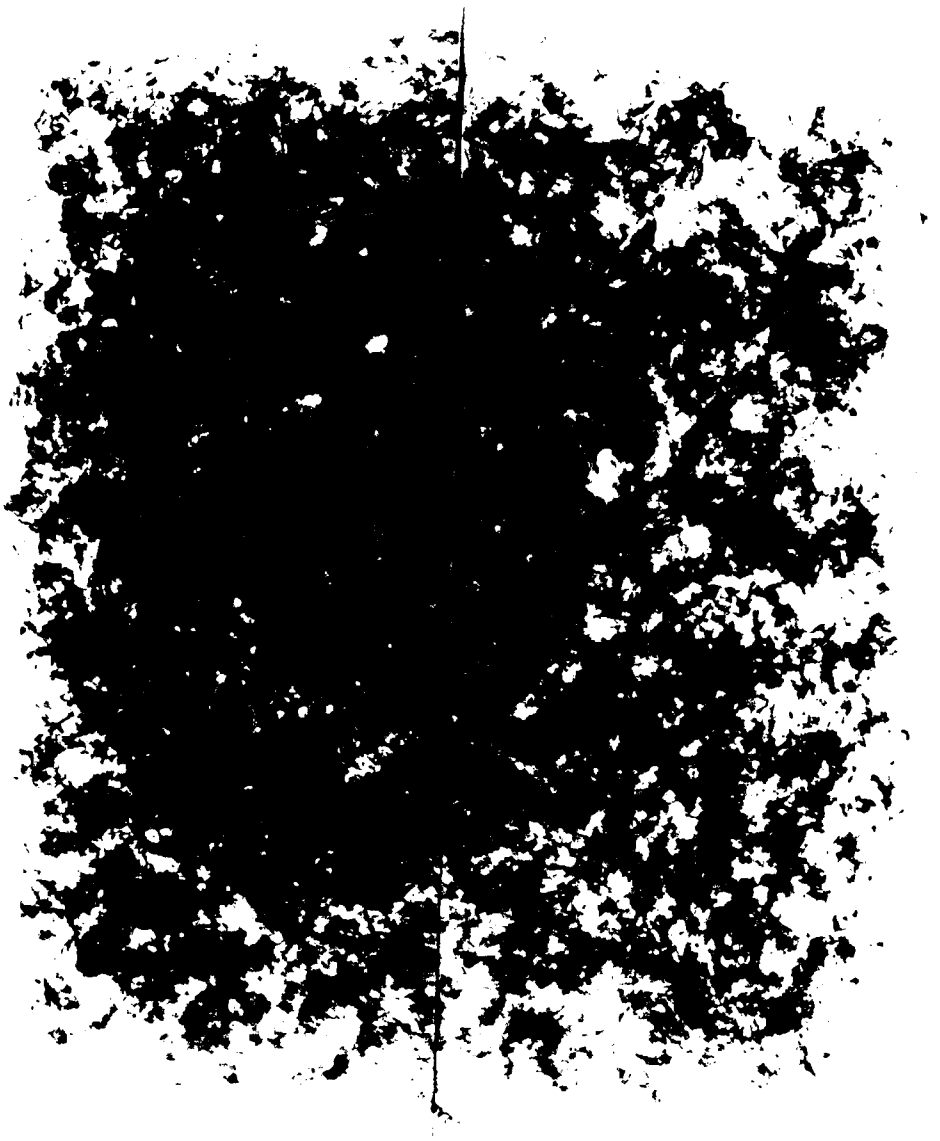
4. Charge Preparation

Chemical analysis of the turnings presented in Table 85 indicated that a 50/50 blend of chips and virgin material could be made with a resultant O_2 content well within specification limits. Two 5,000-lb heats were therefore formulated, one from each of the two lots represented. Theoretical oxygen content of the two heats was calculated at 0.150%. A complete list of the materials applied to these two heats is presented in Table 86.

The 50% virgin portion of each heat was blended and split off into a large number of small drums. Chips were uniformly mixed in which each drum. A number of these small drums were then dumped together into larger 50-gal drums, and later several large drums were dumped together into the feeder tube of the nonconsumable furnace. No purposeful addition of high-density particles was made to either of the two lots of chips to Teledyne-ALLVAC.

FC 34253

Figure 116. X-ray Positive of Ti-6Al-4V Chips As-Received from Frankel Company, Lot B, Drum 1, Approximately ½ Times Original Size



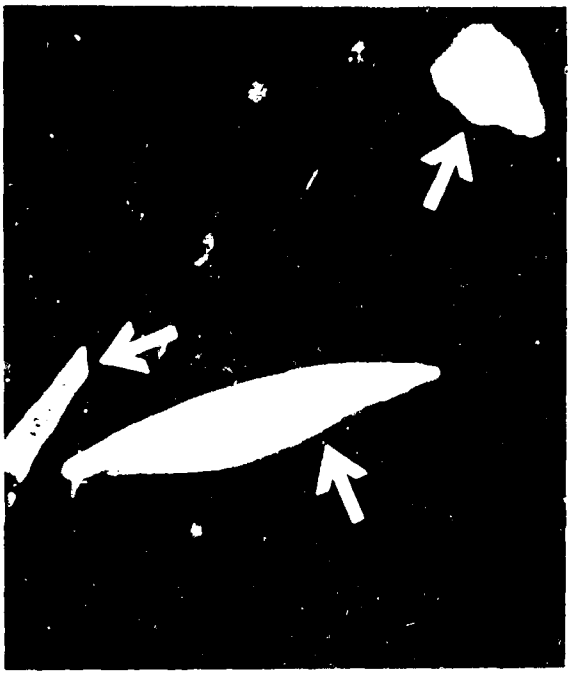
FC 34253

Figure 117. X-ray Positive of Ti-6Al-4V Chips As-Received from Frankel Company, Lot A, Drum 1, Approximately ½ Times Original Size

FC 34255

Figure 118. X-ray Positive of Ti-6Al-4V Chips As Received from Frankel Company, Lot A, Drum 1. Approximately Times Original Size

Lot A, 100X



Lot B, 100X

Markers Point to W/C Particles

Figure 119. SEM Photographs of Tungsten Carbide Particles Found in As Received Chips

TABLE 86
RAW MATERIAL APPLIED TO Ti-6Al-4V CHIP, NONCONSUMABLE HEATS

Material	*Heat No. F551 Lot A, (lb)*	*Heat No. F552 Lot B, (lb)*
Chips	2,500	2,500
Ti Sponge (0.06% O_2)	2,235	2,236
VAl Masteralloy (65/35)	170	172
Al	93	90
Fe	1	1
Total Pounds	4,999	4,999

It was originally planned to compact the sponge-chip mixture into a 16-in. dia pressed compact and then shear this into many smaller pieces. The purpose was to obtain pieces of a size ideally suited for feeding to prevent any unmelted material, particularly high-density particles, from missing the skull melting crucible and accidently entering the withdrawal casting mold.

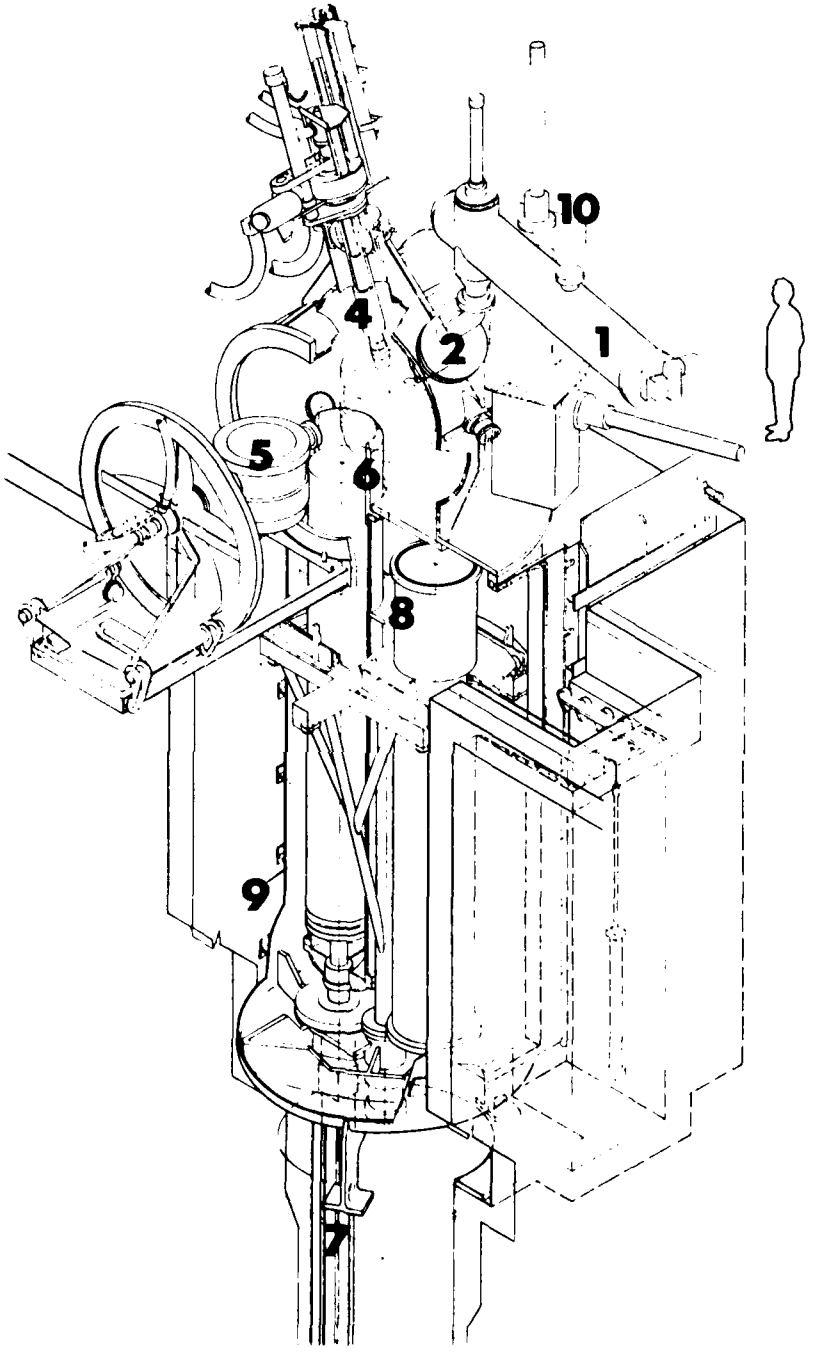
It was apparent after the attempts at shearing the sponge/virgin compacts that they had insufficient green strength to yield the solid pieces required regardless of the pressing conditions employed with sponge contents up to 70%. It was concluded that the melting equipment would have to be modified to permit the direct feeding of a loose mixture of chips and sponge.

5. Nonconsumable Melting Equipment

a. Description

The nonconsumable rotating electrode furnace at Teledyne ALLVAC and its operation have been described in detail earlier in this report. The furnace is shown in perspective in Figure 120 and an interior view in Figure 121. *Major features include:*

1. Separately valved vacuum chamber equipped for the continuous charging and controlled feeding of the raw materials
2. A chute to direct the feeding of the raw materials into the melting crucible
3. A 20,000-amp power supplied by two SCR-type rectifier sets (Not Shown)
4. A 300 rpm rotating electrode with a 7-in. dia water-cooled copper tip capable of being directed within the circumference of the melting crucible
5. An 800-lb capacity 18-in. dia water-cooled copper tiltable melting crucible enclosed in a seven-ft dia vacuum melting chamber
6. Water-cooled steel casting mold, 21-in. in dia
7. Hydraulic ram mechanism for the bottom withdrawal of cast electrodes from the casting mold
8. Sixteen-ft long steel cooling can vacuum sealed to the melt chamber and withdrawal ram mechanism
9. Eight ft dia by 20 ft high vacuum casting chamber equipped to repetitively remove completed castings and position new cooling cans under the mold
10. Optical viewing system for observing the arc



FC 34256

Figure 120. Teledyne-ALLVAC's Nonconsumable Rotating Electrode Furnace, Perspective

FC 34257

Figure 121. Nonconsumable Furnace Interior Following a Melt. 1 Skull Melting Crucible, 2 Rotating Electrode, 3 Withdrawal Casting Mold

b. Modifications

A number of modifications to the furnace were necessitated by the requirement of direct feeding a loose mixture of reclaimed chips, sponge and alloying additions directly into the skull melting crucible. The object of these modifications was to ensure that the unmelted material would not enter the casting mold directly from the feed chute, thereby negating the refining portion of the melting operation. These modifications included:

1. An air operated movable shield to permit completely covering the mold chamber while feeding and melting. This was operated in such a manner that the opening to the casting withdrawal mold was exposed only during the pour.

2. A new sight port to permit improved observation of the feeding and pouring operations.

3. A new feeder chute to improve the direct feeding of the chip/virgin blend. This single change proved to be extemely difficult and resulted in several weeks of delay in the final melting while a succession of designs were fabricated, installed and tested by actual trial melting of chips.

   It was deemed desirable that the new feeder chute be as small a diameter tube as possible to direct the raw materials accurately into the melt crucible. This increased the chances of bridging, so an air-operated vibrating device was attached to the feed chute. It was also desirable that the chute extend as far out over the lip of the crucible as possible. This presented serious problems due to the very high temperatures to which the tip of the chute was exposed and to the rapid splatter buildup characteristic of nonconsumable arc melting. This buildup, which welded to the tip of a steel feed tube, immediately caused a small amount of feed material to hang up on the lip of the feed chute, which served to catch more splatter and stop more feed material, and so on until feeding was stopped off completely. The problem was solved by fabricating the entire lower portion of the feed chute out of double-walled, water-cooled copper which minimized the accumulation of the molten Ti splatter. A bridge breaker was also also installed to permit mechanically removing any blockages from the feed tube.

4. Due to the length of the redesigned feed shoot, it became necessary to install a hydraulic lift to raise the shoot to clear the furnace when it was tilted for pouring.

6. Nonconsumable Melt Practice

After the above modifications had been tested and successful trial melts with chips made, it was concluded that the system was workable and was the best which could be achieved without a truly major redesign and furnace modification requiring several months. However, as a further precaution, a special melting practice was employed to prevent the possibility of chips landing on the top of the melting crucible and falling into the mold during the pour. This practice consisted of extinquishing the arc after the crucible was full and allowing the melt to solidify. With the mold cover plate closed, the furnace was tilted as far forward as it would go (approximately 100 deg) and shaken to permit any loose particles to fall off. It was then brought back to horizontal, the arc restruct and a molten pool established. The mold cover plate was then opened, the arc terminated and the charge poured.

To begin operation, the furnace was pumped down to less than 100 microns, leak checked and then backfilled with argon to a pressure of 8- to 10-mm Hg. The initial melt crucible skull was formed by feeding approximately 125 lb of charge in an empty crucible and striking an arc at a power setting of 8,000 amps and 32 volts. After a pool was established and a satisfactory skull was formed, power was incrementally increased to 15,000 amps and 44 volts the nominal melting parameters for this alloy. After establishing the skull and bringing the pool out to the wall of the melting crucible, the blended raw materials were fed at the maximum rate at which they could be melted in. As soon as the furnace was filled to the desired capacity, melt power was terminated and the molten metal allowed to solidify for the tilt-shake sequence. Upon reestablishment of this molten pool, a stabilized heating condition was maintained at 12,000 amps and 42 volts for a period of 10 min prior to pouring. This lower power input was used in order to remelt the charge to a lesser depth than was present during initial melt-in, thus assuring that any carbide particles trapped in the skull would not be released and poured with the molten charge.

At the end of each of these cycles, the crucible was charged with additional raw materials on top of the remaining skull, and the complete cycle was repeated. The total lapse time for each cycle was on the average 70 min. This was considerably longer than the normal 30 to 40 min due to the time required to freeze, shake and remelt.

During the course of melting, it was necessary to refill the feeder. This was accomplished by valving off the feed chamber, backfilling with argon to atmospheric pressure, withdrawing the empty feeder can, charging a previously loaded second feeder can into the chamber, sealing and pumping down to furnace pressure. This procedure typically required about 30 min, which permitted the melting to proceed with only a minor interruption.

Twenty-one-in. dia electrodes were cast using a bottom withdrawal system. Internal chips generated with noncarbide tools were used as a pouring pad at the bottom of the 3-ft long water-cooled steel casting crucible. Upon solidification of each pour, the electrode was positioned for the next pour by withdrawing it the necessary distance by means of a hydraulic ram. The resulting electrodes consisted of 16 and 17 successive pours and weighed 4,218 and 4,334 lb for Lots A and B, respectively. Figure 122 shows the second of these two electrodes, F552.

The same melting procedures were followed for each heat except that the skull produced, while melting lot A was also used to melt lot B. Melting of both heats generally proceeded without delay except for some chute blockages which occurred during the last pours of each. These blockages created only minor delays, however, since they were easily dislodged via the use of the built-in bridge breaker rod.

7. Consumable Melt Practice

The 21-in. dia nonconsumable melted electrodes were inverted and vacuum arc consumable remelted in a 26-in. dia crucible. Melting was initiated at 6,000 amps, 32 volts, at a pressure of 8 microns and was increased at approximately 1,000 amps/min until peak power of 24,000 amps was obtained. After a short time at peak amperage, the power was gradually reduced to a steady-state melting condition of 20,000 amps followed by a gradual reduction in power to complete a hot top cycle at 3,500 amps. VAR melting was completed without incident and resulted in ingots of 3,645 lb (F551) and 3,769 lb (F552) for Lots A and B, respectively. Figure 123 shows the 26-in. dia ingot, heat F551.

FC 34258

Figure 122. A 21-in. Diameter Bottom Withdrawal Electrode Cast from the Nonconsumable Furnace, Ti-6Al-4V, Heat No. F552

FC 34259

Figure 123. A 26-in. Diameter Single Consumable Remelted Ti-6Al-4V Ingot, Heat No. F551

Chemistry was obtained by drilling near top and bottom samples from both heats. Results presented in Table 87 indicate that chemistry was well within specification limits, with O_2 content falling almost right on anticipated aims. There were no significant differences between top and bottom chemistries of either ingot. Grinding was performed on heat F552 due to a rough surface condition. Although the exact cause was not determined, it was speculated that inadequate cleaning of the VAR crucible may have caused this situation. No conditioning was necessary on heat F551.

8. Ingot Conversion

The ingots were sent to the Ladish Company of Cudahy, Wisconsin for conversion to 9-in. dia forging billet. While specific details of the conversion operation are proprietary, it can be said that the conversion was by means of cogging on a 3,000-ton press. The finishing passes were conducted in the alpha-beta region using round dies to produce the billet. The overall reduction ratio of 8.35:1 yielded approximately 30 ft of barstock from each ingot. For ease of handling during the cogging operation, each ingot was cut into thirds to produce three 10-ft lengths of barstock from each of the heats F551 and F552.

9. Billet Evaluation

Figure 124 indicates the location of the four etch slices taken from master heat billet BCB, corresponding to Lot A (unseeded) turnings, Teledyne-ALLVAC master heat number F551. The three lengths of billet are of equivalent size so that etch slices H6-485T and H6-486T are spaced at 1/3 intervals along the length of the master heat bar. Figure 125 likewise shows the etch slice locations from master heat BBA, corresponding to Lot B (seeded) turnings, Teledyne-ALLVAC master heat number F552.

All eight etch slices were polished on one face by standard metallographic procedures before being etched by Kroll's reagent. Figures 126 through 133 are macrophotographs of the respective etch slices. These photos were produced by a collage of smaller negatives in order to maintain resolution, hence the mottled appearance of some figures. The linear defects in Figures 126, 129, and 132 are due to tne etch slice location being too close to the end of the billet and/or insufficient material removal from the polished face.

Figures 134 through 137 are 100X micrographs of acetate replicas pulled from the central area of the respective etch slices. No evidence of foreign material was found in any of the etch slices. A few isolated areas suggestive of excess alpha stabilizer concentration were noted in etch slice H6-483B, but in no case was this condition worse than that occasionally observed in triple melted stock, and not severe enough to be cause for rejection. An example of such an area is shown in Figure 138.

10. Disk Evaluation

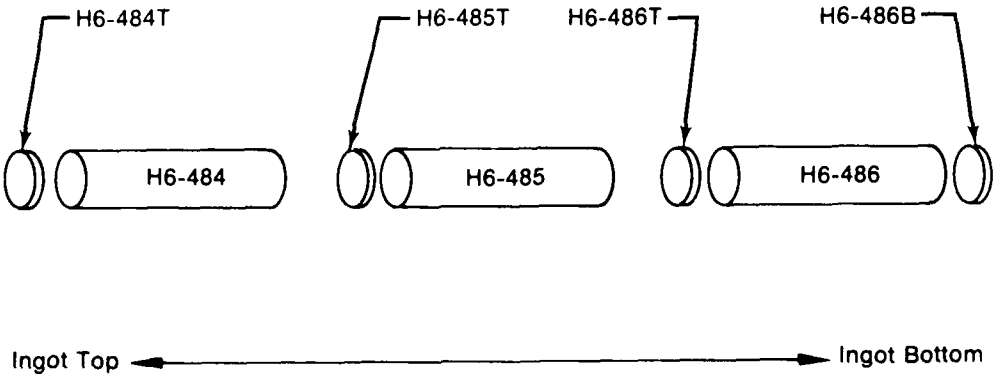
Four TF-33 2nd-stage fan disks were successfully forged from the reclaimed Ti alloy barstock, two from each master heat. All four disks were sonically inspected and accepted according to flight quality inspection criteria.

The four disks were also blue-etch anodize inspected and were shown to meet the flight quality standards of this inspection technique. Blue-etch anodizing is a very sensitive inspection technique for the detection of foreign material massive phase agglomeration. One of the disks in the sonic machined and blue-etch anodized condition is shown in Figure 139.

TABLE 87
CHEMICAL ANALYSES OF Ti-6Al-4V SINGLE REMELT INGOTS

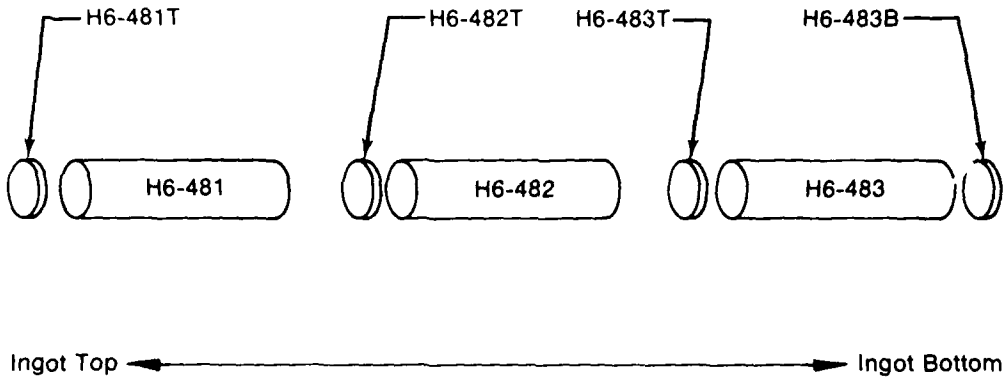
Element	Spec AMS 4928G Min	Max	Aim	Top F551 Lot A	Bottom F551 Lot A	Top F552 Lot B	Bottom F552 Lot B
C		0.10	Lap	0.036	0.034	0.038	0.032
Mo				0.01	< 0.01		
Sn				0.02	0.02	< 0.01	< 0.01
Cr				0.0061	0.0071	0.0076	0.0079
Ni				0.0088	0.0096	0.0151	0.0158
Fe		0.30	0.15	0.15	0.16	0.17	0.16
Cu		0.10	Lap	0.0198	0.0124	0.163	0.0254
Zr				0.01	< 0.01	< 0.01	< 0.01
Mn				0.0051	0.0035	0.0027	0.0038
V	3.50	4.50	4.20	4.05	4.00	4.10	4.10
Al	5.50	6.75	6.30	6.35	6.25	6.15	6.25
Si		0.10	Lap	0.02	0.02	0.02	0.02
Y		0.005	Lap	< 0.005	0.005	< 0.005	< 0.005
H_2		0.0125	Lap	0.0006		0.0014	
N_2*		0.05	Lap	0.008	0.008	0.009	0.009
O_2*		0.20	0.15	0.161	0.152	0.158	0.151
B		0.003	Lap	< 0.001	< 0.001	< 0.001	< 0.001
Ti			Bal	Bal	Bal	Bal	Bal

*Average of Multiple Runs



FD 108102

Figure 124. Billet Cut-Up of Master Heat BCB. (Teledyne — ALLVAC F551, Lot A Chips)



FD 108103

Figure 125. Billet Cut-Up of Master Heat BBA. (Teledyne — ALLVAC F552, Lot B Chips)

Figure 126. Billet Etch Slice H6-484T, 9-in. dia

Figure 127. Billet Etch Slice H6-485T, 9-in. dia.

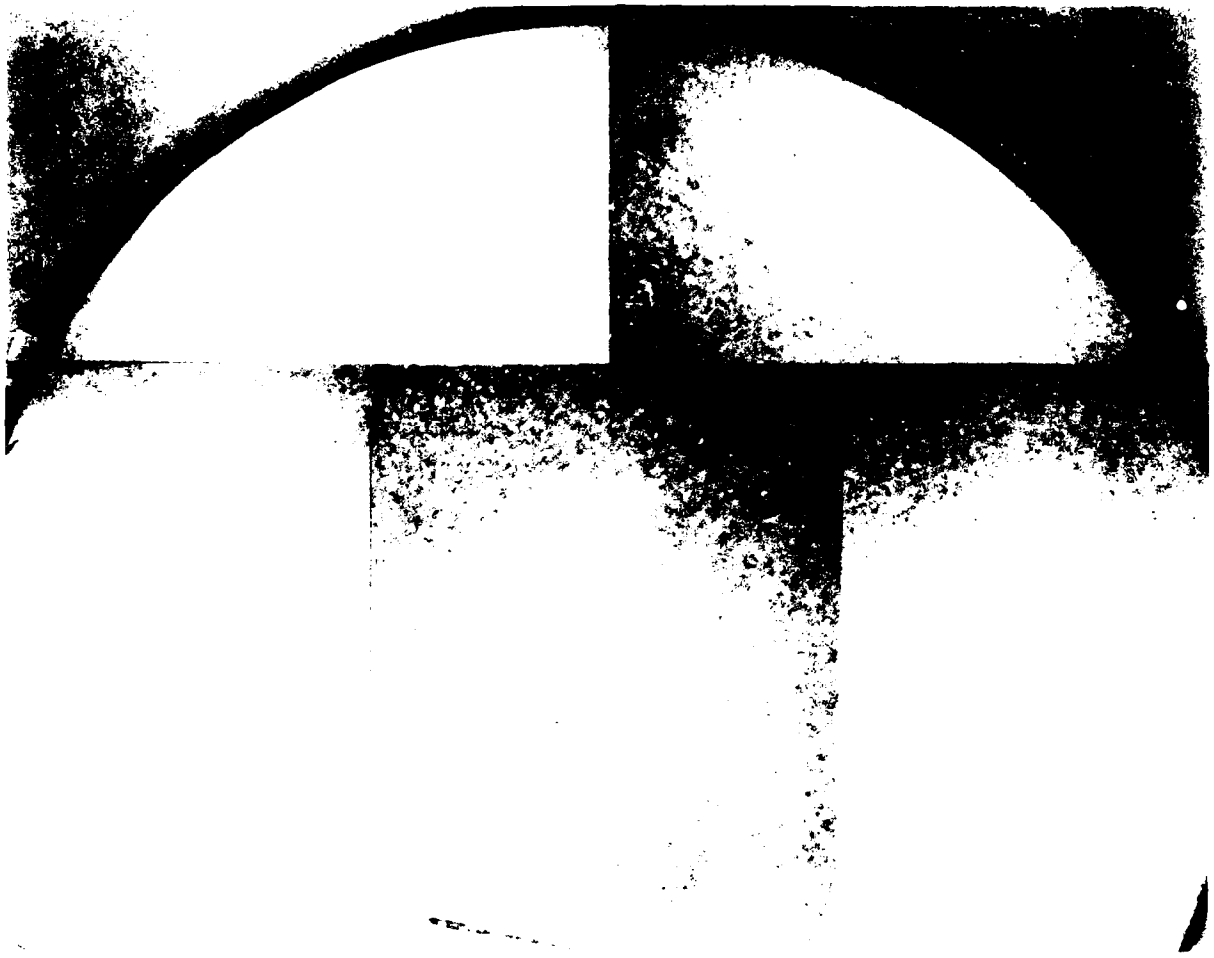


Figure 128. Billet Etch Slice H6-486T, 9-in. dia.

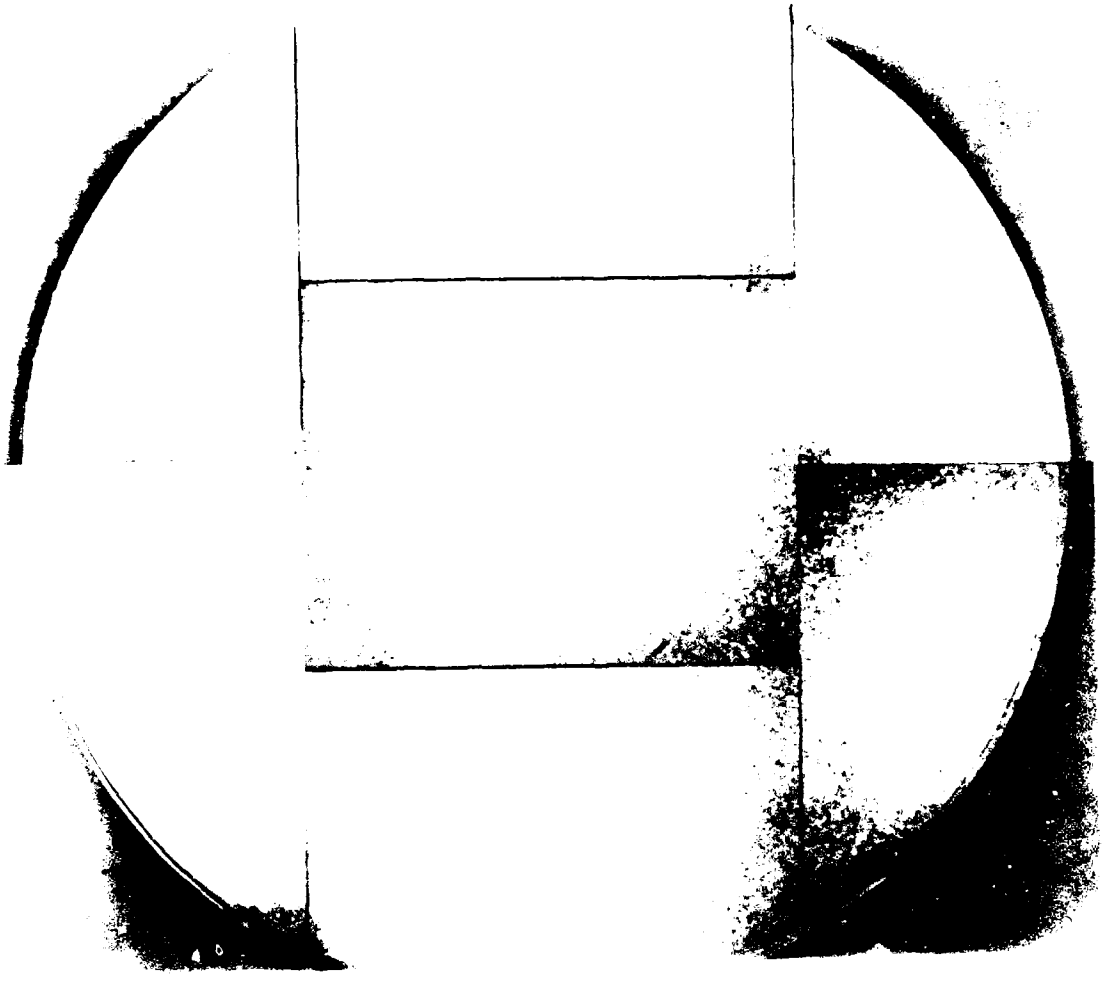


Figure 129. Billet Etch Slice H6-486B, 9-in. dia.

AD-A083 089 PRATT AND WHITNEY AIRCRAFT GROUP WEST PALM BEACH FL G--ETC F/G 13/8
MANUFACTURING METHODS FOR STRATEGIC MATERIALS RECLAMATION.(U)
DEC 79 H K GREGORY, T D TILLMAN F33615-74-C-5019
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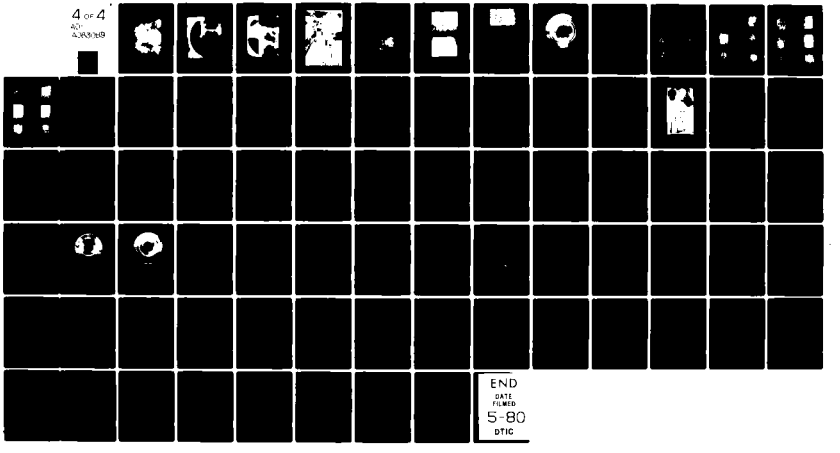

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Figure 130. Billet Etch Slice H6-481T, 9-in. dia.

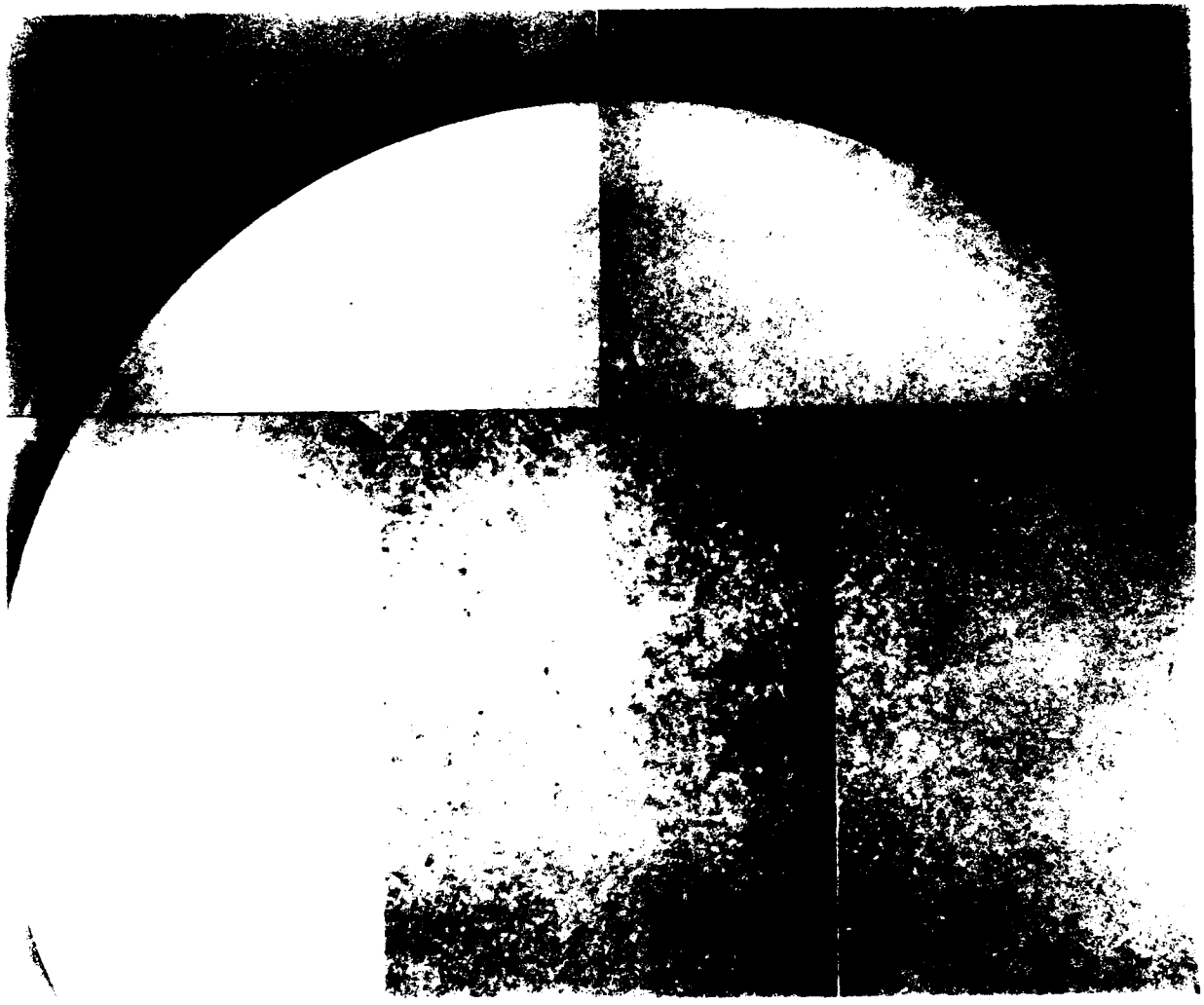


Figure 131. Billet Etch Slice H6-482T, 9-in. dia.

Figure 132. Billet Etch Slice H6-483T, 9-in. dia.

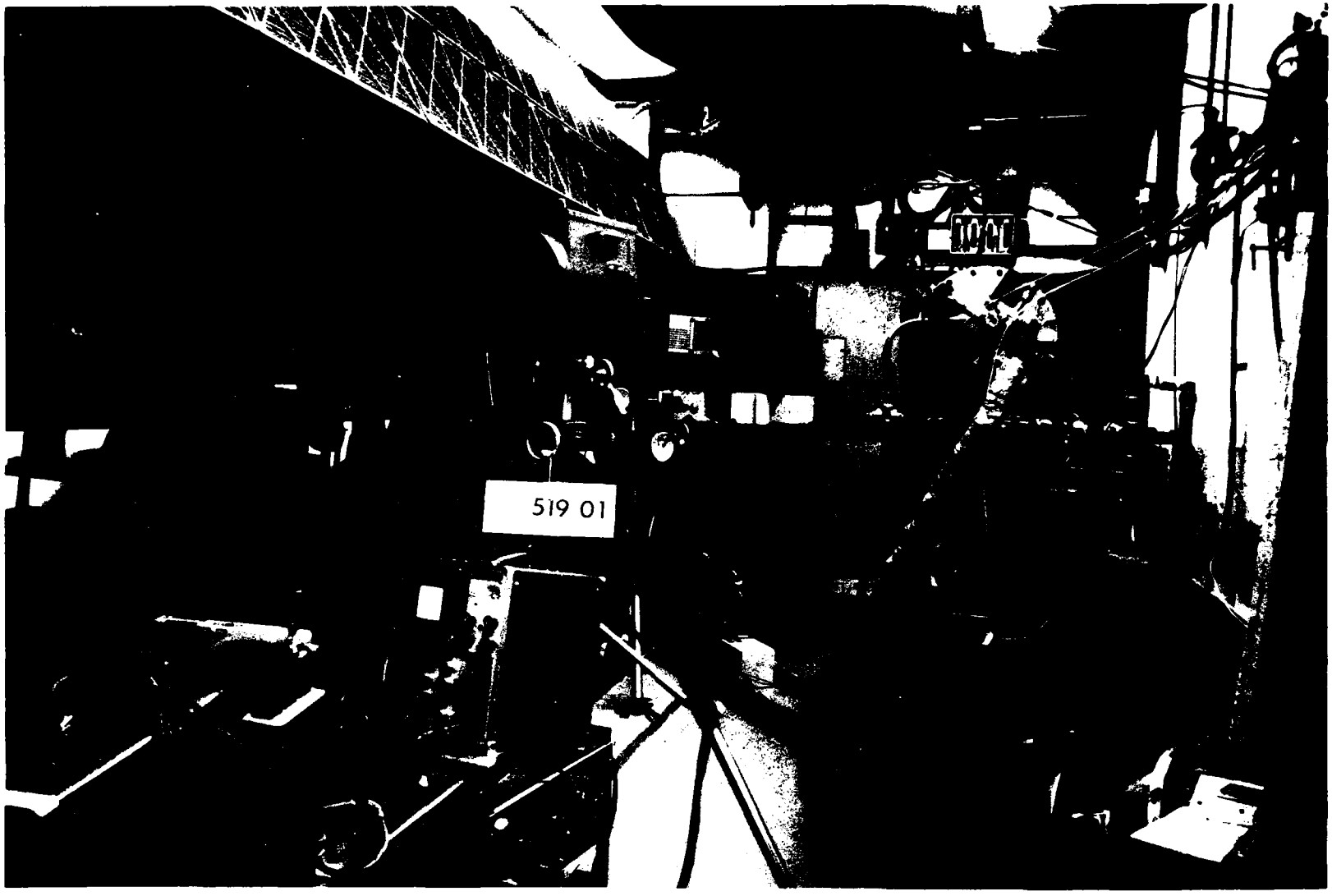
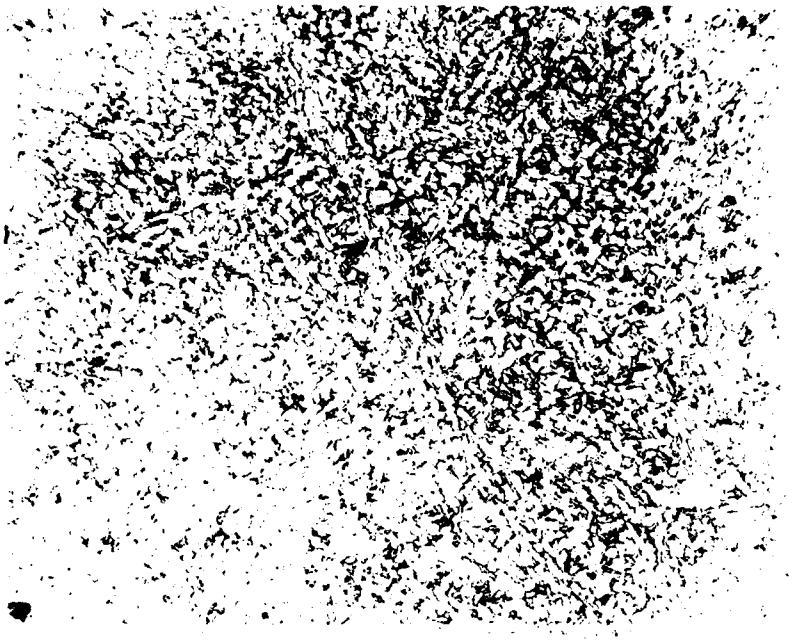


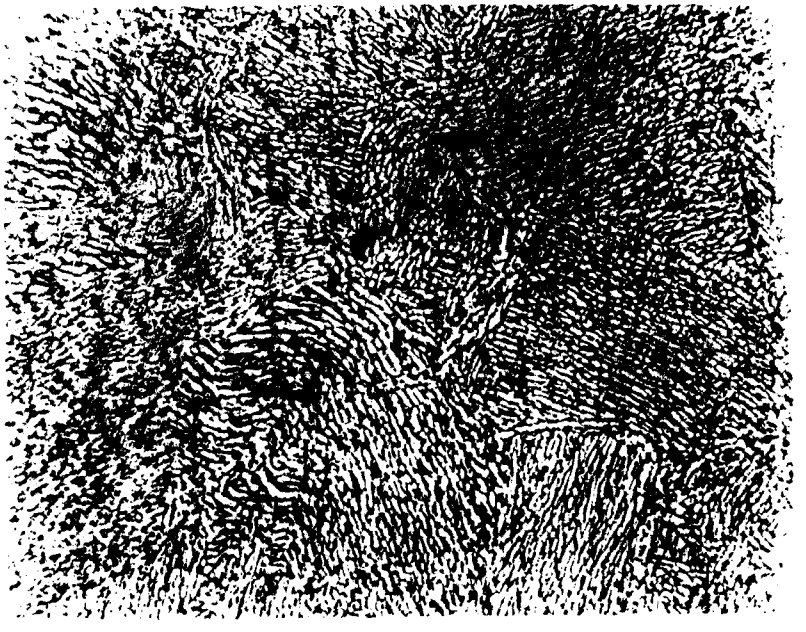
Figure 133. Billet Etch Slice H6-483B, 9-in. dia.



100X

FAM 87480
FD 108104

Figure 134. Replica from Center of H6-484T



100X

FAM 87489
FD 108105

Figure 135. Replica from Center of H6-486B

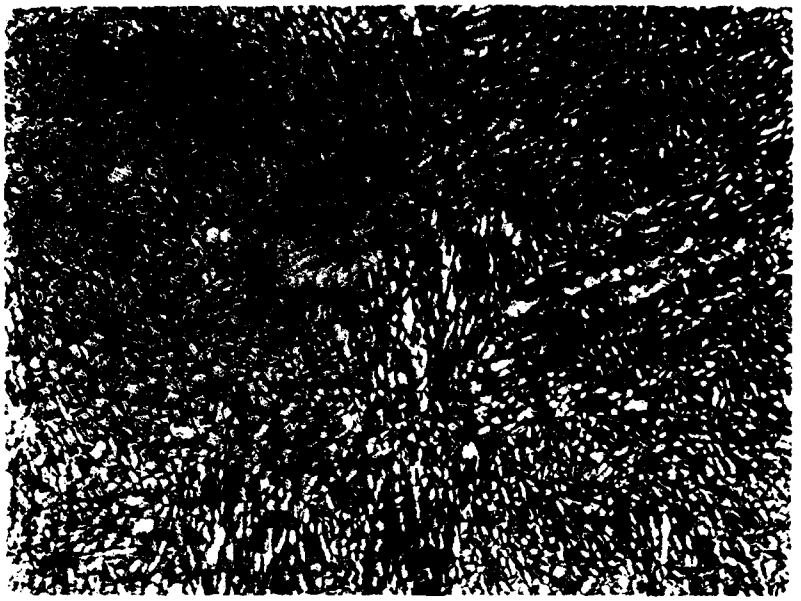


Figure 136. Replica from Center of H6-481T

Figure 137. Replica from Center of H6-483B

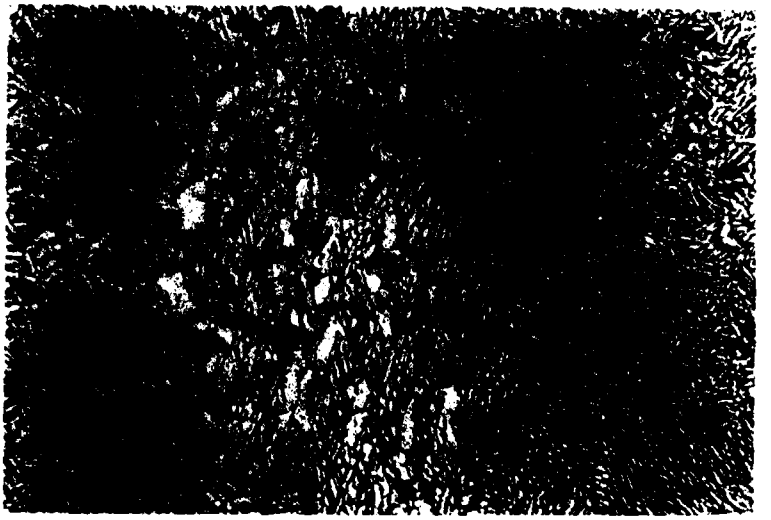


Figure 138. Isolated Coarse Alpha Morphology Observed in Etch Slice H6-483B

FE 153978

Figure 139. Reclaimed Ti-6Al-4V Fan Disk in Blue Anodized Condition

The part is first anodized to a uniform dark blue color. It is then immersed in a nitric/hydrofluoric acid solution for a short period of time in order to dissolve most, but not all of the anodic oxide film. After this step the disk is a light pale blue color. Defective areas appear either dark blue or metallic silver in color. This is due to the fact that the chemically different defects produce anodic oxides which dissolve at different rates than the anodic oxide formed by the bulk alloy composition.

Each of the four disks was evaluated metallographically at six locations. This was accomplished by polishing a spot about 1 in. in diameter at the desired location followed by etching and replication of the surface with acetate tape. The resulting micrographs and their locations are shown in Figures 140 through 143. In all cases the etchant used was Kroll's reagent.

11. Chemistry Analysis of Billets

Chemical analyses were performed on 24 specimens selected to give a representative sampling of the billets. Tables 88 and 89 summarize the results of these analyses. Chemical variations were found to exist along the length of the ingots as well as through their cross sections. Figures 144 through 149 depict the variation in chemistry vs corresponding ingot location and cross section. Although most of the heats were generally within specification limits, Figures 26 and 28 show that heat F552 locally exceeded the requirements for hydrogen and oxygen levels. In general, the graphs show that the beta-stabilizing elements tend to segregate toward the center of the billet/bars while the alpha-stabilizing elements are segregated to the mid-radius and outer radius of the mill products.

Thus, the chemistry evaluation of disk forgings indicated a general conformance to specification, with high gas elements and no significant trace element levels.

D. SUPERALLOY GRINDINGS AND SLUDGE SEPARATION PROCESS (PHASE II)

1. Background Information

Phase I efforts, substantiated by data, have realized economic potential from a superalloy grinding/sludge scrap product that is presently considered relatively worthless. Separation (molten salt and melting) effort was made in Phase I on developmental quantities to establish process feasibility. Additional processing, including quantity scale-up, was required to establish process economics. On this basis, P&WA pursued Ni-base grinding and sludge reclamation during Phase II contract activities, as originally scheduled.

2. Phase II Processing

Two lots of nickel-base superalloy sludge were procured, each lot consisting of about 1100 lb (wet). This quantity of material was considered adequate for evaluation studies at an intermediate level of process scale-up.

Operational difficulties were encountered, including an apparent inability to maintain a fully-molten salt bath pool, in the Frankel Co. molten salt facility utilized in Phase I for ecological incineration of sludge combustibles. Salt bath equipment malfunction, including localized sheet metal buckling and cracking, indicated that an alternate grindings/sludge drying method would be required to maintain Phase II schedule commitments. Accordingly, sludge drying was accomplished in conventional extraction/drying equipment. Although this procedure does not attain the absolute dryness level of salt bath facility incineration, it was deemed advisable to determine process limitations of the subsequent melting operation.

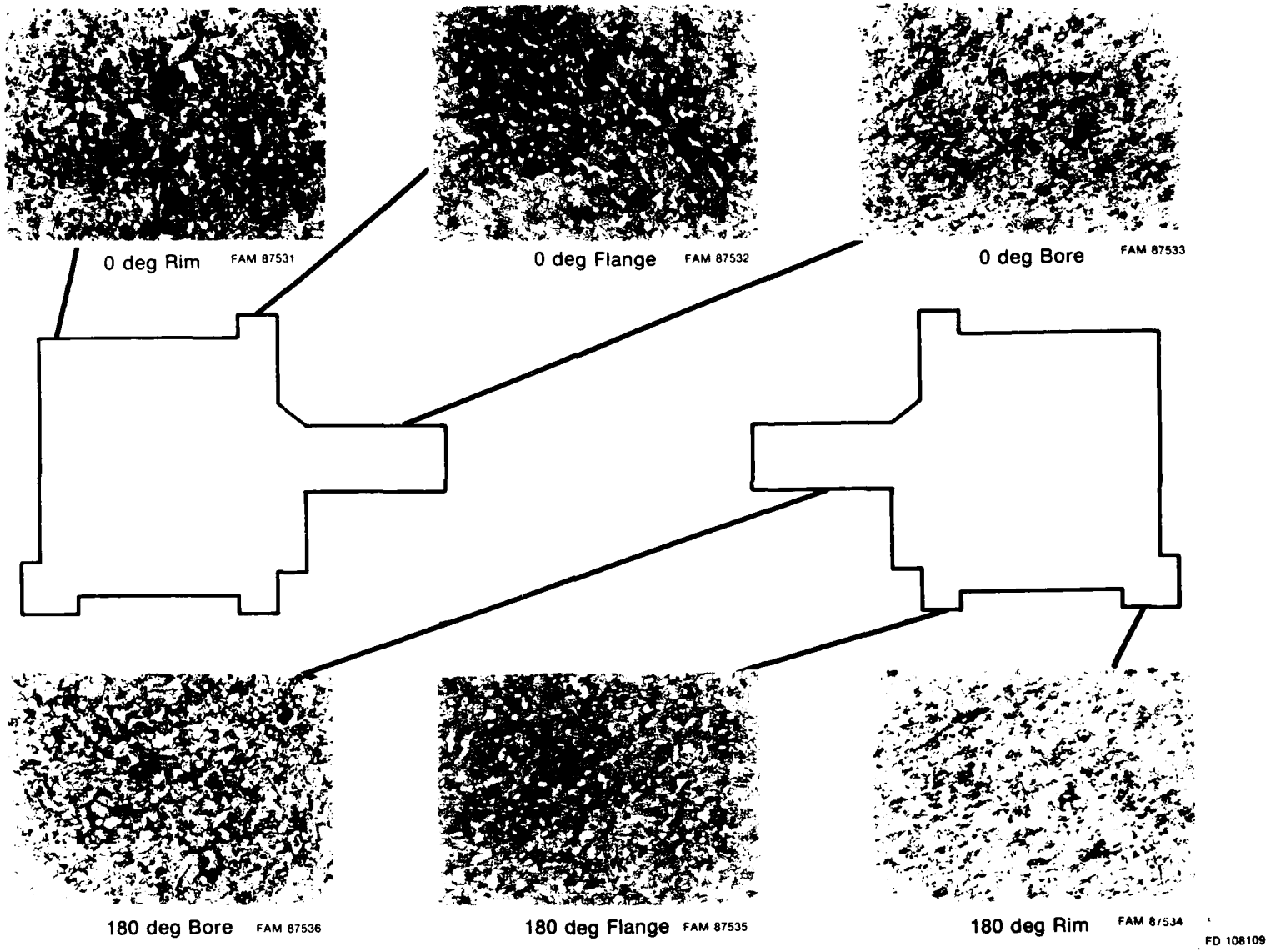


Figure 140. Metallographic Review of Disk BCB-2001 (All Micrographs 100X)

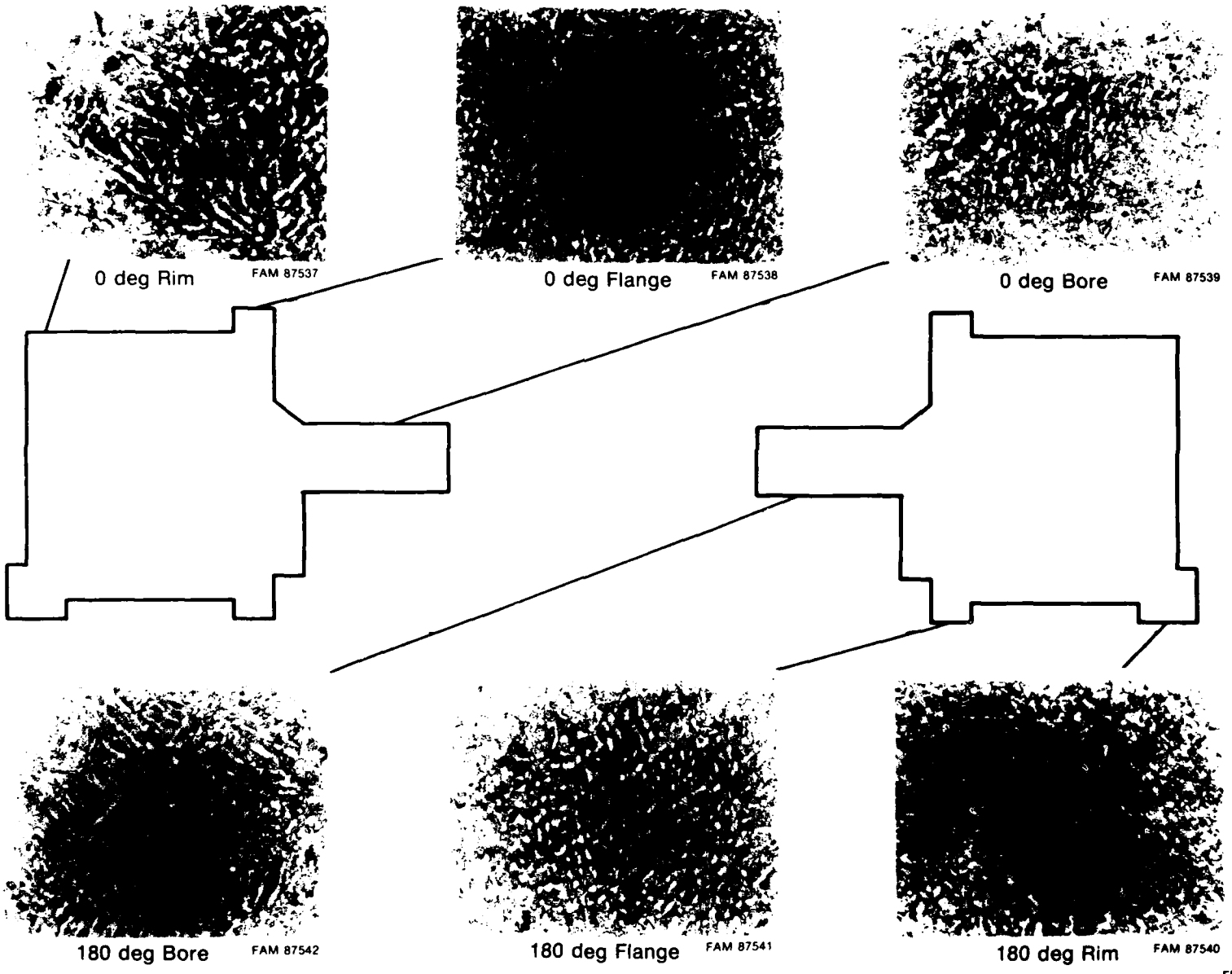


Figure 141. Metallographic Review of Disk BCB-2002 (All Micrographs 100X)

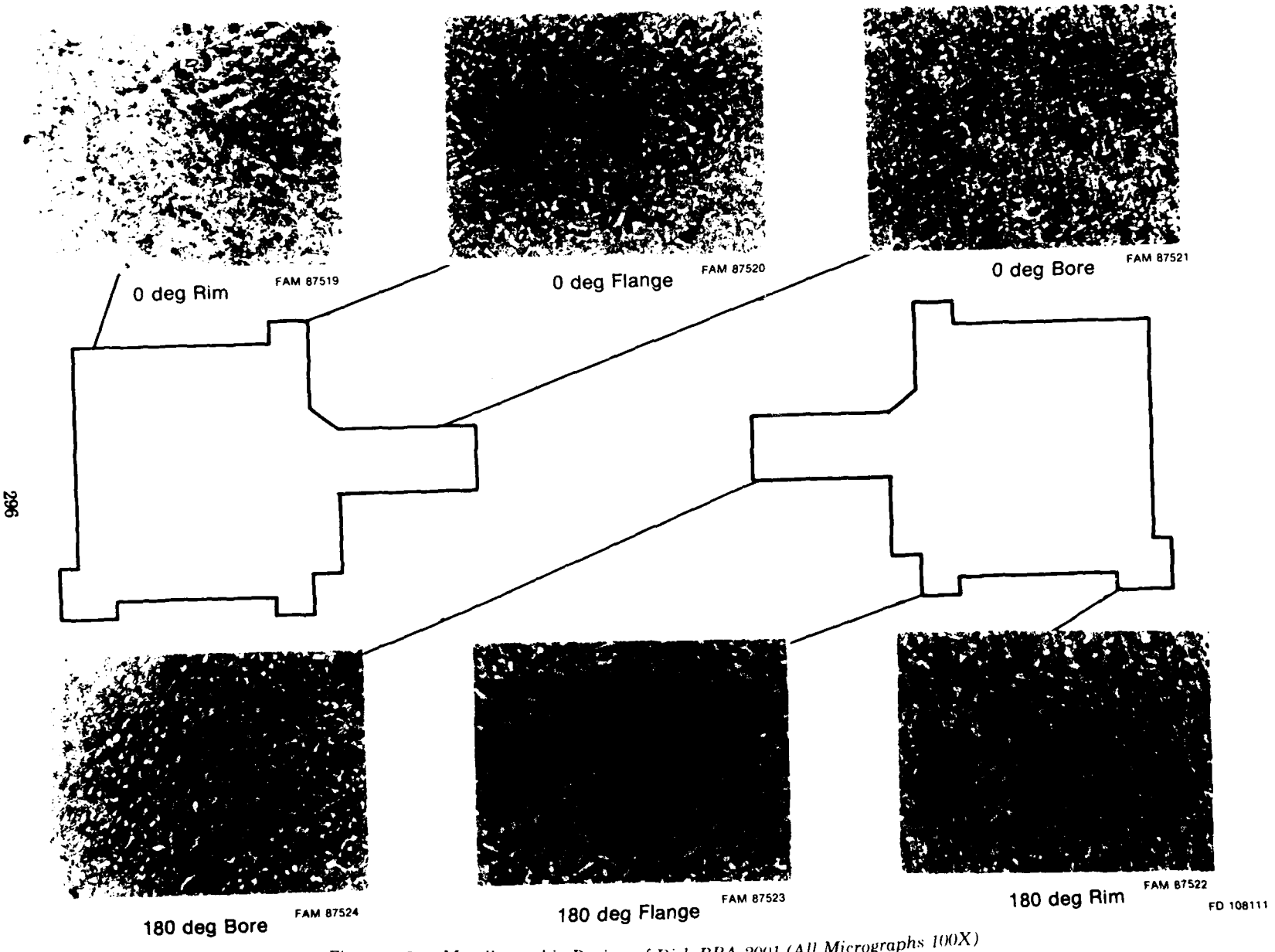


Figure 142. Metallographic Review of Disk BBA-2001 (All Micrographs 100X)

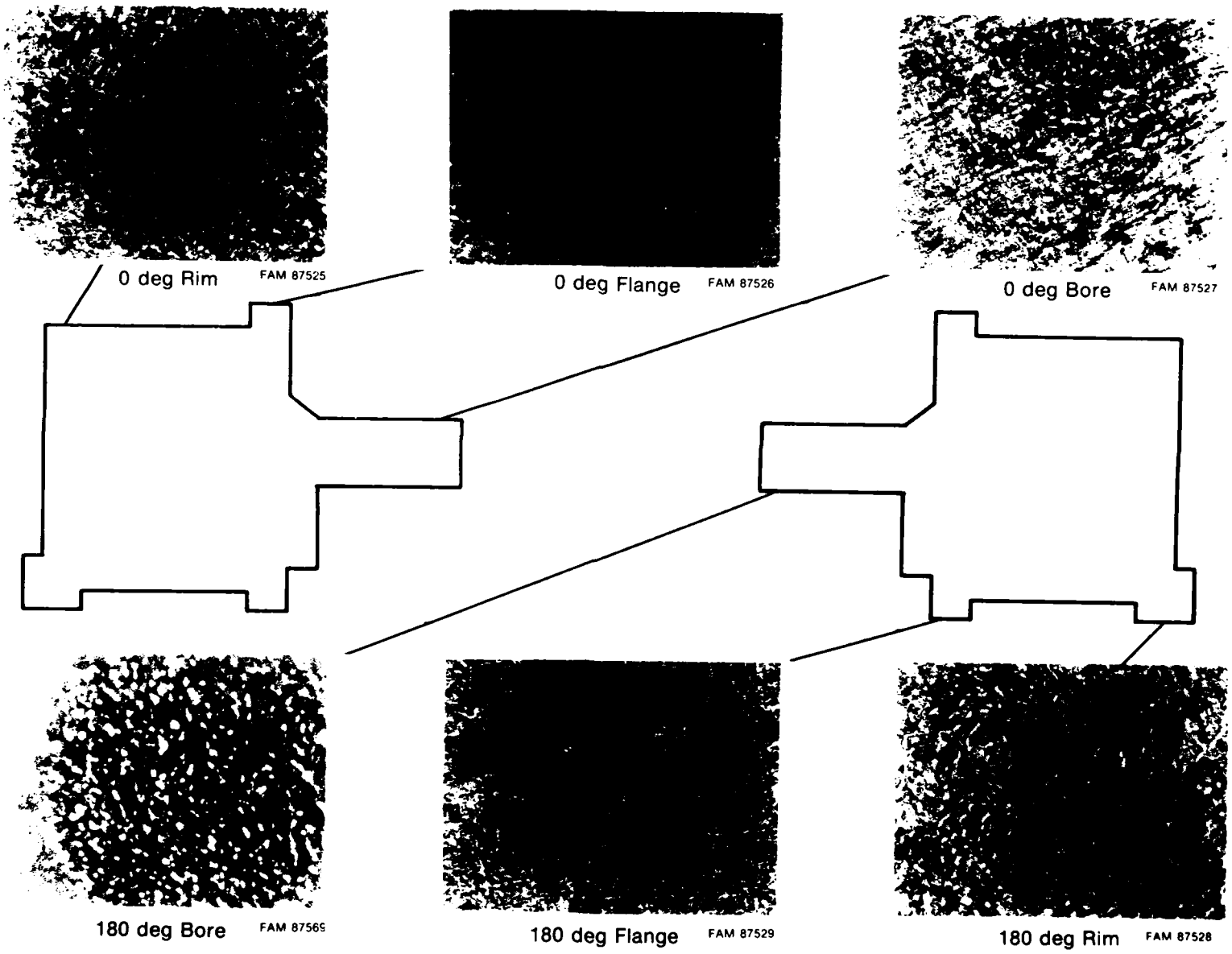


Figure 143 Metallographic Review of Disk BBA-2002 (All Micrographs 100X)

TABLE 88
CHEMICAL COMPOSITIONS OF TELEDYNE CONVERTED BILLET HEAT F551

		Corresponding Ingot Positions				
Element	*Location*	*Top*	*Top Middle*	*Bottom Middle*	*Bottom*	*Average*
Al	Edge	6.082	6.102	6.064	6.171	
	Midradius	5.724	5.845	6.163	6.101	
	Center	5.944	6.074	5.805	6.040	6.010
V	Edge	3.672	3.711	3.753	3.434	
	Midradius	3.791	3.802	3.650	3.572	
	Center	3.571	3.852	3.801	3.615	3.685
CR	Edge	0.125	0.111	0.083	0.122	
	Midradius	0.135	0.143	0.102	0.105	
	Center	0.144	0.162	0.151	0.130	0.126
MN	Edge	0.103	0.105	0.110	0.083	
	Midradius	0.105	0.113	0.103	0.103	
	Center	0.114	0.125	0.112	0.105	0.107
FE	Edge	0.182	0.160	0.181	0.130	
	Midradius	0.182	0.203	0.174	0.151	
	Center	0.215	0.232	0.193	0.151	0.180
MO	Edge	0.030	0.032	0.022	0.033	
	Midradius	0.022	0.033	0.021	0.021	
	Center	0.031	0.034	0.023	0.021	0.027
H	Edge	0.0094	0.0120	0.0099	0.0059	
	Midradius	0.0080	0.0100	0.0063	0.0086	
	Center	0.0051	0.0074	0.0052	0.0053	0.0078
N	Edge	0.0034	0.0080	0.0036	0.0047	
	Midradius	0.0074	0.0035	0.0047	0.0048	
	Center	0.0037	0.0031	0.0043	0.0082	0.0050
O	Edge	0.1405	0.1580	0.1690	0.1785	
	Midradius	0.1565	0.1490	0.1665	0.1465	
	Center	0.1465	0.1435	0.1385	0.1580	0.1543

TABLE 89
CHEMICAL COMPOSITIONS OF TELEDYNE CONVERTED BILLET HEAT F552

		Corresponding Ingot Positions				
Element	*Location*	*Top*	*Top Middle*	*Bottom Middle*	*Bottom*	*Average*
Al	Edge	5.853	5.874	5.951	6.182	
	Midradius	5.774	5.813	6.062	6.224	
	Center	5.672	5.833	5.950	5.933	5.927
V	Edge	3.805	3.844	4.013	3.621	
	Midradius	3.975	3.952	3.702	3.443	
	Center	4.032	4.011	3.515	3.630	3.795
CR	Edge	0.275	0.203	0.153	0.122	
	Midradius	0.212	0.234	0.160	0.161	
	Center	0.235	0.270	0.240	0.232	0.208
MN	Edge	0.105	0.0.041	0.034	0.021	
	Midradius	0.030	0.041	0.051	0.024	
	Center	0.042	0.024	0.041	0.022	0.034
H	Edge	0.0117	0.0081	0.0150	0.0086	
	Midradius	0.0094	0.0100	0.0057	0.0069	
	Center	0.0093	0.0059	0.0057	0.0079	0.0087
N	Edge	0.0035	0.0071	0.0043	0.0037	
	Midradius	0.0086	0.0087	0.0038	0.0043	
	Center	0.0083	0.0042	0.0044	0.0084	0.0058
O	Edge	0.1335	0.1515	0.1720	0.1675	
	Midradius	0.1565	0.1685	0.1610	0.1750	
	Center	0.1590	0.1570	0.1700	0.2400	0.1676

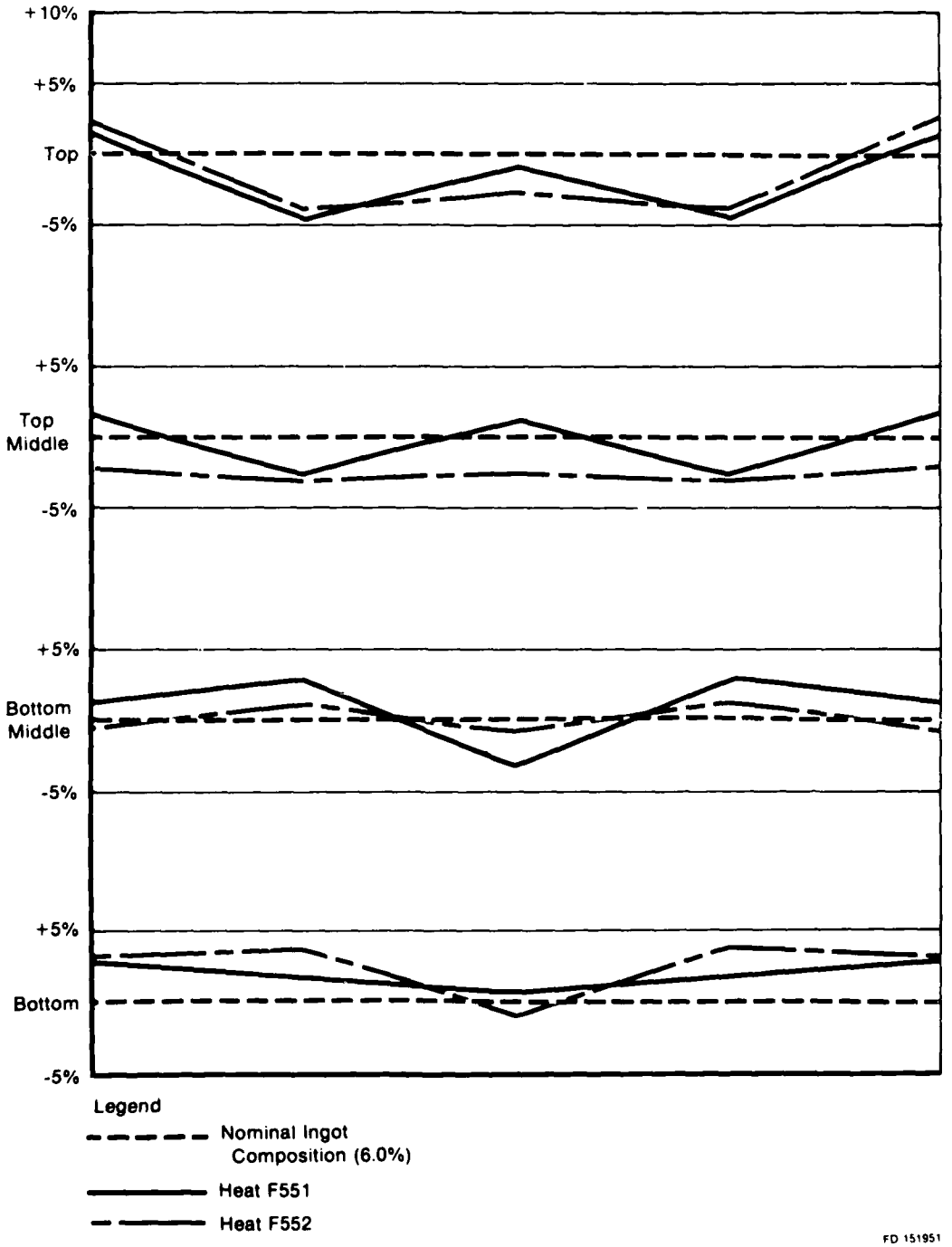


Figure 144. Variation in Aluminum Content vs Ingot Position

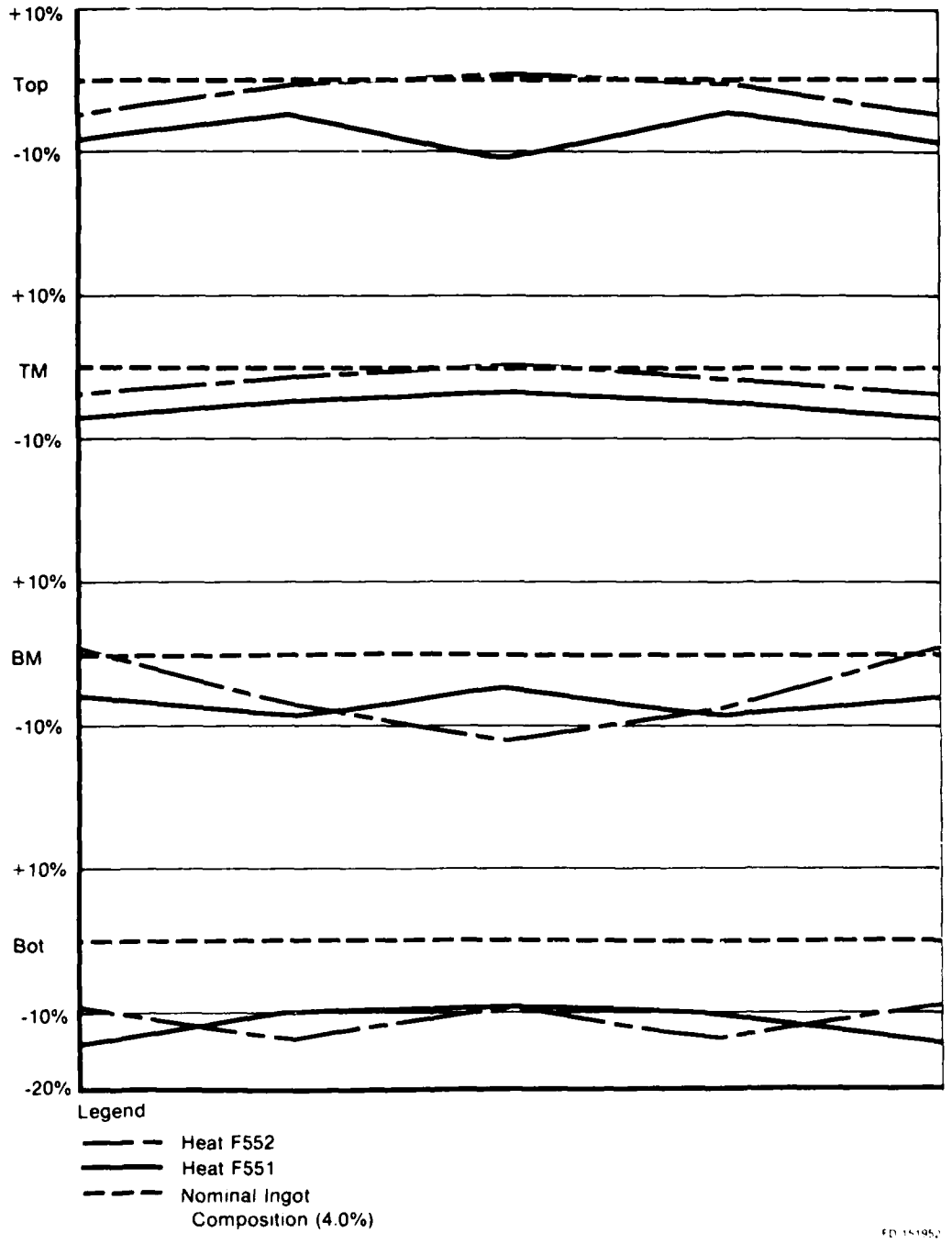


Figure 145. Variation in Vanadium Content vs Ingot Position

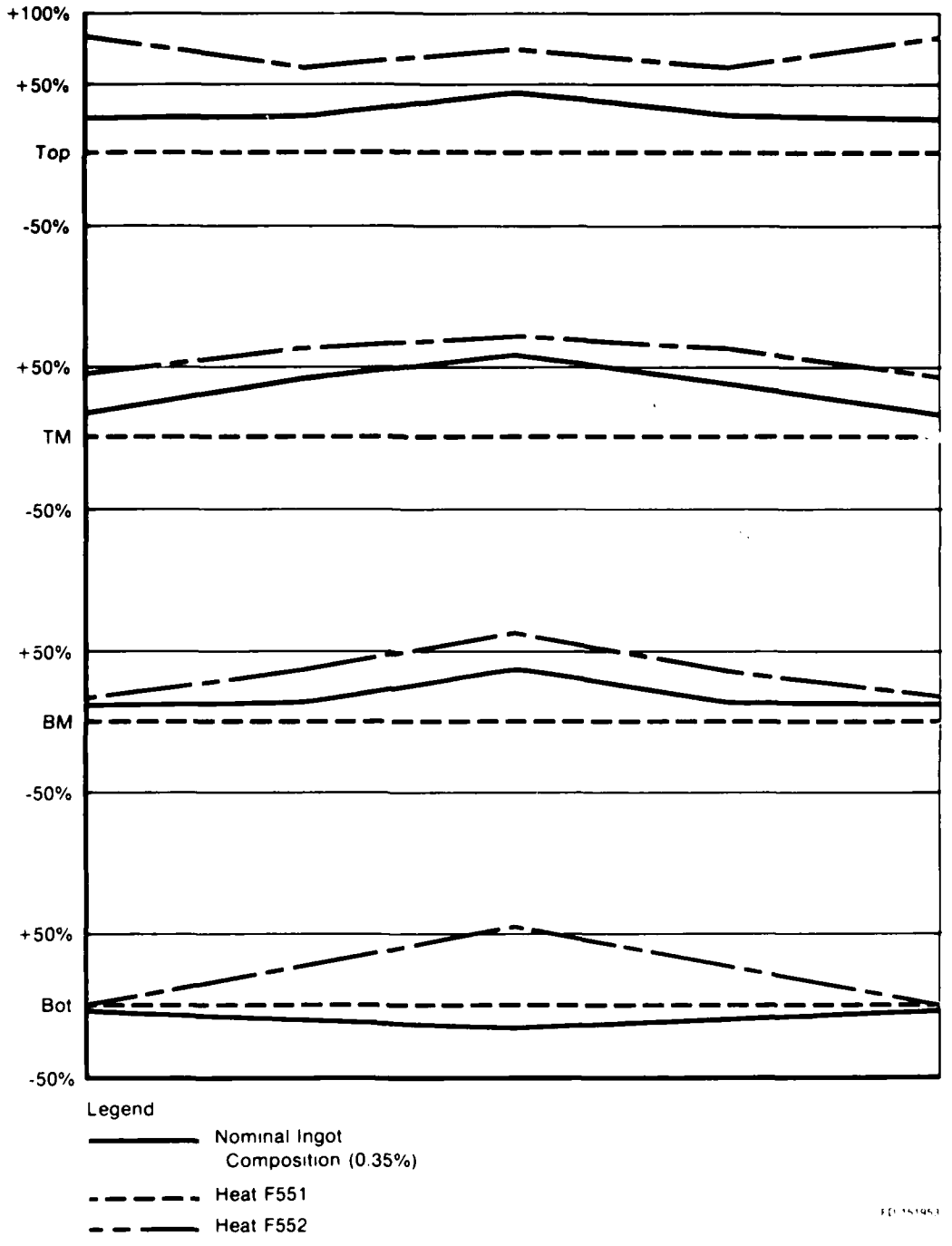


Figure 146. Variation in Composition vs Ingot Position (Fe + Mo + Mn + Cr)

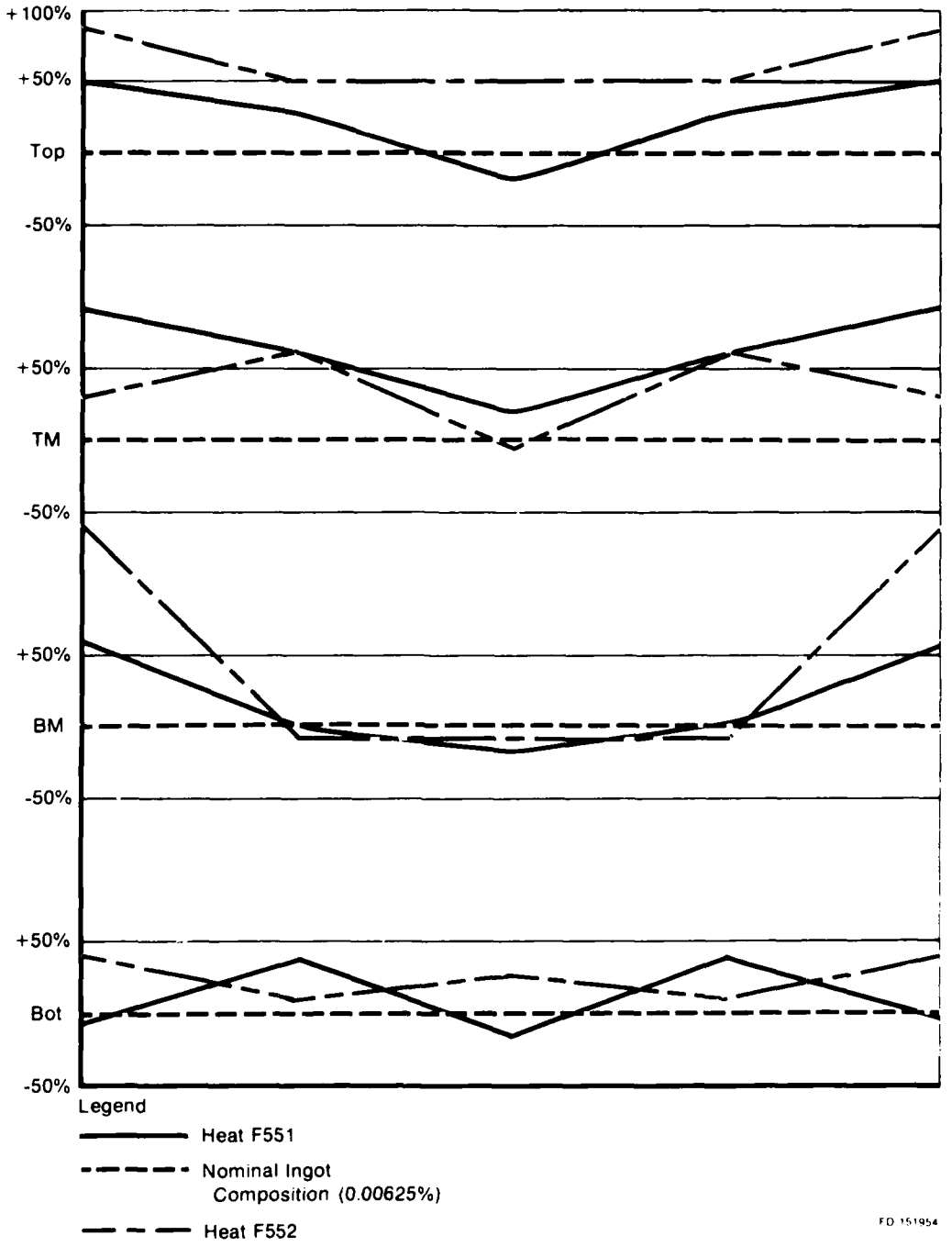


Figure 147. Variation in Hydrogen Content vs Ingot Position

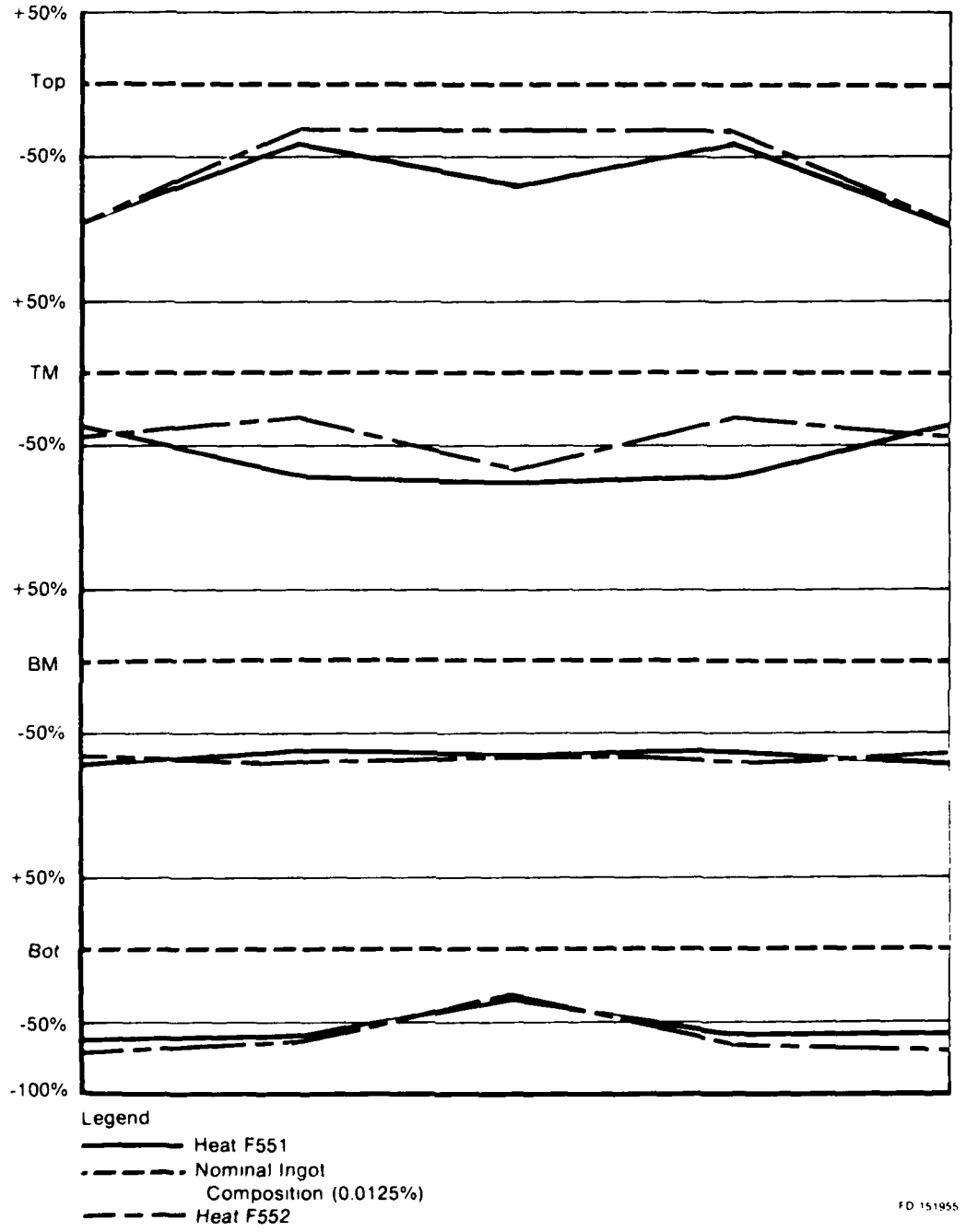


Figure 148. Variation in Nitrogen Content vs Ingot Position

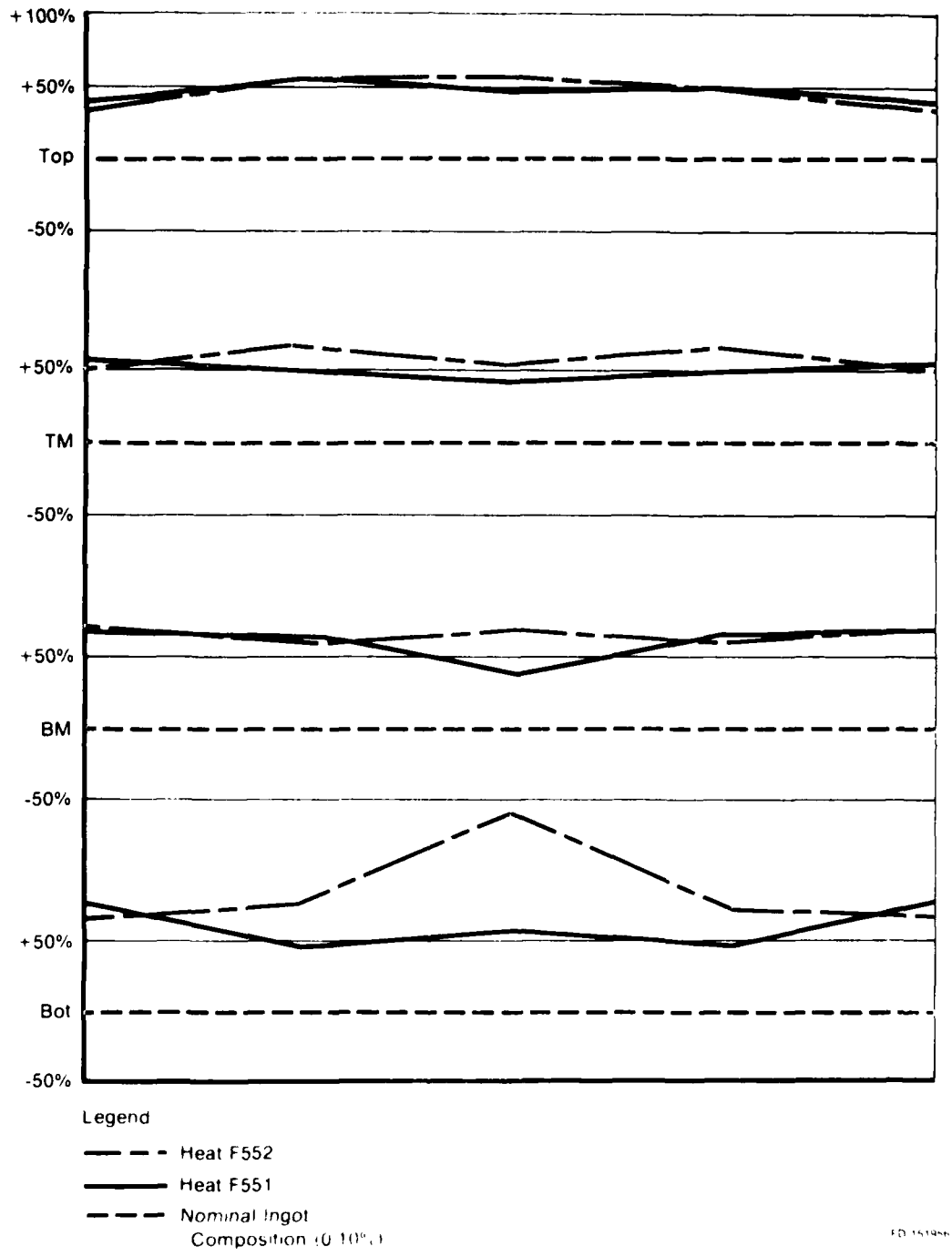


Figure 149 Variation in Oxygen Content vs Ingot Position

Separation of metallic particles from abrasive compounds, i.e., Al_2O_3, SiC, etc., was then achieved in a Moore Rapid Electromelt electric arc melting furnace at Exomet, Inc. of Greenville, Pa. This air furnace, rated at 350-lb max output load, is of conventional three-phase carbon electrode design. As the furnace interior had been relined, a "wash-heat" of iron sheet clippings was not required.

The furnace was charged with 950 lb of dried sludge product A. This charge was not sufficiently conductive to sustain the furnace arc; approximately 3 lb of iron-sheet clippings were thereupon added to the charge to initiate melting. Minor additions to the charge were made during the 3-hr melting cycle to promote fluidity for slag/metal separation (28 lb of fluorspar) and to protect the furnace liner (10 lb of lime). A voluminous quantity of offensive smoke ensued, evidently related to the higher moisture plus oil content, but melting proceeded smoothly without interruption. The fluidity additives aided in separation of the heavier metallic phase from the less-dense slag of contaminant abrasives, the slag being poured off intermittently as melting proceeded. Upon completion of the melting, plus a 15-min holding period to permit final slag/metal separation, the molten metal was poured into a sand mold to obtain a large pancake-shaped ingot.

Sludge product B, of 908-lb input of a different chemical composition, was dried and melted in a similar procedure except that this batch was electrically conductive and did not require the addition of iron sheet clippings to initiate melting. Additives used during this 3-hr melt were 31 lb of fluorspar and 10 lb of lime. Smoke evolution was again severe.

Slag contaminant that remained with the molten metal at the moment of ingot-pour was readily removed upon ingot solidification; the attached brittle slag fractured easily upon hammer impact. As a final attempt to upgrade the quality of the recovered metal product from superalloy grinding sludge, a 15-lb portion of each air melt ingot was vacuum-induction remelted and recast into cast iron ingot molds.

3. Material Balance — Phase II Conventional Drying Process

Data enabling a materials balance (input vs output) for the conventional drying of two batches of superalloy sludge (chemically different) are given in Table 90. The level of volatiles (moisture) and obnoxious combustibles (oils) was reduced about 50% and thereby enabled subsequent arc-melt processing; however, smoke evolution would not attain acceptable standards without additional absorption controls during the melting operation.

4. Material Balance — Phase II Pyrometallurgical (Melting) Process

A materials balance (input vs output) is presented in Table 91 data for the Exomet, Inc. electric arc melting process that was utilized to convert the two dried superalloy sludge products into cast ingots. A finite amount of metal was retained in the furnace lining after each melt thereby slightly reducing the accuracy of the recovery measurements, but this was deemed insignificant since the furnace was charged to capacity.

A photo of the electric arc melting facility is given in Figure 150; intermittent pouring off of slag by reverse tilting of the furnace is shown in the top view while pouring of the molten metal into the ingot mold is shown in the bottom view.

5. Chemical Analyses — Phase II Processed Superalloy Sludges

Pertinent chemical analyses of volatiles plus mositure and elements were performed during the processing steps of both batches of superalloy sludge. Specifically, analyses were made of the as-received condition, conventionally dried residue, electric arc melted ingot, and vacuum induction remelted ingot. Resultant data are presented in Tables 92 and 93.

TABLE 90
MATERIAL BALANCE OF SUPERALLOY SLUDGE DRYING PROCESS

History		
Run No.	1	2
Material	Sludge A	Sludge B
Condition	Wet	Wet
Source	Custom Tool	Custom Tool
Input		
Weight, lb	1146.0	1130.0
Oil Plus Moisture, %	21.0	21.8
Nickel, %	38.7	43.2
Nickel Weight, lb	350.4	425.7
Output		
Weight, lb	962.0	912.0
Oil Plus Moisture, %	9.1	6.2
Nickel, %	38.1	40.3
Nickel Weight, %	333.2	344.7
Recovery		
Nickel Recovery, %	95.1	81.0

TABLE 91
MATERIAL BALANCE OF ELECTRIC ARC FURNACE MELTING PROCESS

History		
Run No.	1	2
Material	Sludge A	Sludge B
Condition	Wet	Wet
Source	Custom Tool	Custom Tool
Input		
Weight, lb*	950.0	908.0
Oil Plus Moisture, %	9.1	6.2
Nickel, %	38.1	40.3
Nickel Weight, lb	329.0	343.2
Output		
Weight, lb	514.5	515.6
Nickel, %	62.0	64.1
Nickel Weight, %	319.0	330.5
Recovery		
Nickel Recovery, %	97.0	96.3

* Minor input weight discrepancies between output and these data are related to shipping losses and/or scale accuracies at different locations.

Figure 150 *Electric Arc Air Furnace for Melting of Dried Superalloy Sludge. Top View — Intermittent Pouring of Slag. Bottom View — Pouring of Superalloy Molten Metal Into Ingot Mold*

TABLE 92
CHEMICAL ANALYSES OF PHASE II SUPERALLOY SLUDGE BATCH A

	As-Received Condition	*Conventionally Dried Residue*	*Electric Arc Melted Ingot*	*Vacuum Induction Remelted Ingot*
History				
Run No. 1 Material — Sludge A Source — Custom Tool				
Analysis (W/O)				
Oil + Moisture	21.0	9.1	—	—
Ni	38.7	38.1	62.0	62.4
Co	1.22	1.05	2.70	2.65
Cr	10.5	8.6	12.5	13.0
Cu	—	0.05	0.15	0.13
Fe	6.80	4.28	8.0	9.8
Mn	—	0.07	0.21	0.21
Mo	0.6	0.52	1.30	1.29
Ti	1.0	2.07	1.46	—
V	—	—	0.04	0.04
W	—	<0.1	0.3	0.2
Al	—	0.83	0.58	0.48
Cb	—	0.4	0.5	0.4
Si	—	—	3.57	3.57
C	—	—	3.57	3.57
S	—	—	0.104	0.096

TABLE 93
CHEMICAL ANALYSES OF PHASE II SUPERALLOY SLUDGE BATCH B

	As-Received Condition	*Conventionally Dried Residue*	*Electric Arc Melted Ingot*	*Vacuum Induction Remelted Ingot*
History				
Run No. 2 Material — Sludge B Source — Custom Tool				
Analysis (W/O)				
Oil + Moisture	12.8	6.2	—	—
Ni	43.2	40.3	64.1	62.3
Co	3.50	2.45	3.35	3.32
Cr	12.8	8.3	12.4	13.8
Cu	—	0.04	0.10	0.18
Fe	3.90	3.25	7.1	8.3
Mn	—	0.06	0.15	0.15
Mo	2.2	1.10	1.30	1.63
Ti	—	1.0	0.55	0.94
V	—	—	0.02	0.03
W	—	<0.1	0.2	0.7
Al	—	0.92	0.10	0.05
Cb	—	0.4	0.2	0.4
Si	—	—	5.00	5.01
C	—	—	1.92	1.92
S	—	—	0.197	0.163

6. Analysis of Phase II Data

Based upon the chemical analysis of the electric arc melted ingot and the vacuum induction remelted ingot (Tables 92 and 93), there were no appreciable benefication attained during vacuum induction remelting. This conclusion agrees with earlier Phase I ingot data generated on developmental quantities (500-lb input weight) of grinding sludges. An inexpensive, alternate choice to vacuum induction remelting for benefication may be melt oxidation during electric arc melting; this choice is discussed later in this text.

Metal recovery is considered high in both of these Phase II runs conducted with about 1100 lb of input sludge per run; this quantity was considered adequate for the Phase II intermediate level of process scale-up. An appreciable cost-effectiveness could be realized by the 62-64 W/O nickel recovery obtained (Tables 92 and 93). However, as discussed earlier, it may not be prudent to consider these data as representative of all grindings and sludges when arriving at firm process conclusions.

Two pertinent criteria exist that influence the ultimate cost-effectiveness, namely (a) the compositional grade of the as-received grinding sludge product and (b) the quantity and type of elemental contaminants retained in the melted ingot. These criteria are discussed in the following paragraphs in more detail.

a. Grinding Sludge Input Composition

Phase II runs originated with commercially generated superalloy sludges of little economic value. No attempt was made to select the composition or quality of the input sludge except that a reasonable level of superalloy content should be present. It has been determined to be difficult, if not impractical to effect a scrap management system on grindings and sludges since industrial production, collection and handling is far less sophisticated as compared to the area of scrap turnings and solids. As the input composition of grindings and sludges can vary over a wide range, the recovered value of the melted product will vary accordingly. Input material that is "lean" in desirable nickel content will yield a reduced-level nickel ingot product with a corresponding reduction in value. Therefore, process economics will be affected by specific input compositions and must be treated accordingly.

b. Retained Contaminants in Output Ingot

The market value of the output ingot from grinding sludge superalloy scrap will be influenced by the quantity and type of elemental contaminants retained in the melted ingot. Fe is usually not of concern; however, ingots containing even a minor level of Co will have restricted use for alloying additions in the stainless steel industry although it may have usage in the cobalt-base alloy industry. Appreciable W levels are undesirable; both Co and W are not readily removable. Some elements, when present in objectionable levels, may be reduced by effecting oxidation (commonly termed "air-lancing" or "blowing") during the electric arc melting operation. It would be expected that contaminant levels of C, Si, Ti, Al, Cb, etc., would be effectively reduced by incorporating this step in the melting operation. As this further metal benefication can improve grinding sludge scrap recovery value with minimal additional process cost, it is deemed advisable to add this "air-lancing" step to future melting operations planned in Phase III.

It must be recognized that the final composition of recovered superalloy grinding sludges will always vary from lot-to-lot due to the lack of alloy segregation controls in generation of this scrap. This will influence the market acceptability of this recovered product and thereby reduce its value; however, there does appear to be a market in the stainless and alloy steel industries for this material.

SECTION XI

MODEL SCRAP HANDLING SYSTEM

A. IMPLEMENTATION OF A MODEL SCRAP HANDLING SYSTEM

1. Selection of a Company for System Implementation

Analyses of the aerospace Scrap Survey data were made with emphasis on the generated quantities of titanium alloy (Ti) open-market scrap (i.e., saleable scrap of varying quality grade that is generally marketed to scrap processors and dealers). Specific attention was given to the quantity of mixed and/or contaminated Ti alloy scrap as reported by the 29 industrial companies who responded to this portion of the survey. It was assumed that those companies generating appreciable quantities of mixed/contaminated scrap might not have an effective scrap handling system, and therefore, would be interested in the potential economic benefit of a Model Scrap Handling System.

The character and quantity of the mixed/contaminated Ti scrap were again reviewed, and 10 of the 29 responding companies were isolated as prospective candidates. Subsequently, the list was narrowed down to three companies, and these prospects were contacted to determine their potential interest in a trial implementation of the Model System. Visits were subsequently made by a joint P&WA/Subcontractor (Suisman and Blumenthal, Inc.) team to further define and assess System applicability at these companies.

A forging company with extensive, affiliated machining requirements was finally selected for implementation of the Model System. It was agreed to attempt implementation during Phase II over a 6-month trial period consistent with system development; Suisman and Blumenthal, Inc., under a subcontract to P&WA, would monitor the progress of Model System implementation, and provide appropriate guidance during the trial period.

2. Analysis of Implementor's Scrap Generation Data

Data were requested and received from the implementor regarding details of scrap generation (quantity, alloys, degree of segregation/contamination, value, etc.) over a period of the last 2 years. These data compared favorably with 1973 Scrap Survey Data. The implementor data indicated a strong effort was applied (with very successful reported results) in maintaining the segregation of titanium and superalloy scrap when in the solid configuration. In contrast, the applied effort to separate and maintain segregation of titanium and superalloy turnings had not been optimum and a high degree of unsegregation resulted for the turnings configuration. These data are not uncommon and reflect normal machine shop/fabrication industry data. In general, it appears that it is far easier to segregate and maintain the segregation of solid scrap as compared to turnings, especially when components of many alloys are being fabricated. The implementor subsequently recognized the need for segregative action on superalloy scrap turnings and effectively corrected the superalloy contamination problem.

3. Suggested Model System Guidelines

Subsequent to the review of data, the P&WA Program Manager and the Scrap Management subcontractor visited the implementor to review the pertinent aims and goals of the Model Scrap Handling System, with particular regard for cost-effectivity (see Appendix A), and to observe the features of the implementor's present system, i.e., scrap generation, handling and transportation procedures, and final disposition of aerospace scrap metal.

Based on this visit, P&WA summarized the information obtained in a letter to the implementor, comparing their existing handling system with features of the Model System, and highlighting general areas where significant cost savings appear possible. Detailed specific and practical scrap handling system procedural changes and/or modifications directly applicable to the forging and machining operation were suggested. Each of the suggested changes should improve the value of their scrap metal, and the scrap handling system implementor had the option to select any or all of the suggestions which are briefly summarized as follows:

1. Appoint a "Scrap Manager."

2. Install a formal "Scrap Recycling Department."

3. Procure necessary equipment — install platform scale, testing equipment such as thermoelectric testers and hand magnets, and barrel containers. (Note that barrels inherently reduce misgrading losses since a misgraded barrel volume is far less than a hopper volume.)

4. Segregation control at source of scrap generation — proper tagging color coding of barrels at individual machines.

5. Initiate segregation of Ti turnings by alloy type.

6. Spot-check scrap before shipping — avoid future misgrading by applying corrective segregation control.

7. Systematic reporting of segregation/misgrading to departments and individuals involved — promotes active participation.

8. Standardize scrap selling procedure — provide allowances for magnetic and moisture contents.

4. Accepted Model System Guidelines

The implementor reviewed all the guidelines of the suggested system and thereupon established acceptable guidelines that were deemed fully appropriate for his specific forging and machining operations. Pertinent guideline highlights of the acceptable system are as follows:

1. System implementation would be applied for the period 1 October 1976 through 31 March 1977.

2. System direction would be to initiate and maintain effective separation of Ti alloy scrap. (Note — superalloy separation has been effectively utilized at this company.)

3. Ti segregation (turnings and solids) would be applied to CP Ti, Ti-6Al-4V, Ti-5Al-2.5Sn, and Ti-8Al-1Mo-1V alloys.

4. Segregated collection would be effected by the procurement and installation of one yd^3 volume transport containers at individual machine areas. Appropriate color coding for alloy designation would be maintained.

5. Custom-manufactured lugger boxes of 10-yd^3 volume, with the aforementioned alloy color coding, would be fabricated for receiving scrap by fork lift transportation from the machine-area containers.

6. Lugger boxes would be sampled (for complete elemental analyses of melted samples) when full and transported by lugger truck to the scrap dealer/processors yard.

7. The dealer would perform cursory spot checking of each lugger box load (thermometric contact meter for elemental vs alloy Ti, chemical test kit for alloy elemental identification, visual inspection) before dumping acceptably segregated loads into concrete storage areas that appropriately maintain the alloy color coding.

8. The dealer would advise the implementor of segregation quality of each lugger load within 2 days of pickup for feedback of system effectiveness.

9. Combined monitoring by P&WA/Subcontractor of the lugger box analyses, reported dealer spot checks, scrap market value, etc., would enable the determination of Model System cost-effectiveness over this implementation period.

10. A firm decision on standardization of the scrap selling procedure, namely a provision for inherent magnetic and moisture content, would be resolved later.

B. MODEL SYSTEM RESULTS

1. Scrap Generator (i.e., — System Implementor) Results

A total of 23 lugger-box loads of turnings were collected by the scrap generator (i.e., system implementor) during the 6 months duration of System Implementation. The total quantities of various segregated titanium alloys collected during this period, along with average recovered values, are summarized as follows:

AMS	*Alloy*	*Color Code*	*Total Weight (lb)*	*Average Value ($/lb)*
4921	Pure Ti	Blue	11,630	0.35
4928	Ti-6Al-4V	Red	27,480	0.30-0.35
4966	Ti-5Al-2.5Sn	Yellow	7,050	0.20
4972	Ti-8Al-1Mo-1V	Orange	2,010	0.20
—	Mixed (contaminated)	—	3,140	0.10

A review of these data indicate that the scrap generator (implementor) realized a financial gain of about $8,700 during this collection period by the implementation of a Model System to segregate Ti alloy turnings. It is estimated that $5,100 of this savings was attributed to true segregation and the remainder was due to improving market conditions during this period. Complete details of collection rates for the various alloys during the implementation period are given in Table 94.

2. Scrap Dealer/Processor Results

The scrap dealer/processor conducted prompt (usually within 48 hr) chemical testing of each full lugger box of Ti alloy turnings upon delivery of the box to his plant. Multiple "grab-samples" were taken from each box to obtain random, representative sampling. This "spot-checking" (i.e., — thermometric contact meter to identify pure Ti from Ti alloy, chemical test kit for elemental alloys) was cursory in nature but it did enable quick judgment on the segregated

quality of each lugger box. This information was thereupon provided to the scrap generator (i.e., — system implementor) for his "feedback" on System effectiveness. Appropriate System remedial action could then be taken, as required, to correct System deficiencies before large volumes of mixed (contaminated) scrap were generated.

TABLE 94
SUMMARY OF TITANIUM SCRAP TURNINGS COLLECTED DURING MODEL SYSTEM IMPLEMENTATION

Month	*AMS 4921 Pure Ti (lb)*	*AMS 4928 Ti-6Al-4V (lb)*	*AMS 4966 Ti-5Al-2.5Sn (lb)*	*AMS 4972 Ti-8Al-1Mo-1V (lb)*	*Mixed Contaminated (lb)*
Oct (1976)	—	—	2,304	—	—
Nov	—	13,616	1,872	—	3,140
Dec	—	5,152	—	4,960	—
Jan (1977)	—	3,940	1,445	—	—
Feb	1,804	3,287	—	—	—
Mar	205	1,480	6,010	2,090	—
Total	2,009	27,475	11,631	7,050	3,140

A total of 502 spot-checks were conducted during the implementation period. These data, summarized in Table 95, indicate that system implementation was highly effective in maintaining scrap segregation.

TABLE 95
"SPOT-CHECK" ANALYSES DATA OF COLLECTED SCRAP DURING MODEL SYSTEM IMPLEMENTATION

Ti Alloy	*Quantity of Spot Checks Performed*	
	Acceptable (Segregated)	*Unacceptable (Contaminated)*
AMS 4921 (Pure Ti)	*	*
AMS 4928 (Ti-6Al-4V)	314	2
AMS 4966 Ti-5Al-2.5Sn)	121	1
AMS 4972 (Ti-8Al-1Mo-1V)	64	0

*Data unavailable

3. Contractor/Subcontractor Results

Representative samples of Ti turnings from all 23 lugger-box loads collected during the system implementation period were than arc melted into cast buttons. Melting was performed in purified argon using a water-cooled, thoriated tungsten nonconsumable electrode and a water-cooled copper button mold. Complete chemical and interstitial analyses were than performed on the cast buttons. Analyses data are summarized in Table 96.

TABLE 96
CHEMICAL AND INTERSTITIAL RESULTS OF COLLECTED SCRAP DURING MODEL SYSTEM IMPLEMENTATION

Lugger Box No.	Ti Alloy Composition (Intended)	Chemical Analysis (w/o)				Interstitial Analysis (w/o)		
			Specification				Specification	
		Element	Minimum	Maximum	Actual	Element	Maximum	Actual
Blue No. 0	AMS 4921 (Pure Ti)	Al	—	—	<0.005	C	0.08	0.049
		V	—	—	<0.005	O	0.40	0.35
		Sn	—	—	<0.005	N	0.05	0.013
		Mo	—	—	<0.001			
		Fe	—	0.50	0.16			
		Ni	—	—	0.01			
		Cr	—	—	0.01			
		Si	—	—	0.03			
Red No. 0	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.6	C	0.10	0.035
		V	3.50	4.50	4.1	O	0.20	0.26
		Sn	—	—	0.01	N	0.05	0.013
		Mo	—	—	0.005			
		Fe	—	0.30	0.10			
		Ni	—		0.01			
		Cr			0.01			
		Si	—		0.03			
Yellow No. 1	AMS 4966 (Ti-5Al-2.5Sn)	Al	4.00	6.00	5.2	C	0.08	0.042
		V			0.05	O	0.20	0.26
		Sn	2.00	3.00	2.2	N	0.05	0.014
		Mo			0.01			
		Fe	—	0.50	0.20			
		Ni	—		0.01			
		Cr	—	—	0.03			
		Si			0.03			
Orange No. 0	AMS 4972 (Ti-8Al-1Mo-1V)	Al	7.35	8.35	7.8	C	0.08	0.041
		V	0.75	1.25	1.11	O	0.12	0.16
		Sn	—		0.05	N	0.05	0.005
		Mo	0.75	1.25	0.99			
		Fe	—	0.30	0.03			
		Ni	—		0.01			
		Cr	—	—	0.01			
		Si	—	—	0.05			
Yellow No. 2	AMS 4966 (Ti-5Al-2.5Sn)	Al	4.00	6.00	5.2	C	0.08	0.10
		V	—	—	0.13	O	0.20	0.36
		Sn	2.00	3.00	2.5	N	0.05	0.014
		Mo			0.03			
		Fe	—	0.50	0.35			
		Ni	—		< 0.002			
		Cr	—	—	0.02			
		Si	—	—	0.03			
		Y	—	0.005	< 0.001			
		B	—	—	< 0.005			
		Mn	—	—	< 0.05			
		W	—	—	< 0.005			
		Cu	—	—	< 0.01			
		Mg	—	—	< 0.001			

TABLE 96
CHEMICAL AND INTERSTITIAL RESULTS OF COLLECTED SCRAP DURING MODEL SYSTEM IMPLEMENTATION (CONTINUED)

Lugger Box No.	Ti Alloy Composition (Intended)	Chemical Analysis (w/o)				Interstitial Analysis (w/o)		
			Specification				Specification	
		Element	Minimum	Maximum	Actual	Element	Maximum	Actual
Red No. 1	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.3	C	0.10	0.10
		V	3.50	4.50	4.4	O	0.20	0.30
		Sn	—	—	< 0.004	N	0.05	0.014
		Mo	—	—	0.02			
		Fe	—	0.30	0.13			
		Ni	—	—	< 0.002			
		Cr	—	—	< 0.005			
		Si	—	0.10	0.02			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.005			
		Mn	—	—	< 0.05			
		W	—	—	< 0.005			
		Cu	—	0.10	< 0.01			
		Mg	—	—	< 0.001			
Red No. 2	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.4	C	0.10	0.10
		V	3.50	4.50	4.2	O	0.20	0.27
		Sn	—	—	< 0.004	N	0.05	0.0087
		Mo	—	—	0.01			
		Fe	—	0.30	0.20			
		Ni	—	—	< 0.002			
		Cr	—	—	0.01			
		Si	—	0.10	0.02			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.005			
		Mn	—	—	< 0.05			
		W	—	—	< 0.005			
		Cu	—	0.10	< 0.01			
		Mg	—	—	< 0.001			
Red No. 3	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.5	C	0.10	0.10
		V	3.50	4.50	4.1	O	0.20	0.24
		Sn	—	—	0.006	N	0.05	0.0098
		Mo	—	—	0.02			
		Fe	—	0.30	0.21			
		Ni	—	—	< 0.002			
		Cr	—	—	0.01			
		Si	—	0.10	0.02			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.05			
		Mn	—	—	< 0.05			
		W	—	—	< 0.05			
		Cu	—	0.10	< 0.01			
		Mg	—	—	< 0.001			
Red No. 4	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.3	C	0.10	0.19
		V	3.50	4.50	4.1	O	0.20	0.27
		Sn	—	—	0.006	N	0.05	0.012
		Mo	—	—	0.02			
		Fe	—	0.30	0.17			
		Ni	—	—	< 0.002			
		Cr	—	—	0.01			
		Si	—	0.10	0.03			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.05			
		Mn	—	—	< 0.05			
		W	—	—	< 0.05			
		Cu	—	0.10	< 0.01			
		Mg	—	—	< 0.001			

TABLE 96
CHEMICAL AND INTERSTITIAL RESULTS OF COLLECTED SCRAP DURING MODEL SYSTEM IMPLEMENTATION (CONTINUED)

Lugger Box No.	Ti Alloy Composition (Intended)	Chemical Analysis (w/o)				Interstitial Analysis (w/o)		
			Specification				Specification	
		Element	Minimum	Maximum	Actual	Element	Maximum	Actual
Red No. 5	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.5	C	0.10	0.06
		V	3.50	4.50	3.9	O	0.20	0.27
		Sn	—	—	< 0.004	N	0.05	0.013
		Mo	—	—	0.02			
		Fe	—	0.30	0.14			
		Ni	—	—	< 0.002			
		Cr	—	—	< 0.005			
		Si	—	0.10	0.04			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.005			
		Mn	—	—	< 0.05			
		W	—	—	< 0.005			
		Cu	—	0.10	< 0.01			
		Mg	—	—	< 0.001			
Red No. 6	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.2	C	0.10	0.14
		V	3.50	4.50	4.1	O	0.20	0.27
		Sn	—	—	0.03	N	0.05	0.014
		Mo	—	—	0.02			
		Fe	—	0.30	0.18			
		Ni	—	—	< 0.002			
		Cr	—	—	0.01			
		Si	—	0.10	0.03			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.005			
		Mn	—	—	< 0.05			
		W	—	—	< 0.005			
		Cu	—	0.10	< 0.01			
		Mg	—	—	< 0.001			
Red No. 7	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.2	C	0.10	0.22
		V	3.50	4.50	4.3	O	0.20	0.35
		Sn	—	—	< 0.004	N	0.05	0.0098
		Mo	—	—	0.01			
		Fe	—	0.30	0.21			
		Ni	—	—	< 0.002			
		Cr	—	—	0.01			
		Si	—	0.10	0.04			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.005			
		Mn	—	—	< 0.05			
		W	—	—	< 0.005			
		Cu	—	0.10	< 0.01			
		Mg	—	—	< 0.001			
Red No. 8	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.3	C	0.10	0.24
		V	3.50	4.50	4.2	O	0.20	0.34
		Sn	—	—	0.006	N	0.05	0.0091
		Mo	—	—	0.02			
		Fe	—	0.30	0.25			
		Ni	—	—	< 0.002			
		Cr	—	—	0.01			
		Si	—	0.10	0.05			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.05			
		Mn	—	—	< 0.05			
		W	—	—	< 0.05			
		Cu	—	0.10	< 0.01			
		Mg	—	—	< 0.001			

TABLE 96
CHEMICAL AND INTERSTITIAL RESULTS OF COLLECTED SCRAP DURING MODEL SYSTEM IMPLEMENTATION (CONTINUED)

Lugger Box No.	Ti Alloy Composition (Intended)	Chemical Analysis (w/o)				Interstitial Analysis (w/o)		
			Specification				Specification	
		Element	Minimum	Maximum	Actual	Element	Maximum	Actual
Orange No. 1	AMS 4972 (Ti-8Al-1Mo-1V)	Al	7.30	8.50	8.0	C	0.08	0.061
		V	0.75	1.25	1.00	O	0.12	0.03
		Sn	—	0.20	< 0.02	N	0.05	0.008
		Mo	0.75	1.25	1.0			
		Fe	—	0.30	0.12			
		Ni	—	—	0.026			
		Cr	—	—	0.007			
		Si	—	0.10	0.02			
		Y	—	0.005	< 0.001			
		B	—	0.003	0.009			
		Mn	—	—	0.005			
		W	—	—	< 0.01			
		Cu	—	0.10	0.007			
		Mg	—	—	< 0.001			
Orange No. 2	AMS 4972 (Ti-8Al-1Mo-1V)	Al	7.30	8.50	8.0	C	0.08	0.074
		V	0.75	1.25	1.00	O	0.12	0.08
		Sn	—	0.20	< 0.02	N	0.05	< 0.005
		Mo	0.75	1.25	1.0			
		Fe	—	0.30	0.12			
		Ni	—	—	0.027			
		Cr	—	—	0.007			
		Si	—	0.10	0.02			
		Y	—	0.005	< 0.001			
		B	—	0.003	0.009			
		Mn	—	—	0.005			
		W	—	—	< 0.01			
		Cu	—	0.10	0.009			
		Mg	—	—	0.001			
Red No. 10	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.3	C	0.10	0.042
		V	3.50	4.50	4.1	O	0.20	0.1
		Sn	—	—	< 0.02	N	0.05	< 0.005
		Mo	—	—	< 0.02			
		Fe	—	0.30	0.27			
		Ni	—	—	0.032			
		Cr	—	—	0.012			
		Si	—	0.10	0.03			
		Y	—	0.005	< 0.001			
		B	—	0.003	0.043			
		Mn	—	—	0.008			
		W	—	—	< 0.01			
		Cu	—	0.10	0.008			
		Mg	—	—	< 0.001			
Red No. 11	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.7	C	0.10	0.44
		V	3.50	4.50	4.0	O	0.20	0.11
		Sn	—	—	< 0.02	N	0.05	< 0.0005
		Mo	—	—	< 0.02			
		Fe	—	0.30	0.23			
		Ni	—	—	0.017			
		Cr	—	—	0.004			
		Si	—	0.10	0.03			
		Y	—	0.005	< 0.001			
		B	—	0.003	0.048			
		Mn	—	—	0.009			
		W	—	—	< 0.01			
		Cu	—	0.10	0.005			
		Mg	—	—	< 0.001			

TABLE 96
CHEMICAL AND INTERSTITIAL RESULTS OF COLLECTED SCRAP DURING MODEL SYSTEM IMPLEMENTATION (CONTINUED)

Lugger Box No.	Ti Alloy Composition (Intended)	Chemical Analysis (w/o)				Interstitial Analysis (w/o)		
			Specification				Specification	
		Element	Minimum	Maximum	Actual	Element	Maximum	Actual
Red No. 12	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.9	C	0.10	0.042
		V	3.50	4.50	4.2	O	0.20	0.14
		Sn	—	—	< 0.02	N	0.05	< 0.0005
		Mo	—	—	< 0.02			
		Fe	—	0.30	0.26			
		Ni	—	—	0.021			
		Cr	—	—	0.006			
		Si	—	0.10	0.03			
		Y	—	0.005	< 0.001			
		B	—	0.003	0.038			
		Mn	—	—	0.008			
		W	—	—	< 0.01			
		Cu	—	0.10	0.007			
		Mg	—		< 0.001			
Red No. 13	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.9	C	0.10	0.041
		V	3.50	4.50	4.1	O	0.20	0.04
		Sn			< 0.02	N	0.05	< 0.0005
		Mo	—	—	< 0.02			
		Fe	—	0.30	0.23			
		Ni			0.020			
		Cr	—		0.006			
		Si	—	0.10	0.04			
		Y		0.005	< 0.001			
		B		0.003	0.037			
		Mn	—		0.010			
		W			< 0.01			
		Cu	—	0.10	0.007			
		Mg	—		< 0.001			
Red No. 14	AMS 4928 (Ti-6Al-4V)	Al	5.50	6.75	6.8	C	0.10	
		V	3.50	4.50	4.0	O	0.20	0.20
		Sn			< 0.02	N	0.05	< 0.0005
		Mo	—		< 0.02			
		Fe	—	0.30	0.16			
		Ni	—	—	0.015			
		Cr	—	—	0.003			
		Si	—	0.10	0.03			
		Y	—	0.005	< 0.001			
		B	—	0.003	0.032			
		Mn	—		0.014			
		W	—	—	< 0.01			
		Cu	—	0.10	0.010			
		Mg	—		< 0.001			
Yellow No. 3	AMS 4966 (Ti-5Al-2.5Sn)	Al	4.00	6.00	5.3	C	0.08	0.0058
		V	—		0.09	O	0.20	0.051
		Sn	2.00	3.00	2.5	N	0.05	< 0.001
		Mo	—		0.013			
		Fe		0.50	0.56			
		Ni			0.04			
		Cr			0.01			
		Si			0.09			
		Y		0.005	< 0.001			
		B			< 0.05			
		Mn			0.007			
		W			< 0.005			
		Cu			0.02			
		Mg			< 0.001			

TABLE 96
CHEMICAL AND INTERSTITIAL RESULTS OF COLLECTED SCRAP DURING MODEL SYSTEM IMPLEMENTATION (CONTINUED)

Lugger Box No.	Ti Alloy Composition (Intended)	Chemical Analysis (w/o)				Interstitial Analysis (w/o)		
			Specification				Specification	
		Element	Minimum	Maximum	Actual	Element	Maximum	Actual
Yellow No. 4	AMS 4966 (Ti-5Al-2.5Sn)	Al	4.00	6.00	5.4	C	0.08	0.041
		V	—	—	0.09	O	0.20	0.027
		Sn	2.00	3.00	2.5	N	0.05	< 0.001
		Mo	—	—	0.006			
		Fe	—	0.50	0.60			
		Ni	—	—	0.03			
		Cr	—	—	0.01			
		Si	—	—	0.03			
		Y	—	0.005	< 0.001			
		B	—	—	< 0.01			
		Mn	—	—	0.007			
		W	—	—	< 0.005			
		Cu	—	—	0.01			
		Mg	—	—	< 0.001			
Orange No. 3	AMS 4972 (Ti-8Al-1Mo-1V)	Al	7.30	8.50	7.7	C	0.08	0.067
		V	0.75	1.25	2.1	O	0.12	0.004
		Sn	—	0.20	0.02	N	0.05	< 0.001
		Mo	0.75	1.25	0.70			
		Fe	—	0.30	0.47			
		Ni	—	—	0.08			
		Cr	—	—	0.02			
		Si	—	0.10	0.09			
		Y	—	0.005	< 0.001			
		B	—	0.003	< 0.05			
		Mn	—	—	0.006			
		W	—	—	< 0.005			
		Cu	—	0.10	0.02			
		Mg	—	—	< 0.001			

C. DISCUSSION OF RESULTS

1. Segregation of Ti Turnings

An analysis of aforementioned results indicates that effective Ti scrap segregation has been initiated and maintained during this period of implementation. A radical change in segregation techniques was applied by the scrap generator: the implementing company was successful in switching from almost wholly unsegregated (mixed) Ti turnings to between 89 and 100% segregated Ti turnings. The annual percentage of segregated Ti alloy turnings for this company over the last few years can be expressed as follows:

Year	Segregated Ti Turnings	
	lb	%
1974	81,211	33
1975	0	0
1976 (9 mos)*	0	0
1976 (3 mos)*	27,904	89
1977 (3 mos)*	20,274	100

*Implementation of Model Scrap Handling System

A direct comparison of two 6-month collection periods shows the dramatic improvement in scrap segregation attained with system implementation; comparison data are as follows:

	1975 to 1976 (Prior to Model System)		*1976 to 1977 (Model System)*	
Month	*lb Segregated*	*lb Mixed*	*lb Segregated*	*lb Mixed*
Oct.	0	30,768	2,304	0
Nov.	0	0	15,488	3,140
Dec.	0	28,056	10,112	0
Jan.	0	11,612	5,385	0
Feb.	0	27,670	5,007	0
Mar.	0	7,040	9,882	0

Further corroboration of the effectiveness of the implementation was the improvement in price levels received by the implementing company. In the 9 months prior to implementation, the company received an unweighted (wet) average scrap value of 8.7¢/lb. During the six month implementation period, it received an unweighted average of 27.3¢/lb. It is correct that the general market for Ti turnings improved during that time; we estimate that this market increase accounted for approximately 8.0¢/lb of the improved average price. Therefore, 11.3¢/lb of the increase was due to the improved segregation system. Even this increase seems conservative, based on increased real values of the segregated Ti turnings in the market place.

In summary, the implementing company set out to segregate its Ti turnings into segregated grades and this objective was accomplished.

2. Chemical and Physical Analysis of Scrap

a. Basic Chemistry

Table 96 summarizes the chemistry/interstitial data of all segregated lugger boxes that were collected during the system implementation. In general, these data meet the nominal chemical specifications of each specific titanium alloy. Chemical deviations, as specifically determined, are discussed individually as follows.

b. Boron

Analyses data indicate that boron levels may occasionally exceed specification limits. In reality, boron chemistry was determined by both emission spectroscopy and ultraviolet spectrophotometry. The latter procedure quantitatively is dependent upon standards and has generated the slightly higher boron values during this program. There is reason to believe that variations in chemical procedure may account for these minor boron discrepancies.

c. Nickel-Chrome-Iron

Paradoxially, these levels appear to be abnormally low, that is, "too-well" controlled. Past experience at aerospace manufacturing companies indicates that these elements are difficult to control because many alloys containing these elements are machined in the same areas and often at the same times as Ti alloys. Such was the case at the implementing company.

While admittedly very desirable, the cause of these "too-low" levels is not apparent. The implementing company may simply have produced an "angel effect," caused inadvertently by the experiment itself. Concern does exist if the results are reproducible on a day-to-day basis. The system of sampling may also have contributed to these unexpected results of low Ni/Cr.

d. Tin

As in the cases of nickel, chrome and iron, these data of low Sn levels also appear overly favorable. During the implementation period, 11,631 lb of AMS 4966 were segregated which was approximately 24% of the total segregated. Long experience has shown that some intermixture between Ti grades can be expected. AMS 4966 has 2½% tin in its chemistry, so that even a minor intermixture would cause the tin to move up beyond the virtually tin-free levels reported in the non-tin-bearing grades.

e. Dense Inclusions

Dense, insoluble inclusions are a very serious problem that are intolerable in critical aerospace applications. A specific problem is the tungsten carbide tool bit that fractures during titanium machining operations and falls into the lathe turnings. Subsequent melting of the turnings during scrap consolidation will not appreciably dissolve the tool bit incusion due to its extremely high melting point and an inclusion is the result. Dense inclusions are tolerable and insignificant in steel or aluminum industries where critical fatigue considerations are not paramount. No evidence of tungsten contamination or inclusions were detected in this evaluation of the Model System.

f. Moisture

No report was received regarding the moisture content of the turnings. Since most titanium is machined with a coolant, the adherence of some cutting fluid to the chips would unquestionably be challenged by an aerospace melter seeking to use the chips for rotating-grade application. This problem can be resolved by a specialized aerospace processor, but it has not been resolved (nor was it planned to be resolved) in this implementation.

g. Physical

These turnings were in a bushy form. Until they were reduced to a uniform size, they would not be suitable for aerospace recycling. In addition, the ability to sample these turnings for a random sample would be greatly inhibited if not impossible in the present form.

h. Yttrium

Trace contamination of yttrium (specification limit of 0.005%) is very difficult to consistently control when some commercial titanium alloys contain 0.04Y as an alloying element and past history has not strongly avoided the mixing of Y/non-Y grades.

Although Y concentrations determined during this work were well within specification, it is likely in future scrap processing that Y concentrations may exceed specification levels. Yttrium presence, when found, will need to be evaluated for overall material performance properties.

i. Oxygen

Oxygen level (0.24 to 0.36% range) consistently exceeded the AMS 4928 specification limit of 0.20%. This is a common occurrence with Ti scrap turnings. Oxygen contamination of turnings is caused by frictional "burning" during lathing operations and/or surface scaling during prior heat treat operations.

Oxygen levels in Ti melted for aerospace or nonaerospace end-use mill products are well defined and strictly controlled. Contamination levels in this work can readily be reduced to acceptable specification range by blending of processed turnings wih virgin sponge Ti during consumable electrode fabrication.

j. Carbon

Carbon level (0.1 to 0.24%) often exceeded the AMS 4928 specification limit of 0.1%. This is probably related to extraneous dirt and debris retained in the scrap turnings which would be removed during subsequent fluidized bed or ferrofluid processing operations. The aforementioned blending with virgin sponge Ti would also alleviate this excessive carbon content.

3. Analysis of Model System Implementation

a. Response of the Implementing Company

The response to the most critical parts of the Model System by the implementing company were, on the whole, quite good.

A model system for good aerospace recycling requires first and foremost the complete backing of top management. In this instance, the Manager of Sales, the Chief Engineer and the Vice-President and General Manager all supported the installation of a Model System for titanium turnings recycling.

Secondly, a determined, interested Scrap Manager is necessary. This area was also supported, but unfortunately, company priorities and work-force limitations would not permit appointment of a full-time Scrap Manager. Responsibilities of this function had to be distributed among several people which thereby tends to dilute the overall importance of scrap management in maintaining the full-time or part-time overall control in the plant of all aerospace (and other) scrap.

The final requirement was that a qualified scrap firm be closely integrated into the program. Fortunately, a local and quite skilled firm was already handling the plant's scrap and was available to assist in the Model System.

b. Suggested vs Accepted Guidelines for Model System

The Model System, as originally outlined for this Air Force study, was tailored to fit the needs of the implementing plant and eight recommendations were made. The following were the recommendations and the implementation:

1. Appoint a "Scrap Salvage Manager." As discussed previously, company priorities and work-force limitations would not permit appointment of a full-time Scrap Manager. Functional responsibilities were distributed among several people.

2. Set up a formal "Scrap Salvage Department." This was not fully accomplished; rather, the implementation centered on the segregation of Ti turnings.

3. Buy and/or install the necessary equipment. Central to this recommendation was the concept that a scrap salvage area must be established and a covered, but perhaps open, shed in the plant's yard was the least costly method. Further, it was recommended that the Ti scrap be accumulated in steel drums or other small volume containers, so that if some mixture did take place, the few drums of contaminated material could be "quarantined," instead of contaminating a much larger lot.

Neither of these recommendations were fully implemented. Instead, the Ti turnings were placed at the machines into one cubic yard self-dumping hoppers which were color coded for the four basic Ti alloys. The hoppers were transported by forklift truck to larger roll-off steel containers which were placed in the plant's yard; each roll-off container was color coded in the same way. After a 10 cubic yard container was filled, it was removed by the scrap dealer and transported to the dealer's yard where it was checked for quality and sampled.

4. Closer control at the source of generation. This recommendation was clearly accepted and implemented. The implementing company, though its inplant manager, made clear to each foreman and in turn to the machine operators the need for segregating Ti turnings. Of all the recommendations, this one is the absolute minimum for a successful aerospace scrap program.

5. Start a system for segregating titanium turnings. Systems for accomplishing this have been mentioned above.

6. Spot check scrap before it is shipped. The first quality check actually took place at the dealer's yard.

7. Install systems to reinforce correct segregation and to correct misgradings. To some extent these systems were implemented. The dealer was instructed to report back misgradings and contamination promptly and the management was organized to search out sources of contamination. Monthly reports were issued which would highlight contamination.

8. Simplify method of sale, by initiating allowances for magnetics and moisture. The implementing plant correctly predicted that the segregation system would sharply cut down or eliminate magnetics; the first part of this recommendation was found not to be necessary. The question of allowance for moistures was deferred until the program's conclusion.

SECTION XII

SCRAP RECLAMATION TECHNOLOGY

A. TITANIUM RECLAMATION PROCESS

1. Background Information

The titanium reclamation process is aimed at the economical recycling of titanium alloy turnings back into the aerospace material cycle. The reclamation process begins with material which has been collected under the Model Scrap Handling System guidelines.

In Phase I, two separation systems (AVCO and Frankel) were evaluated. The Frankel system was chosen for further Phase II titanium reclamation process analysis. In Phase II, titanium ingots were converted to 9-in. diameter forging billets. For ease of handling, each ingot was cut into thirds to produce three 10-foot lengths of barstock from each of the heats F551 and F552.

2. Disk Evaluation

Each of the four disks was evaluated metallographically at six locations. This was accomplished by polishing a spot about 1 in. in diameter at the desired location followed by etching and replication of the surface with acetate tape. The resulting micrographs and their locations are shown in Figures 140 through 143. In all cases, the etchant used was Kroll's reagent.

A full cutup of one disk from each master heat was performed for the purpose of mechanical property determination. Figures 151 and 152 depict the cut-up layout of the disks that were tested. The types of tests being performed, the amount of samples chosen for each test, and the location on the disk of the samples is delineated in Table 97. Preliminary mechanical property tests were conducted by Teledyne-ALLVAC on specimens machined from disk bore integral test rings.

3. Mechanical Property Test Results

These properties exceed the AMS 4928 specification requirements and appear typical for forgings of this section size. The data are presented in Table 98. The uniformity of properties suggests no deleterious effects due to material reclamation on this admittedly small sample size.

Of the Phase III mechanical property tests, the majority of the results indicate no debit in the properties of the reclaimed Ti-6Al-4V fan disk forgings when compared to those of AMS 4928. The results of the tests are discussed individually. The test conditions, chosen early in the program, reflect the Ti-6Al-4V specifications and available baseline data.

Two stress ratios were tested in the crack propagation tests. The test conditions chosen were room temperature and a frequency of 20 Hz. The crack growth rates for the reclaimed titanium were similar to those of the PWA 1215 (AMS 4928) material for both stress ratios over the entire ranges of data. The crack propagation data for the reclaimed material were generated using compact specimens with 0.6-in. nominal thickness. The data for the PWA 1215 specimens were obtained from 0.3-in. compact specimens. Figures 153 and 154 depict the results of the tests.

The notched stress-rupture baseline data consists only of the AMS 4928 specifications, which call for a 5-hr min at room temperature and a stress level of 170 ksi. All the specimens exceeded this specification. Their results are tabulated in Table 99.

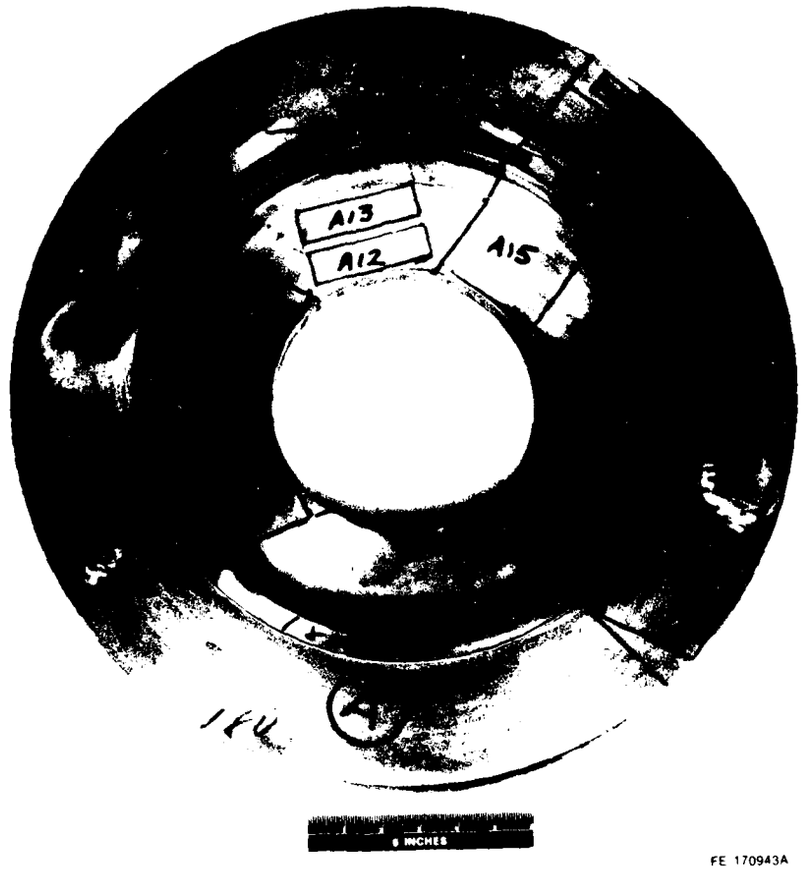


Figure 151. Disk Cut-Up Layout of Disk BBA-2001, Heat F551

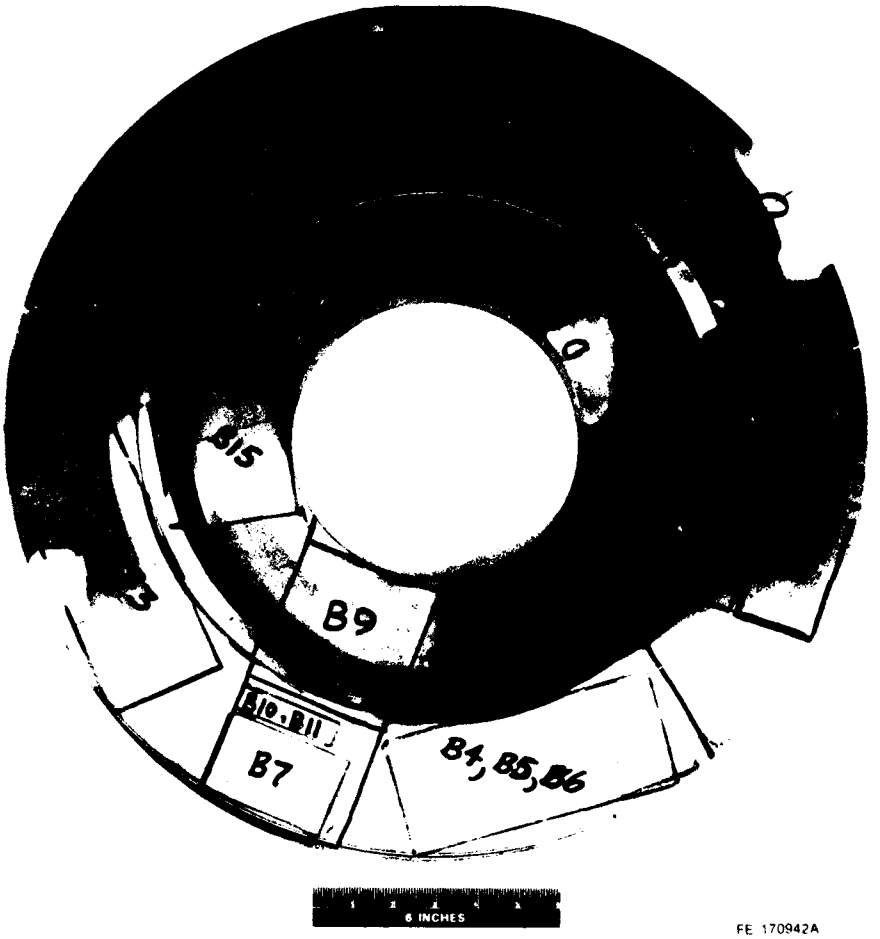


Figure 152. Disk Cut-Up Layout of Disk BCB-2001, Heat F552

TABLE 97
MECHANICAL PROPERTY TESTS

Specimen	Test	Quantity		Location
		Disk BBA2001	Disk BCB2001	
1-6	Full Bolthole	6	6	Rim
7-8	One-Half Bolthole	2	2	Rim
9	Crack Propagation	1	1	Bore
10-11	Notched Stress Rupture	2	2	Rim
12-14	Tensile	3	3	2 Bore/1 Rim
15-16	Fracture Toughness	2	1	Bore
17-22	Sonntag (K_t = 1)	6	0	3 Bore/3 Rim
23-28	Sonntag (K_t = 2)	6	0	3 Bore/3 Rim

TABLE 98
INTEGRAL TEST RING PROPERTIES

INTEGRAL TEST RING PROPERTIES
TF33 2ND-STAGE FAN DISK 2SZ-482402
TELEDYNE-ALLVAC
HEAT NOS. F551 AND F552
SPECIFICATION: AMS 4928

Room Temperature Tensile Properties:

P&WA Identity	Heat No.	Yield (psi) Strength	Ultimate Strength (psi)	El. (%)	RA. (%)
CBCB. 2001	F551	139,500	151,600	16	52
CBCB. 2002	F551	142,600	154,800	16	48
CBBB. 2001	F552	139,100	152,000	16	53
CBBA. 2002	F552	141,600	154,000	15	51
Specification:		120,000	130,000	10	25

Room Temperature Notched Stress Rupture:

P&WA Identity	Heat No.	Load (psi)	Hours at Load	
CBCB. 2001	F551	170,000	5.0 hr	No Fracture
CBBA. 2001	F552	170,000	5.0 hr	No Fracture
Specification:		170,000	5.0 hr	No Fracture

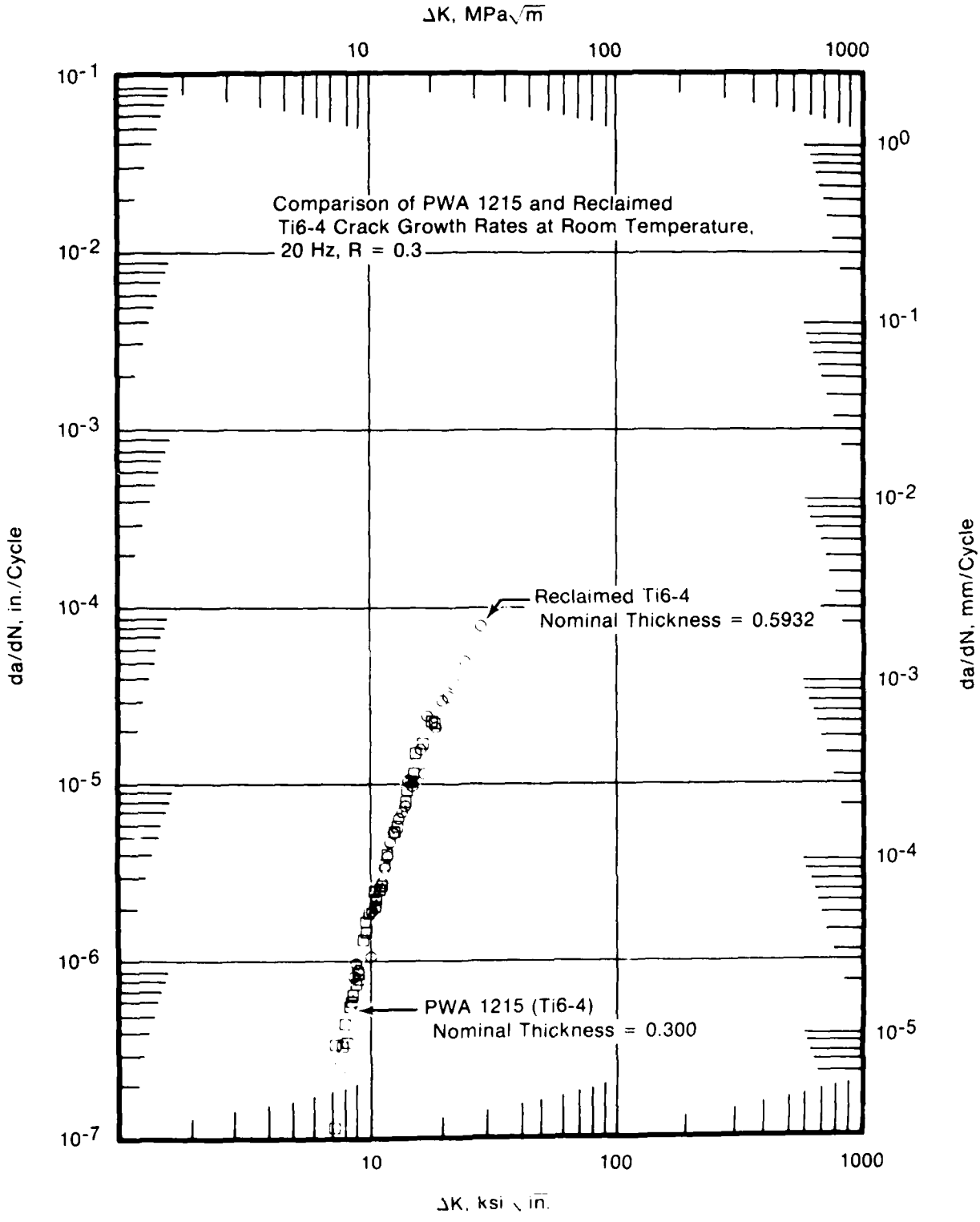


Figure 153. Reclaimed Ti-6Al-4V Crack Propagation Results at Room Temperature, 20 Hz, R = 0.3

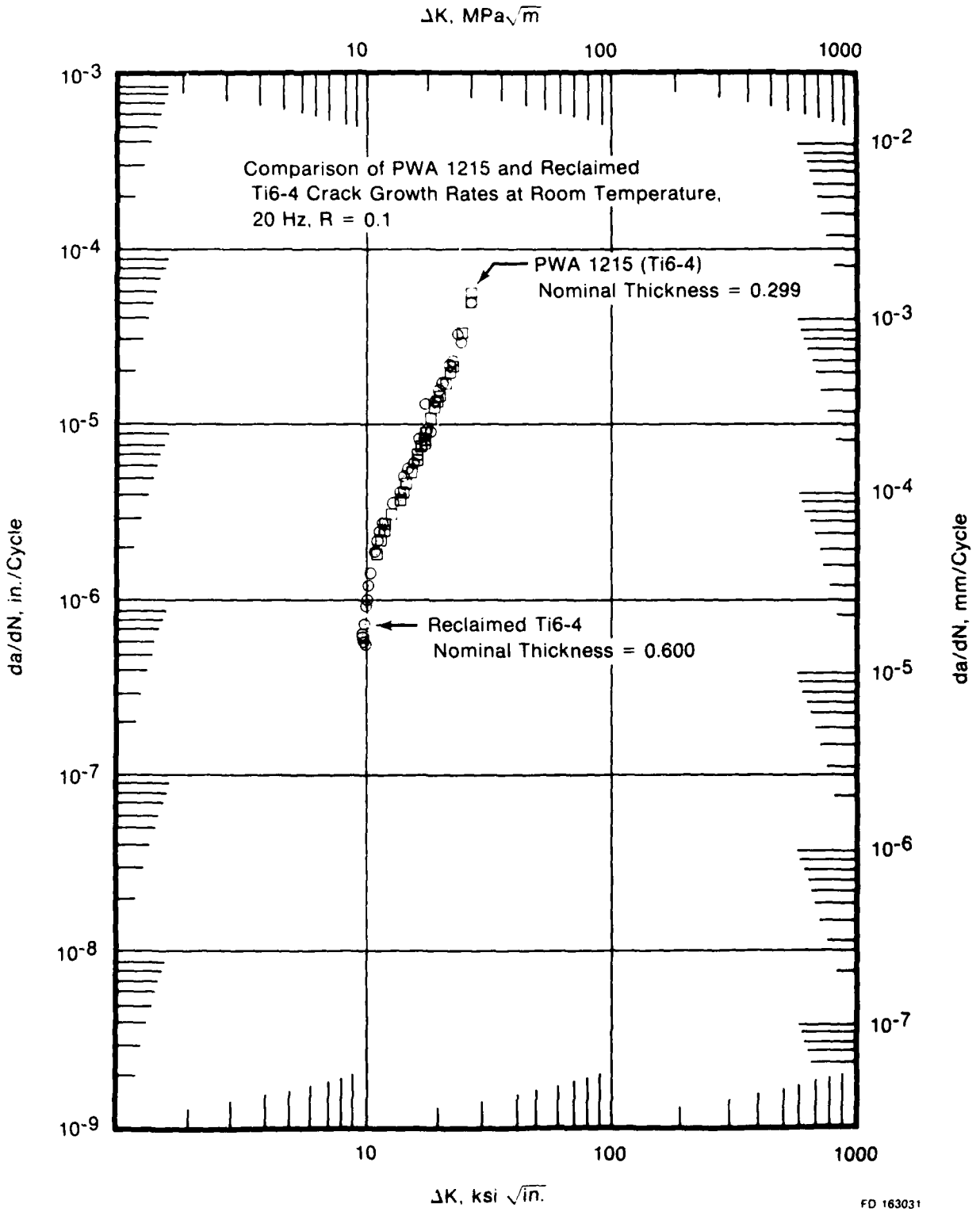


Figure 154. Reclaimed Ti-6Al-4V Crack Propagation Results at Room Temperature, 20 Hz, R = 0.1

TABLE 99
RECLAIMED Ti-6Al-4V DISK NOTCHED STRESS RUPTURE SPECIMENS AT ROOM TEMPERATURE AND 170.0 ksi STRESS

Specimen[1]	*Time at Stress (hr)*
A10	2270.8
A11	2693.4
B10	2393.6
B11	2409.7

[1] "A" indicates Disk BBA2001, Heat F551.
"B" indicates Disk BCB2001, Heat F552.

The tensile tests, catalogued in Table 100, show no debit in properties of the reclaimed Ti-6Al-4V when compared to those of AMS 4928. The ductility, in fact, exceeded the AMS 4928 values. The tests were conducted at room temperature.

The fracture toughness tests had no baseline data for guidelines. One specification quoted a minimum of 15 ksi $\sqrt{\text{in.}}$ for Ti-6Al-4V. All of the samples exceeded this value. The results are listed in Table 101.

The Sonntag tests (LCF) showed a slight debit in reclaimed material properties when compared to some experimental work done on AMS 4928 by Pratt & Whitney Aircraft. The test conditions were room temperature and a stress of 170.0 ksi. Two stress concentrations were studied; $K_T = 1.0$ and $K_T = 2.16$. No data was available for comparison in the $K_T = 2.16$ tests, but the values appeared slightly low. The results are depicted in Figure 155 and described in Table 102.

Four specimens, two from each heat, were subjected to half bolthole fatigue testing. The results, tabulated in Table 103, show that all but one of the specimens greatly surpassed the available baseline data listed in Table 104 for a specimen that was surface finished, reamed, and Sutton-Barreled. These results suggest that the reclaimed titanium meets the specifications overall.

Finally, the full bolthole test results compared very favorably with the limited test data available. None of the specimens failed and only 3 of the 12 specimens showed any cracking at 20,000 cycles. The test conditions and results are shown in Table 105.

4. Titanium Reclamation Conclusions

Both the metallographic evaluation and the mechanical property determination proved the reclamation of titanium alloy turnings to be feasible. AMS 4928 mechanical property specifications were met or exceeded in all cases. It should be noted that the LCF results were lower but are not covered by the AMS 4928 specification.

TABLE 100
RECLAIMED Ti-6Al-4V DISK TENSILE SPECIMENS AT ROOM TEMPERATURE

Specimen	*Disk*	*Yield Strength (ksi)*	*Ultimate Strength (ksi)*	*Elongation (%)*	*Reduction in Area (%)*
12A	BBA2001	136.3	144.8	15.5	44.4
13A	BBA2001	133.9	142.6	15.5	44.4
14A	BBA2001	137.1	146.2	15.5	42.0
12B	BCB2001	130.7	146.0	15.5	41.6
13B	BCB2001	138.0	146.1	16.5	45.7
14B	BCB2001	132.3	142.3	16.0	42.0
Mean:					
Disk A		135.7	144.5	15.5	43.6
Disk B		133.6	144.8	16.0	43.1
AMS 4928		134.0	146.5	12.0	*
97.5% Lower Bound:					
Disk A		126.2	138.1	13.8	32.3
Disk B		124.1	138.3	14.3	31.8
AMS 4928		123.0	135.5	9.0	*

*Values for Reduction in Area Were Not Available.

TABLE 101
RECLAIMED Ti-6Al-4V DISK FRACTURE TOUGHNESS SPECIMEN TEST AT ROOM TEMPERATURE

Specimen[1]	K_{Ic} *(ksi $\sqrt{in.}$)*
A15	65.9
A16	70.5
B15	51.1

[1]"A" indicates Disk BBA2001, Heat F551.
"B" indicates Disk BCB2001, Heat F552.

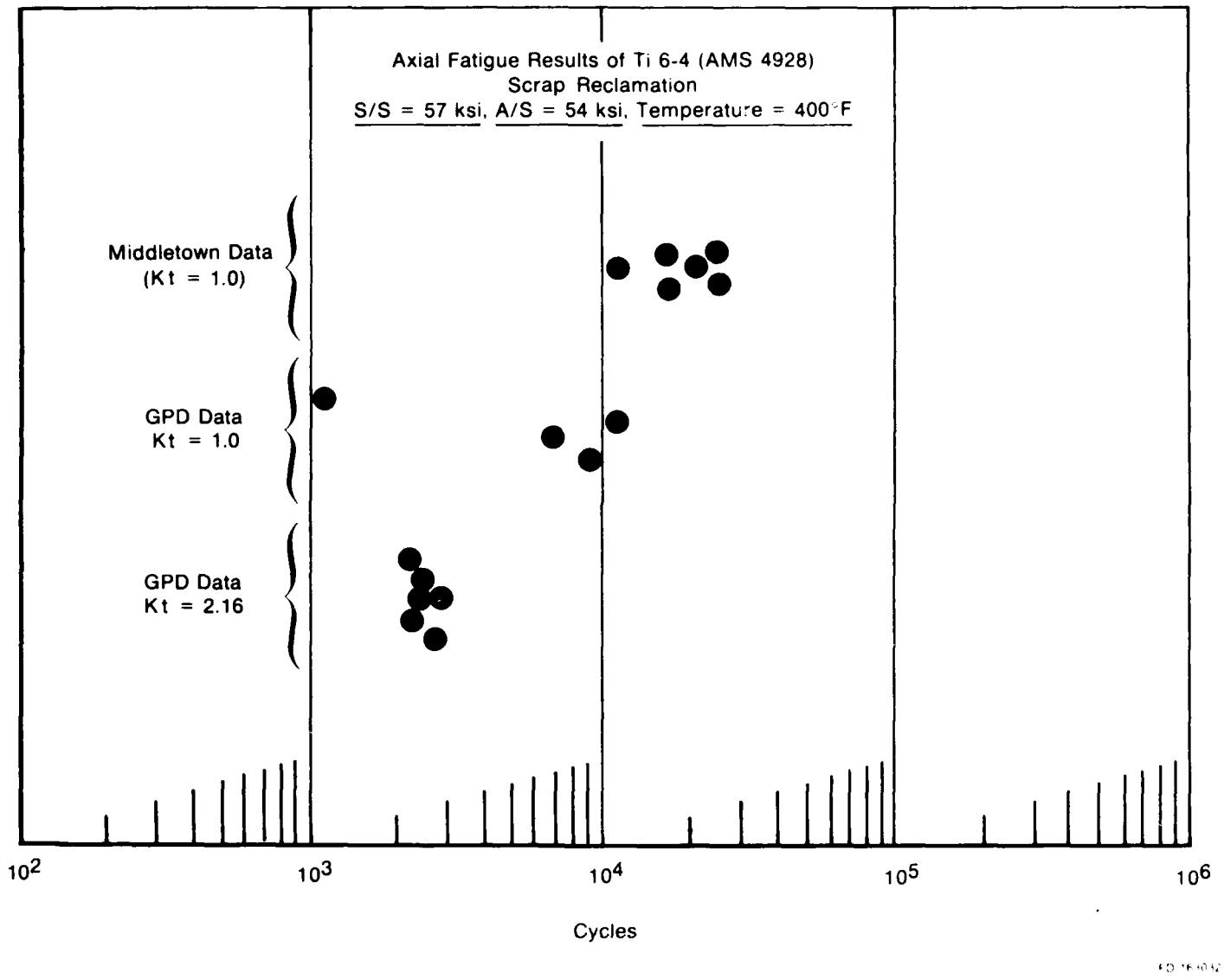


Figure 155. Reclaimed Ti-6Al-4V Disk Sonntag Test Specimens

TABLE 102
RECLAIMED Ti-6Al-4V DISK SONNTAG SPECIMEN TEST AT 400°F, 57 ksi, ALTERNATING STRESS OF 54 ksi

Specimen[1]	*K*	*1st Crack Indication (Cycles)*	*Failure (Cycles)*	*Remarks*
A17	1.0	—	—	Failed on set-up
A18	1.0	1.0×10^8	1.07×10^8	Many cracks 360 deg failed in gage area
A19	1.0	8.0×10^8	9.86×10^8	Many cracks 360 deg
A20	1.0	—	—	Tensiled
A21	1.0	5.0×10^8	6.8×10^8	360 deg crack indications
A22	1.0	—	1.08×10^8	Failed in gage during run
A23	2.16	2.0×10^8	2.9×10^8	360 deg crack indications
A24	2.16	2.0×10^8	2.75×10^8	180 deg crack indications
A25	2.16	2.0×10^8	2.48×10^8	180 deg crack indications
A26	2.16	2.0×10^8	2.39×10^8	180 deg crack indications
A27	2.16	2.0×10^8	2.5×10^8	360 deg crack indications
A28	2.16	2.0×10^8	2.3×10^8	360 deg crack indications

[1] "A" indicates Disk BBA 2001, Heat F551.

TABLE 103
RECLAIMED Ti-6Al-4V DISK HALF BOLTHOLE SPECIMEN TEST AT ROOM TEMPERATURE, 0.9% AT ROOM TEMPERATURE, 0.9% δ STRAIN, WITH STANDARD INSPECTION

	Cycles to Crack		
Specimen[1]	*Pinpoint Crack*	*1/64 in. Crack*	*1/32 in. Crack*
A7	None at 100,000	102,000	103,000
A8	5,000	8,000	9,000
B7	None at 150,000	—	—
B8	22,000	26,000	32,000

[1] "A" Indicates Disk BBA2001, Heat F551
"B" Indicates Disk BCB2001, Heat F552

TABLE 104
AMS 4928 BASELINE DATA FOR HALF BOLTHOLE TESTS AT ROOM TEMPERATURE AND 0.9% δ STRAIN

Condition of the Hole	*Cycles to Pinpoint Crack*		
	Minimum	*Average*	*Maximum*
As Machined	12,000	33,000	45,000
Surface Finished	8,000	27,500	47,000
Surface Finished and Reemed	8,000	12,000	24,000
Surface Finished, Reemed and Sutton-Barreled	8,000	10,000	11,000

TABLE 105
RECLAIMED Ti-6Al-4V DISK FULL BOLTHOLE SPECIMEN TEST WITH A MAXIMUM STRESS OF 56 ksi, A MINIMUM STRESS OF 2.8 ksi, K_T = 2.5, R = 0.05, AND A DWELL TIME OF 18 MINUTES AT MAXIMUM LOAD AND 2 MINUTES AT MINIMUM LOAD

Specimen[1]	*Test Temperature (°F)*	*Cycles to*	
		Inspection Prior to Crack	*Crack*[2]
A1	Room Temperature	20,000	
A2	Room Temperature	20,000	
B1	Room Temperature	20,000	
B2	Room Temperature	20,000	
A3	600		20,000 (0.010 in.)
A4	300	20,000	
B3	600		20,000 (0.023 in.)
B4	300	20,000	
A5	300		20,000 (0.025 in.)
A6	600	20,000	
B5	300	20,000	
B6	600	20,000	

[1] "A" Indicates Disk BBA2001, Heat F551
"B" Indicates Disk BCB2001, Heat F552

[2] Crack Size Indicated in Parentheses. None of the Specimens Failed.

B. SUPERALLOY GRINDINGS SLUDGE RECLAMATION PROCESS

1. Background Information

Processing of developmental quantities (Phase I) and intermediate scale-up quantities (Phase II) of grinding sludge scrap has given evidence of potential cost-effectiveness of this recovery. Phase III efforts evaluated the process repeatability while scaling up to a production-quantity melt run. Specifically, it was planned to process 3 tons of superalloy sludge through conventional drying and electric arc air melting operations. The latter operation was performed in a production furnace with "air-lance" capabilities. This furnace is equipped with a water-scrubbing device in the exhaust stack to remove objectionable soot and attain acceptable EPA standards for smoke control.

2. Process Direction

a. Cleaning

All past melting experience with superalloy grindings has substantiated the fact that voluminous quantities of smoke and steam are evolved during melting. The smoke is mainly due to retained oil and moisture in the furnace charge (between 6 and 9% by weight). This is somewhat difficult to remove mechanically due to the fine particle size of the grinding swarf leading to a large surface area per unit mass and enhanced capillary action.

The motivation to remove this fluid arises from the necessity of meeting EPA industrial emission standards. To do so will require melting facilities with associated exhaust scrubbers. This will add to the capital equipment cost, and therefore, result in higher melting costs for the sludge material. Also, the last superalloy grindings which were melted produced a product containing about 2% carbon by weight. Such a concentration limits the utility of the ingot product, and its removal lengthens melt cycle time which also adds to the melting cost.

b. Melting

The objective of the melting operations was twofold: first, to document the scale-up of electric furnace melting of superalloy grinding swarf, and second, to assess the effect of melt oxidation on the undesirable elements Mn, Si, S, O, and C.

Scale-up data includes a cycle time of approximately 4 hr. No nonstandard procedures were used to process the unique sludge composition. Factors such as refractory life and the like were naturally unobtainable from one cycle.

The melt cycle parameters were based on past reclamation trials together with prior equipment experience with lanced melts. High melt temperatures were desirable from the standpoint of low equilibrium carbon concentrations due to the increase in entropy associated with the reaction:

$$2C + O_2 \rightarrow 2CO$$

This entropy increase results in the reaction having an increasingly negative free energy of formation with increasing temperature which results in the low equilibrium carbon concentration in the melt.

It can be expected that high melt temperatures have a negative effect on lining life and power consumption. Also, the removal of Si, Mn and S by oxidation involves a decrease in the free energy change for the following reactions with an increase in temperature:

$$Si + O_2 \Rightarrow SiO_2$$
$$2Mn + O_2 \Rightarrow 2MnO$$
$$S + O_2 \Rightarrow SO_2$$

This results in higher equilibrium concentrations of these elements in the melt with increases in melt temperature for oxygen lancing operations.

3. Processing

Three tons of Ni superalloy grinding sludge were purchased from VAC Air Alloys Corporation and sent to Air Products and Chemicals, Inc. for refinement. The grinding sludge was processed by Air Products and Chemicals, Inc. in an electric arc air melting furnace with the use of an air lance. The furnace was charged with approximately 6000 lb of dried nickel sludge. As the material heated, additions were made to the charge. These additions included fluorspar, to enhance fluidity for slag/metal separation, and lime, to protect the furnace liner and help desulphurize the sludge. The 4-hr melt cycle consisted of desulphurizing the charge and removing the unwanted abrasives and excess material until the remaining metal met the required melt cycle parameters.

During the melting, periodic samples were taken from the charge to determine the chemistry of the melt. The fluidity additives aided in the separation of the less-dense slag and the heavier metal. Slag was poured off intermittently (see Figure 149). Finally, four consumable air lances were used to remove the remaining excess trace elements.

After the final slag/metal separation, the metal was poured into a transfer ladle, taken to the molds and poured into pig ingot form. A final composition sampling was then taken. The pig ingots were later shot-blasted and shipped to P&WA. The final weight of the sludge was 5242 lb. Table 106 lists the final composition of the sludge, with the as-received composition listed for comparison.

4. Economic Analysis

The economic analysis performed on the reclamation of the sludge is presented in Table 107. It shows a net gain of $846.10 to process the 6000 lb of sludge. This would seem to indicate that it is not very profitable to reclaim sludge and grindings; however, several factors combine to negate this observation. They are as follows:

1. In researching this process, the sludge was both bought and processed. In actual practice, a cost-conscious company would want to reclaim the scrap that it had itself generated. Thus, the initial outlay for sludge that was presented in Table 107, $7500, would be zero for this company. It could then have the scrap refined for approximately $0.40/lb and sell it for about $2.05*/lb. This would result in a profit near $1.45/lb of original material after taking into account the material lost in remelting.

2. This analysis was done on a one time, 6000 lb basis. As quantities increase, and if a regular melting schedule were contracted, the refining costs would probably decrease. Thus, this reclamation would provide an even greater return on investment

TABLE 106
CHEMICAL ANALYSES OF
SUPERALLOY SLUDGE

Analysis (w/o)	*As-Received Condition*	*Electric Arc Melted Ingot*
Oil Plus Moisture	< 0.1	
Ni	51.56	66.20
Co	0.26	0.58
Cr	14.3	10.20
Cu	0.04	0.024
Fe	17.28	19.15
Mn	0.17	0.050
Mo	0.56	3.43
Ti	0.77	< 0.03
V	0.04	< 0.02
W	< 0.25	< 0.05
Al	0.71	0.030
Cb	4.00	0.004
Si	2.02	0.087
C	0.15	0.026
S	< 0.088	0.005
P	< 0.03	0.003
Pb	0.01	< 0.002
Sn	< 0.05	< 0.01
Ta	< 0.2	< 0.01
N		130 ppm
O		82 ppm

TABLE 107
ECONOMIC ANALYSIS FOR RECLAMATION OF
NICKEL-BASE SLUDGE

Sludge[1] *6000 lb at $1.25/lb*		$ 7,500.00
Processing[1]		
6000 lb at	$ 0.33/lb Refine Sludge	1,980.00
6000 lb at	$ 0.04/lb Sulphur Surcharge	240.00
	$35.00/ft Certificate of Chemical Analysis	35.00
6000 lb at	$ 0.01/lb Shot Blasting	60.00
11 Pallets at	$ 7.75/pallet	85.00
	Total Cost	$ 9,900.00
Remelt Stock Sale 5242 lb at $2.05/lb		$10,746.10
	Net	$ 846.10

[1]Includes Shipping and Handling

3. If the reclaimed alloy were used as revert material and brought back to initial alloy chemical specification, an even greater savings could be realized. The reclaimed material is effectively free, except for the small cost of refinement as compared to the high price of 100% virgin raw material.

* Selling price based on Ni content which would be a variable as starting alloy composition varies.

5. Conclusions

It has been shown, even on a one-time basis, that reclamation of Ni-base superalloy grinding sludge is quite profitable, as compared to discarding or giving it away. If the material were recycled back to the original or a similar alloy composition, an even greater savings would occur as compared to purchasing 100% virgin alloy.

SECTION XIII

SUMMARY AND CONCLUSIONS

During Phase I of this three-phase AFML contract, scrap reclamation, scrap reclamation technologies and management systems were investigated and analyzed in search of their optimums. Technologies reviewed were sometimes alternatives; from these alternatives evolved a single technology for further study in Phase II. Phase I scrap management effort included an industry seminar, a survey, a review of industry controls, and the definition of a Model Scrap Handling System.

The scrap reclamation technologies that were investigated were two density separation methods, two nonconsumable melt processes, and a molten salt bath process. The two density separation methods were the AVCO ferrofluid method, applied to both titanium and nickel; and the Frankel fluidized bed method, applied to titanium only. After review and analyses of all the titanium separation data, the following conclusions can be drawn:

1. Both separation processes (ferrofluid and fluidized bed) appear equally effective in removal of most contamination, but neither can presently remove all contamination.

2. Any high interstitial or residual elements can be diluted to specification level by less than 50% virgin addition.

3. Both separation processes, as evaluated, result in a chip product containing minor, but unacceptable, levels of high density particles.

4. The unacceptable, retained high density particles should be completely removed by a subsequent nonconsumable melting process.

To evaluate the nickel separation process, AVCO separated Waspaloy chips. After review and analyses of the data, the following valuations can be made:

1. The AVCO ferrofluid separation process appears very effective in removing objectionable high density materials, such as lead, from superalloys.

2. The AVCO ferrofluid separation process appears capable of removing most, but not all, of the titanium contaminant in Waspaloy.

3. Process acceptability therefore appears dependent upon practical virgin dilution requirements to attain melt specification chemistry. Acceptability would appear likely, since about a three-to-one dilution in melting of a given raw material would be the maximum acceptable limit for production melting.

The two nonconsumable melt processes that were evaluated in Phase I were the AIRCO-Temescal Electron-Beam Cold Hearth Melting Method and the Teledyne Nonconsumable Rotating Electrode Melting System. The conclusions of the AIRCO-Temescal Process are:

1. Seeded WC tool bit contamination was completely separated to give an uncontaminated ingot.

2. Chemical specification levels were attained with the exception of both ingot ends.

3. Nonhomogeneous ingot-end chemistry (i.e., % Al) must be resolved; feed system alteration is a likely corrective action.

4. Desirable ingot homogeneous macrostructure (equiaxed and relatively fine grain) and bar microstructures were attained.

5. All mechanical properties data generated (tensile, stress rupture, impact) exceeded applicable specification minimums.

The Teledyne System was evaluated in Air Force Contract F33615-72-C-1126. Testing of the products made from the nonconsumable melted material demonstrated the following:

1. Nonconsumable skull melting, followed by either one or two consumable remelts, effectively eliminates nitride type inclusions.

2. All high density inclusions, such as tungsten carbide tool bit particles, are apparently removed provided they enter the molten pool.

3. High ratios of revert, either machining chips or solid scrap, may be used provided foreign alloy contamination and interstitial elements are at acceptable levels.

4. Single consumable remelting of nonconsumable process electrodes results in better chemical homogeneity than double consumable remelting.

5. Microstructural inhomogeneity (i.e. areas of beta stabilization) increases with residual element content and is more prevalent in double remelted than in single remelted heats.

6. The manufacturing costs for nonconsumable melting exceed those for the conventional process (electrode fabrication plus consumable melt), but overall cost savings result from the ability to use lower cost scrap forms and through elimination of mill product losses for inclusion defects.

An evaluation of engine hardware produced from both single and double remelted Ti-6Al-4V has demonstrated the potential of the process to produce material with acceptable quality and mechanical properties. However, due to the limited scope of LCF testing on forgings with preferred microstructure there was insufficient data to reach a firm conclusion as to the acceptability of nonconsumable melted material for rotating parts in turbine engines.

The investigated molten salt bath process was the Frankel Co. Molten Salt Chemical Purification Process. Phase I efforts, substantiated by data, have realized potential from a grinding/sludge scrap product that is presently considered worthless. The results were promising enough to warrant continued evaluation in Phase II.

The Strategic Materials Reclamation Seminar and Scrap Survey initiated the Phase I scrap management work. They showed that some aerospace plants deny the existence of any scrap problem or treat their scrap metal like rubbish and dispose of it. The individual company's management must recognize the problem and act to replace their insufficient collection system

with a recommended system of recycling. Implementation of the following aerospace scrap recycling areas would lead to an improved system:

- Segregated Titanium Turnings — Any step which eliminates the presence of dense inclusions will tend to increase the use of titanium turnings. The increased use of nonconsumable and EB furnaces should also increase aerospace scrap titanium turnings recycling.

- Cast Superalloys — A major portion of scrap from cast nickel alloys, often blades and vanes, is not recycled for aerospace scrap use. Efforts to find ways to include these valuable alloys in the aerospace recycling program should be fostered.

- All Aerospace Metals — Efforts should be made to increase the allowable scrap used in both rotating and nonrotating parts. Major improvements in processor's abilities to provide guaranteed chemistry should be considered when considering liberalized specifications.

- All Aerospace Scrap Metals — Efforts can and should be made to increase the quality of aerospace scrap metals generated at aerospace manufacturing plants. Methods for accomplishing this are spelled out in the Model System program, another facet of this Strategic Material Reclamation Program.

- All Aerospace Scrap Metals — Consideration should be given, where applicable, to relax restrictions that require alloys to be produced only from the same alloy composition. In addition, all critical material specifications should include maximum tramp elements so that melters may know the limits for those major alloying elements not included in a particular grade. Both of these suggestions were supported at the Strategic Material Reclamation Seminar, May 1974.

The review of industry controls showed that, in general, controls that were applied to scrap usage for nickel alloys were less severe than those applied to titanium alloys. Also, nickel alloys are more tolerant than titanium alloys of common contaminant sources such as frictional or heat treat scaling and fractured tool bits. Finally, many raw material users rely upon the experience and procedures of the principal users with regards to their application of maximum controls over raw material suppliers. Thus, the suppliers benefit from this semi-standardization of procedures.

The Model Scrap Handling System, which was described in detail in this report, maintains that some or many of its techniques could produce a significant improvement in the quality of an aerospace plant's scrap metal. Again, a company must recognize its own problems and apply wholeheartedly the principles of the system if it is to reap the benefits.

Early in Phase II an analysis of the Aerospace Scrap Seminar was made. It was found that the aerospace industry has a predominant need for a cost-effective, reliable separation of titanium scrap as compared to their need for nickel scrap separation. This led to a redirection of the program by termination of separation work on Ni scrap turnings. Ti scrap turnings and Ni grindings and sludges were analyzed for the remainder of the program.

The Frankel Co. fluidized bed density separation process was selected by P&WA for further development under Phase II on the basis that the process was being used in commercial production with reasonably established operating parameters and operating costs. The Phase II Fluidized Bed Processing results are summarized below:

- Machined titanium turnings contained a high concentration of unintended high (WC and steel) and low (stones, etc.) density contaminants. The need for improvements in scrap management and handling at each generator is obvious to reduce/eliminate this contaminant situation.

- The aforementioned separation procedure, including screening, magnetic and fluidized bed separation, appears very effective in removal of these contaminants.

- Almost all detectable contaminants were removed after the first pass of the fluidized bed separation as defined by representative inspection of separation reject material.

- The application of the two additional fluidized bed separation passes, followed by a nonconsumable skull melting separation, should insure removal of these contaminants from the recovered titanium chips and allow the utilization of scrap in rotating gas turbine engine hardware.

The Teledyne-ALLVAC nonconsumable, rotating electrode, arc skull-melting process was chosen for Phase II titanium melting on the basis of the established procedures applied and the acceptable data generated in Air Force Contract F33615-72-C-1126. During Phase II, it was noted that stray chips that fell into the ingot mold cavity frequently contaminated the melting separation process. Teledyne-ALLVAC modified both the furnace and the process operation procedures to correct this problem. They then melted the unseeded and contaminant-seeded lots (nominally 5000 lb each) of titanium chips that completed the triple-pass density separation process described above (Frankel Co. fluidized bed separation).

A quantity scale-up of superalloy grindings and sludges processing, required to establish process economics, were accomplished in Phase II. Two 1100 lb lots of Ni sludge were processed. While metal recovery was considered high in both runs, it was judged, based upon the chemical analysis, that there was no further appreciable benefication attained during vacuum induction remelting following arc melting. This conclusion agrees with earlier Phase I effort. The electric arc melting process warranted further scale-up for Phase III process economica analysis.

In Phase III, four titanium TF-33 disks (two from each heat) were forged from the Teledyne-ALLVAC nonconsumable melting process used in Phase II. Each of the disks was examined metallographically. One disk from each master heat underwent a full cut-up to determine mechanical properties. The results from both the metallographic evaluation and the mechanical property determination proved that reclamation of titanium turnings is feasible for use in rotating gas turbine parts.

Phase III brought a scale-up to production-quantity of superalloy Ni grinding sludge. Six thousand pounds of sludge were processed through conventional drying and electric arc air melting operations, including the use of air-lances. The final chemical composition vs initial chemical composition comparison and the economic analysis performed on the process show that reclamation of Ni superalloy grinding sludge is profitable. Maximum savings occur when the material is recycled into the original, or a similar, composition due to the reduced requirements for purchasing 100% virgin alloy.

APPENDIX A

MODEL SCRAP HANDLING SYSTEM

A. INTRODUCTION

1. Background Information

Pratt & Whitney Aircraft, under Contract F33615-74-C-5019 with the Air Force Materials Laboratory, has conducted a three-phase program to establish systems and techniques for effective reclamation of titanium- and nickel-base scrap. Precontract surveys indicated: (1) significant amounts of aerospace scrap are downgraded beyond reclamation potential by inadequate handling, and (2) aerospace contractors are receiving little or no compensation for the value of scrap removed from their facilities. Both problems projected the opportunity to upgrade the process of aerospace scrap reclamation at the point of origin. As a result, the Air Force approved a contract to define and implement a Model System for the handling of aerospace scrap. P&WA contracted Phase I of the task to Suisman & Blumenthal, Inc., a scrap metal salvage firm located in Hartford, Connecticut. The Model System guidelines, as discussed herein, were implemented at a selected aerospace plant during Phase II, and documented in final form during Phase III of the contract.

2. Work Objectives

The prime objective is to make available guidelines, or techniques, which will assist manufacturing plants in simple and profitable recycling of their titanium and nickel aerospace scrap metals. The overall program, directed towards maximum remelting of titanium- and nickel-base scrap into aerospace use, has the following major objectives:

1. Lower overall aerospace costs
2. Conserve material resources within the United States and reduce foreign dependency
3. Financially strengthen the generators of the aerospace scrap, and
4. Save energy costs.

The U.S. Air Force personnel stressed the objective of lower overall aerospace cost at a Strategic Materials Reclamation Seminar held May 1974 in Hartford, Connecticut. Seminar participants corroborated the idea that melting costs might be lowered if, under controlled steps, greater amounts of aerospace-quality scrap were available and could be melted. In all other areas of scrap metals, the price of scrap which a consumer pays is below that of virgin metal; this pattern is well documented with nickel-alloy scrap metal both in air and vacuum melt purchasing. Titanium scrap has also undersold the virgin material (sponge), usually by wide margins. So, if raw material costs for nickel and titanium are lowered on the average, it is safe to assume overall aerospace costs will be minimized.

For lack of domestic markets, much off-grade titanium scrap and some nickel-bearing scrap (mostly stainless steel) has been exported. Developments under the reclamation program, which tend to produce scrap metal of aerospace quality and provide domestic outlets for that scrap, will conserve nickel and titanium in the United States. Since most virgin materials for both titanium and nickel are imported, increased melting of aerospace scrap metals will exert a positive effect on the United States balance of payments. From a self-sufficiency viewpoint, every pound of scrap consumed lessens dependence on overseas metal supplies.

Increasing the quality of aerospace scrap will financially benefit the generator of the scrap. Conversely, the failure to handle nickel and titanium scrap metals correctly will cause intermixes which are among the most costly, on a percentage drop in value, of any in the metals field.

One neglected area which magnifies the importance of scrap recycling in the aerospace field is energy conservation. Energy savings achievable through recycling titanium scrap compared with virgin material utilization are illustrated below:

Virgin material requirement KWH/Ton	Scrap metal requirement KWH/Ton	KWH savings for each ton of scrap metal used	Barrels of oil saved for each ton of scrap metal
126,115	52,416	73,699	43.4

3. Scope and Purpose of Model Scrap Handling System

The projected Model Scrap Handling System will apply specifically to all aerospace scrap titanium alloys, and to those nickel alloys of 20% or more nickel content which are byproducts of an aerospace manufacturing plant. The system does not purpose to include all methods for aerospace scrap handling. Local variations are expected and, in fact, will be encouraged considering the rate of change in the aerospace industry.

Ideas have come from aerospace manufacturers, processing experience and scrap reclamation programs of the melters, but basically the system has worked successfully in aerospace manufacturing plants. Techniques used over many years to recycle steel, aluminum, copper and other nonferrous scrap at industrial plants were reviewed, combined with methods developed for the problems unique to aerospace metals and finally supplemented on the basis of the subcontractor's wide and specialized experience with aerospace metal salvage systems.

The Model System aims to generate, for sale to dealer or processor, segregated aerospace scrap metal which can most readily be converted to that standard of excellence required by aerospace melters. Experience has shown that manufacturing plants using these guidelines will be able to produce scrap that is 90 to 95% free of serious contamination. If a plant does not have a carefully planned and fully implemented scrap salvage program, the chances are very high that nickel and titanium alloys will be mixed or contaminated. If the physical form is a turning, the scrap may well be lost permanently for aerospace recovery. Even in scrap solids, unnecessary mixtures can inhibit or preempt recycling for aerospace.

B. MODEL SCRAP HANDLING SYSTEM GUIDELINES

The Model System consists of Major and Supplementary Guidelines. The major guidelines (a-o) are presented in chronological order starting at the point of original scrap generation; the supplementary guidelines (p-x) suggest educational and procedural factors to reinforce the major guidelines.

1. Major Guidelines

a. Management Responsibility

Systems designed to obtain maximum quality and quantity depend on leadership; the model system is no exception. The concern of top management, and the dedication of a scrap salvage department manager are vital elements, but the benefits of the system start with a machine shop foreman. His assigned responsibilities over the personnel and the machines that produce scrap must include decisive responsibility for correct segregation of that scrap.

In addition, leadership at the ground level is necessary to defeat certain quasi-historical attitudes about scrap: "Scrap is junk," "I won't bother with that because I only care about making the most parts I can in the least time," "What's the difference if I mix titanium and Inconel; they'll never know...or know it was mine," etc. The machine shop foreman has a clear responsibility for the scrap which his department generates; the absence of this responsibility will unquestionably decrease the quantity of aerospace scrap initially produced and surely diminish the value of the scrap byproduct.

b. Adequate Space Around the Machines

If there is one single difficulty to be found in every type of industrial plant, large or small, which inhibits the flow of quality aerospace scrap, it is overcrowding. The rapid changes characteristic of aerospace manufacture in contrast to the more traditional production operations necessitate "shoehorning" of machines into already crowded areas.

The penalties are clear. Overcrowding can cause mixtures of two different metals because the space for a container for the next metal was not available. Sweepers cannot enter the machine area to remove full containers in sufficient time. In addition, the lack of access around machines can deter machinists or chipmen from properly cleaning between runs of different alloys.

A model scrap handling system demands that adequate space be reserved around the generating machines for facile housecleaning. In addition, sufficient aisles must be established and maintained so that the valuable aerospace scrap can be removed easily. To do otherwise is to forget the byproduct nature of aerospace scrap; it has to be removed on a continuous basis anyway, so why not "engineer" that removal for efficiency and for generation of quality aerospace scrap metal.

c. Machine Dedication

In a model system, careful planning would take into account not only the standard demands of machine layout, but also the benefits of machine dedication.

The processing of one alloy on a single machine, or the machining of one type of metal (e.g., titanium or high-nickel alloys) around one area of machines, tends to produce cleaner, less contaminated scrap. The validity of this premise is evident by the variation in qualities of aerospace scrap now available from American manufacturers. Larger companies, which generally make longer production runs, produce scrap of markedly superior quality. The small machine shop not only has limited scrap control, but also tends to work a wide variety of metals and alloys on each machine. The multiuse of machines breeds scrap intermixture. Although most plants already have well-established machine placements, the goal would be to establish machine dedication in terms of metals and alloys as departments are moved or rearranged, or whenever aerospace plant engineering has the luxury of starting from scratch.

d. Take-Away Systems

Take-away systems refer to those mechanical devices, predominantly conveyors, which transport scrap from the cutting area of machines to nearby containers. The most common "system" of scrap removal from machines is by hand labor, i.e., there is no conveyor. The operator simply pushes, shoves or scoops the scrap which accumulates around his machine into some nearby container. Not only is this type of removal an absolute maximization of labor, but contrary to our purposes, it is the greatest guarantee of mixed aerospace scrap. Clips or chips can easily gather around machine areas. Only careful attention to the aerospace scrap will segregate the various grades and alloys between different "runs."

Contrasted with the hand labor method of removal is the system of various conveyors which automatically carry away the scrap. These systems are not panaceas, for they still require that the operator use judgment and careful attention to avoid contamination in the conveyor parts. Conveyor systems are installed either with the original equipment or after a parent machine is in place and operating. The latter installation is unusual since it really amounts to a redesign of the machine operation, and as such, it is costly and inhibited by normal manufacturing inertia.

Concurrent with the original purchase of a machine, it is possible and simple to consider the problems of the machine byproduct, its scrap metal. The simple step of selecting standard or optional scrap disposal equipment with a machine — this step, if incorporated as one of the specifications of machine purchasing — would bring aerospace scrap removal and recycling closer to production. Considering the high cost of such machines in the aerospace industry, such a requirement should be Standard Operating Procedure.

The basic take-away system in an aerospace plant is a mechanical conveyor system from the machine dropping into a nearby hopper or skid. A take-away system, along with a "dedicated" machine, will tend to yield the purest of aerospace scrap, entirely appropriate to the model system.

e. *In-Plant Containers*

The selection, abundance, maintenance and identification of in-plant scrap containers are also of major significance to the model system. Since ideally, the machine operator and his foreman are (or should be) trying to have aerospace scrap removed from the machine and machine area as expeditiously as possible; the process requires the systematic organization of in-plant containers.

As to the type (or types) of container(s), that decision may vary within each plant based on scrap volume. Types of containers include small shop boxes, steel barrels (55-gal), self-dumping hoppers on skids or wheels and pallet boxes (which do not easily dump). The small boxes are almost always placed at machines to capture small pieces which may then be centralized in a nearby larger unit. Although the container size depends on the size and quantity of scrap being generated, the minimum appears to be steel drums with a hopper or skid system.

A major pitfall to be avoided in handling aerospace metal scrap is the use of oversized containers. The 55-gal steel barrels, for example, are almost always the "right" container for high-nickel or titanium turnings, because such a barrel constitutes a segregation system in itself. If the scrap is placed, for example, in 40 barrels, the chances are that good segregation will take place naturally as the chips or turnings come off the machine. Should a few of the 40 barrels be mixed or contaminated, these can be individually sorted out at the plant's scrap salvage department or by the processor. However, with the same 40 barrels intermixed in a large container or truck body, the contamination would in all likelihood pervade the whole lot and eliminate its highest value as aerospace melt material.

Also to be avoided is a continuing short supply of containers. This structure seems elemental, yet one pervasive difficulty of aerospace plant scrap salvage departments is an adequate supply of containers. Why does this happen? Not enough containers to start with, commandeering for nonscrap purposes (e.g., rubbish), normal attrition as containers wear out or are damaged. Whatever the cause, the absence of a proper scrap container at a convenient location generates scrap contamination.

Akin to the quantity of containers is their quality, especially their cleanliness and appearance. Barrels must be cleared of previous material entirely, must be maintained by washing and painting, and kept in good repair. Quality breeds quality — it says to the machinist,

"This is important." An adequate supply of quality containers for aerospace scrap accumulation tacitly encourages careful segregation at the machine.

And finally, the container's identification, if carefully planned, can be do much to encourage aerospace scrap segregation. Color-coded barrels or containers simplify the segregation. If large runs of one metal or alloy are expected, lettering on the containers is worthwhile, but if containers are interchanged among alloys, tags or metal clip-ons, indicating the alloy, are more practical.

f. Sweeper Control

Sweeper personnel remove the aerospace scrap from the machine areas and transport it to the Scrap Salvage Department. Sweepers must not only be well schooled in the plant layout and the manufacturing systems, but must also be aware of their important role in an aerospace scrap metal program. Unfortunately, sweepers seem to be the first victims of an economy drive, but that drive often turns out to be a false economy. The contradiction of the sweeper force presages higher intermixtures of valuable aerospace scrap, and can, in a few days of scrap generation, cost far more than the intended saving.

g. Container Tagging Systems

Tagging systems vary from plant to plant, but the basic principle is that each container should be identified with a securely tied (steel wires are best) tag. Minimally, the tag, attached at the machine or in the machine area, must indicate the basic alloy content of the container.

At the Scrap Salvage Department, additional information is either added to the machine tag or new tags attached. The weight of the scrap, date of shipment and an identifying numbering system all contribute to the careful control of aerospace scrap. Some plants use barrel tags to designate the departments from which the scrap came and color-code the tags to identify which shift generated the scrap.

h. Catch-All Grade

Just as all salmon do not succeed in returning to the spawning grounds, all aerospace scrap will not reach aerospace recycling. Some scrap will be mixed through carelessness, some because it is machined simultaneously with another metal, perhaps some because the metal was bonded (bi-metal) and some will become contaminated in later processing. Whatever the cause, a separate collection system for this scrap should be available so that it will not be intermixed with high quality aerospace scrap, and its value, although low, will not be wholly lost.

i. Scrap Salvage Department Location

The first eight guidelines to a Model Scrap Handling System deal with generation of aerospace scrap at the machine and within the manufacturing areas of the plant. With those guidelines in place, the scrap will make it to the plant Scrap Salvage Department. The word department (singular) is significant, for in the majority of plants, a single, rather than two or more scrap areas, is most desirable. The location of a Model Scrap Salvage Department should be convenient to the generation source (or sources) of aerospace scrap while providing easy access for scrap removal.

Centrality to scrap generation minimizes the distance traveled to the scrap salvage department, and to that extent compresses the cost of intraplant scrap handling. A less obvious benefit, important to aerospace scrap as opposed to less valuable types of metallic scrap, is the ease with which scrap salvage department personnel can move into the manufacturing areas. To

maintain high aerospace quality, with due concern for variations of nickel and titanium alloys and specification change, scrap salvage department personnel must constantly move in and out of the generating departments. Easy access will facilitate this work and pay dividends by anticipating and avoiding aerospace scrap quality problems.

j. Adequate Space in the Scrap Salvage Department

If location is important, so too is the adequacy of space in the Scrap Salvage Department.

The Model System needs a well-laid-out, commodious Scrap Salvage Department that provides elbow room to adequately handle a projected volume of valuable scrap metal. Adequacy of space in the Scrap Salvage Department will accomplish two important tasks: it will allow all material to be moved in and out freely, minimizing "double handling" and it will avoid scrap intermixing within the department.

k. Simple Testing Systems

Once the aerospace scrap metal has arrived at the Scrap Salvage Department, it must be checked for quality. The overall approach is one of spot-checking, attempting to weed out incorrect gradings and contaminated lots. If the Model System approach has been followed, a high percentage of containers will arrive at the Scrap Salvage Department area already in segregated condition suitable for shipment.

Scrap Salvage Department personnel should adjudge the physical uniformity of material within a container. If that uniformity exists, spot-checking pieces and testing with a hand magnet will help spot magnetic contaminations within a container. In addition, several makes of thermoelectric testers are available. These testers operate on a "Go-No-Go" basis, identifying pieces which match a known sample. While not inexpensive, the cost will more than pay for itself in allowing scrap personnel to test, in a simple way, the correctness of individual containers of solids and turnings. The color of aerospace metals is of some value as a means of identity. Copper, brass, aluminum and other nonferrous contaminations can be determined by color. In most common aerospace intermixture, that of titanium with nickel-based alloys and vice versa, can also be detected by competent Scrap Salvage Department personnel, both by color and texture. Another simple, although limited, system to control aerospace scrap quality is an inspection for mill or plant markings on solids. This system is also limited by the lack of markings on many pieces by the time they reach the Scrap Salvage Department. One other caution; mill or plant markings may be incorrectly stamped, and therefore, insidiously dangerous as quality determinants for aerospace end use.

Since the goal is the constant production of aerospace-potential metal at least 95% segregated at the Scrap Salvage Department level, the testing methods listed above will be sufficient if they are preceded by the machine-to-scrap salvage department techniques of the Model System.

l. Scrap Weight

Minimally, a Model System must have a platform scale (with automatic printout) in the Scrap Salvage Department to measure the weight of completed, ready-to-ship aerospace scrap — that weight to be recorded on the container's tag.

If one or more aerospace items are produced in truckload quantities, then a truck scale may be needed. To justify the expense of a truck scale, other nonaerospace items of scrap (e.g., steel scrap) would presumably be generated and with perhaps nonscrap requirements for truckload weighing, also be weighed on the scale. A truck scale should be at least 60 ft in length with a 50-ton capacity, have an automatic printout, and be all-weather in design.

m. Truck and Trailer Access

To assure the flow of metal through and out of the Scrap Salvage Department, easy passage should be engineered for those service vehicles which will transport the metal automatic dock boards, ample width, sufficient truck wells, clear visibility to back up, clear passage to the truck scale (if any) and street, etc. Again, all-weather protection for the aerospace scrap being loaded on the vehicle is part of a Model System.

n. Feedback of Incorrect Grading

Despite a Model System or the diligence of the Scrap Salvage Department manager and his personnel, errors will take place. The yearly generation of aerospace scrap metal at even a medium size machine shop will consist of hundreds of thousands, if not millions, of individual "parts" — the individual pieces of solids and turnings. It is impossible for every "part" to be correctly separated.

One vital facet of aerospace scrap salvage is the feedback of information about grade errors. The receiver of the scrap must promptly advise the aerospace plant of any major and/or continuing misgrade difficulty, noting the precise shipment and container (if possible) in which the contamination took place. Conversely, the aerospace plant personnel should recognize the information as an opportunity to avoid similar contamination in the future by retracing the contaminated scrap to its source. In some cases, misgraded scrap, and/or samples should be returned to the aerospace plant.

A running record should be kept at a Model System plant as to which areas or departments are generating the contaminated materials. This will allow the pinpointing of trouble areas and the highlighting of progress in improving aerospace scrap quality.

o. Quality Dealer/Processor Service

The services of a qualified scrap dealer or processor appear to be "highly desirable" as the final element in a Model Scrap Handling System. This firm could be expected to assist in setting up the Model System, having a working experience with titanium and nickel-base alloys, provide a wide market for aerospace scrap and most significantly, obtain the highest return for the scrap producer.

Customer service by a qualified scrap dealer will include assistance with aerospace scrap segregation, speedy removal of scrap accumulation, feedback information on faulty quality and rapid return of plant containers. A symbolic relationship between scrap dealer and producer becomes most evident when scrap metals are not "moving." The reliable scrap firm will provide a "home" for producer's scrap metal at particular times when consumers (melters) may not be buying.

2. Supplementary Guidelines

A Model System is, hopefully, now established from machine to scrap salvage department and scrap salvage department to receiver. But considering the difficulties to be expected, reinforcement of the system should be effected. These efforts are educational and procedural, but will nonetheless be significant in the success of an aerospace scrap handling program.

a. Name of Department

"Scrap Salvage Department" or "Scrap Control Department" should be employed, not merely "Scrap Department." The constructive facets of the program are thus emphasized, tending to challenge the idea that scrap is "junk," and/or worthless. Younger employees especially, are interested in conserving natural resources and emphasis on recycling salvage should help enlist their support.

b. Color Coding

Wherever possible, try to code the alloys by color. The uses of color-coded barrels and department tags have already been mentioned. Larger containers, such as hoppers, also can be painted to indicate the contained alloy.

c. Simplification of Grades

This process involves the combining of two or more aerospace alloys into one plant scrap classification. Inconel, for example, comes in a variety of designations, all of which contain 70-75% nickel, with a need to isolate only those containing cobalt.

d. Designation Dissimilarity

In a Model System, the aerospace plant should avoid use of company, mill, or trade names in the aerospace field which are very similar to and easily mistaken for other dissimilar alloys. AMS 5382 and AMS 5385 are H.S. 31 and 21, respectively. For clarity, designate AMS 5382 for one type of operation and H.S. 21 for another.

e. Concept Values

For successful aerospace scrap recycling, concepts are necessary ingredients of a Model System, i.e., the importance of aerospace scrap for company profits, saving vital national raw materials, aerospace scrap as a normal and important company product or byproduct. These concepts should be integrated into the training and education programs of the aerospace plant. Eventually, the banana peel will not be tossed into the barrel of commercially pure titanium turnings.

f. Procedures

The Model System should be woven into company procedures spelling out the responsibility for its handling and the forms to be used in fulfilling these responsibilities.

g. Alloy Designation Changes

Change is characteristic of aerospace engineering, and a tangible evidence of that change is the substitution of one titanium alloy for another or a new nickel-based alloy to replace a previous specification. It will be mandatory to advise the byproduct manager, the head of the scrap salvage department, of any alloy change.

h. Plant Problem Areas

The experimental and research areas of a plant are operating in different modes from the production area: different "production" goals, different machining requirements, short spurts of scrap as opposed to production runs. These sources of scrap, as well as others such as repair departments, will inherently produce lower quality nickel-based and titanium aerospace scrap.

The safest course is to quarantine these areas from production runs of aerospace scrap, being certain that it is not intermixed with high quality production scrap unless quality is checked meticulously.

l. Off-Grade Generation

Just as some areas tend to produce contaminated, mixed scrap, some types of aerospace production will spin out scrap which is mixed at the machine itself. Examples of this unavoidably low-grade aerospace scrap are cuttings from two different alloys or metals which are machined simultaneously or the production of scrap punchings, perhaps titanium, so small that intermixture of the alloys is virtually unavoidable.

Again, these possibilities must be faced so that these generations of aerospace scrap, probably a small percentage of the whole, do not contaminate carefully segregated nickel-based or titanium alloys. The off-grade accumulations should be kept out of the mainstream by placing them in one of the catch-all grades.

C. MODEL SYSTEM COSTS AND EVALUATION

1. Costs

The implementation of the Model System will involve costs. These costs can be anticipated and their reality should be neither minimized nor exaggerated. Capital costs will fall into four areas: floor space, internal materials handling equipment, testing equipment, and external transport containers and vehicles.

The floor space need not be an inordinate area. Too large an area would actually decrease the efficiency of aerospace scrap recycling. Conversely, some space would be required for nickel-base and titanium scrap even if it were treated as rubbish, so that the net space needed would be incremental as necessary to do an adequate job.

Likewise, it would be necessary under all circumstances to have transport for moving metallic scrap from machine areas to a centralized area. The incremental needs – an adequate supply of the correctly designed, smartly painted containers – would be minimal. Purchase of the take-away conveyors to facilitate removal of chips and solids from the machines would in most cases necessitate an increased capital expense; an expense, however, that should quickly pay for itself in reduced labor and higher aerospace scrap values.

Testing equipment would require a capital outlay, but the costs are modest and the higher return on scrap values would be immediately realizable and clearly visible.

No capital expense should be incurred for the transport containers and vehicles. If the scrap firm meets the requirements of full service, this equipment should be provided at no cost to the aerospace plant.

Operating costs will overwhelmingly be for personnel – the Scrap Salvage Department Manager, personnel in the Scrap Salvage Department, and chipmen/sweepers. The number of people involved will relate directly to the size of the plant and its volume of aerospace scrap. In a small plant, the Scrap Salvage Department Manager, for example, will in all likelihood have other duties and only a portion of his time will be chargeable to the Model System. Similarly, the number of full- or part-time people in the Scrap Salvage Department will relate to the volume of aerospace scrap. The size of the corps of chipmen/sweepers must be adequate, but only the incremental number of man-hours attributable to the careful flow of good quality aerospace scrap should be chargeable to the program itself.

The story of Plant X is illustrative. This aerospace plant had been throwing all its scrap together, aerospace scrap and steel scrap and having it hauled away to a local outlet. Almost no revenue was being realized and no aerospace quality scrap — not one pound — was segregated. When the General Manager was shown that with a minimum of effort his aerospace scrap could be easily segregated, steps were taken to implement the program. Within two months, a scrap handling program was organized, and 19 items had been segregated. The plant's second monthly check was for $4,100. At last count, this plant had 27 items and received an average of $14,000 per month.

Relative to the costs involved in this accomplishment, the General Manager stated that "it was not a big deal." The most important tasks were to explain the system to management and establish the discipline of the system with salvage personnel and machine operators. He stated that there were virtually no capital costs involved, since the in-plant containers were supplied by the scrap firm which received the aerospace scrap. His operating costs increased by an estimated 40 man-hours per week to collect and weigh the segregated scrap.

Not all conversions to the Model System will come about as simply or as inexpensively as in Plant X, and the results may not be as dramatic. But when the capital and operating costs are totaled, it will become clear that the incremental costs of the Model System are modest. The reason for this is that the most basic part of a Model System is not the equipment/people investment but rather the organizational change.

2. Measurement of Successful Application

Once the key pieces of the system — Management, Scrap Salvage Department Manager and a scrap firm — are in place, the practical goal of aerospace recycling success appears simple. All scrap shipped by the manufacturer should be minimally 95% of the chemistry ascribed to it, and conversely, the scrap firm buying the aerospace scrap should expect no more than 5% off-grade material in any container.

For example, if the manufacturer ships 20 barrels of Hastelloy X turnings and 19 of them are uniform and uncontaminated, then that generator of scrap has accomplished an outstanding job of recycling. Or, if 5000 lb of Ti-6Al-4V solids are sold and at least 4750 lb are Ti-6Al-4V, the Scrap Salvage Department Manager knows that he has met the minimum expected of his department. Not that 5000 lb would have not been better, but the manufacturing plant simply cannot turn out complete chemistry on a daily basis, and even if a shipment were uniform in chemistry, its cleanliness (cutting oil), surface condition, physical size and packaging would in all likelihood be unmatched to melter requirements.

The statistical measure of scrap handling success is profit-oriented. It is the point to which a manufacturer can aspire without incurring major, usually diminishing-return expenses. The cost of removal of that last 5% will be exorbitant and unprofitably expensive to attain. Yet even if the plant is only partially successful, and only a small percentage of the scrap ends up as fully-segregated turnings, the plant will derive larger payments for its aerospace scrap.

Finally, a study at one plant showed that the difference between nickel-based and titanium alloys which were not mixed, and those which were mixed and/or contaminated, varied between $0.04 and $0.94/lb over a 10-year period. During that decade, Ti-6Al-4V solids averaged in the area of $0.31/lb over the unsegregated counterpart grade; in turnings, the comparable average was $0.13/lb. In nickel alloys, Inconel solids averaged about $0.56/lb over mixed nickel alloy solids, while Inconel turnings showed a corresponding price advantage of $0.60/lb over intermixed nickel alloy turnings. These typical values of segregated/unsegregated alloys show the economic benefits attainable with a Model System for scrap reclamation.

D. SUMMARY

No problem can be solved until it is recognized and a serious determination is made to resolve it; techniques for solution can then be implemented.

Aerospace plants have sometimes denied the existence of a scrap problem and some have treated their scrap metal like rubbish and disposed of it. There was no problem except for the extraordinary financial and raw material waste. The will to replace a sloppy collection system with a true system of recycling must exist or be developed by the company's management. With determination, some or many of the techniques of this Model System can produce a significant improvement in the quality of the aerospace plant's scrap metal. Our present and future dependence on the outstanding qualities of nickel and titanium deserves no less.

APPENDIX B

RESIDENCE TIME CALCULATIONS IN FERROFLUID SEPARATOR

The transit time of a titanium particle is a function of the forces acting on it. The equation of motion for this particle is given by:

$$\rho_s v \frac{dy}{dt2} + C_D \frac{dy}{dt} = F_b \quad \text{(A1)}$$

where:

v	=	volume of particle.
ρ_s	=	density of particle.
y	=	distance in vertical direction.
t	=	time.
C_D	=	drag coefficient.
F_b	=	body force.

The body force is given by:

$$F_b = v(\rho_s - \rho_f) g - v(M_f - M_s) G \quad \text{(A2)}$$

where:

g	=	acceleration of gravity.
ρf	=	*physical density of ferro*fluid.
M_f	=	magnetic dipole moment per unit volume of the ferrofluid.
M_s	=	magnetic dipole moment per unit volume of the particle.
G	=	imposed vertical magnetic field gradient acting in the direction of gravity.

The "apparent" densities of the ferrofluid, ρ_{sf}, and the particle, ρ_{ss}, are defined as:

$$\rho_{sf} = \rho_f + \frac{M_f G}{g} \quad \text{(A3)}$$

$$\rho_{ss} = \rho_s + M_s \frac{G}{g} \quad \text{(A4)}$$

The body force relation (Equation A2) may therefore be rewritten as:

$$F_b = v(\rho_{ss} - \rho_{sf}) g \quad \text{(A5)}$$

For the initial conditions of interest:

$$y = 0, \frac{dy}{dt} = 0 \text{ at } t = 0;$$

the integral of Equation (A1) is:

$$y = \frac{F_b}{C_D}\left[t + \frac{\frac{C_D t}{\rho v}}{\left(\frac{C_D}{\rho}\right)} - 1\right] t + \frac{\frac{C_D t}{\rho v}}{(C_D/\rho v)} - 1 \quad \text{(A6)}$$

For small values of the variable $C_D t/\rho v$, Equation (A6), when Equation (A5) is substituted for the value of F_b, reduces to:

$$y = \frac{\rho_{aa} - \rho_{af}}{\rho_a} \quad g\frac{t^2}{2} \tag{A7}$$

Equation (A7) describes the motion of a particle, for small values of time, when the effects of viscosity are small and its trajectory is determined by its inertia. This relation is expected to be valid for larger particles that have a very short transmit time in the ferrofluid pool. It would not accurately describe the motion of a small particle under the influence of viscous drag.

Equation (A7) can be simplified further for nonmagnetic objects when $\rho_a = \rho_{aa}$, the the following form

$$y = (1 - \frac{\rho_{af}}{\rho_a})\ gt^2 \tag{A8}$$

By transporting terms, the transit time is expressed explicitly as:

$$\sqrt{\frac{2y}{\left(1 - \frac{\rho_{af}}{\rho_a}\right) g}} \tag{A9}$$

Equation (A9) may be used to estimate the transit time in a separator if the condition

$$\frac{C_D t}{\rho v} < 1$$

is satisfied.

As a specific example, consider the transit time of a titanium alloy particle that has a density $\rho_a = 4.5$ gm/cm^3 in a ferrofluid separator operating at an apparent density $\rho_{af} = 4.7$ gm/cm^3 with Ferrofluid 1224. In a separator that has a total height of 20 cm, when the scrap is introduced in the middle, the largest value of y is 10 cm. In this case, the transit time according to Equation (A9) is 0.56 sec.

An estimate of the magnitude of the term $C_D t/\rho v$ can be obtained by assuming the particle to be a sphere moving at a Reynolds number low enough for Stokes Law to apply. Under these conditions

$$C_D = 3\ \Pi\ \eta d$$

where:

d = particle diameter,
η = viscosity of the ferrofluid.

By expressing the particle volume in terms of its diameter, the following expression can be written:

$$\frac{C_D t}{\rho v} = \frac{(3\ \Pi\ \eta d)}{p\ \frac{\Pi\ d^3}{6}}\ t = \frac{18\ \eta t}{\rho\ d^2} < 1 \tag{A10}$$

Ferrofluid 1224 has a viscosity of 0.035 poise, and titanium has a density of 4.5 gm/cm^3. For a value of t = 0.56 sec, the condition $C_D t/\lambda < 1$ is satisfied if the particle size is greater than 0.28 cm or approximately 0.1 in.

In the separation test program, the ferrofluid density was 4.7 gm/cm^3 or greater, so that a residence time of 0.56 sec is the longest residence time considered. The various lots of scrap processed, with the exception of Lot 03, were all screened over a 1/8-in. screen to remove fines. The pieces of titanium being treated are thus large enough to satisfy the condition $C_D t/v < 1$ even though they are not spherical. There is less drag on a thin plate than on a sphere of equivalent size. Thus, for Lot 03, the use of the previous equations to characterize transit time may be inaccurate due to the presence of roughly 10% fines in the +1/8-in. turnings.

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