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DEVELOPMENT AND EVALUATION OF PROCESSES FOR DEPOSITION OF NI/CO--ETC(U)
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DEVELOPMENT AND EVALUATION OF PROCESSES FOR DEPOSITION OF Ni/Co-Cr-AlY (MCrAlY) COATINGS FOR GAS TURBINE COMPONENTS

DETROIT DIESEL ALLISON DIVISION
GENERAL MOTORS CORPORATION
INDIANAPOLIS, INDIANA 46206

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TECHNICAL REPORT AFML-TR-76-230
FINAL REPORT FOR PERIOD 1 DECEMBER 1975 - 1 OCTOBER 1976

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pressing was introduced near the end of the Task I effort. As a result of this, no corrosion testing was performed on the NiCrAlY coating. Previous work concentrated on achievement of MCrAlY coating structures by vacuum and hydrogen sintering plus aluminizing. A number of experiments were conducted however only partial sintering was achieved without the use of the press cycle.

The AEP NiCrAlY process feasibility was demonstrated. Additional work is required to develop the potential of the process including achievement of CoCrAlY and Ni/CoCrAl coatings through pressing and aluminizing AEP deposited CoCrY and Ni/CoCrY powders.

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FOREWORD

The objective of this program is to develop a practical and cost-effective process for the deposition of MCrAlY coatings which provide protection of advanced nickel or cobalt base alloys in turbine engine service environments, equivalent to PVD MCrAlY coatings.

The work described herein was performed by the Detroit Diesel Allison Division of General Motors Corporation Materials Development Laboratory, Indianapolis, Indiana under Contract Number F33615-76-C-5047 sponsored by the Air Force Materials Laboratory.

This Final Task I Report covers the period of 1 December 1975 to 1 October 1976. Mr. K. H. Ryan is the Program Director and Mr. J. O. Hodshire is the Principal Investigator. Mr. Jesse J. Crosby, AFML/LLM, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio is Technical Manager for the Air Force.

No implication is intended of Air Force approval of the contents of the report. Data, findings and conclusions are published only for the exchange and stimulation of ideas related to the objectives of the program.

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SUMMARY

The feasibility of producing an MCrAlY type coating structure by AEP deposition of fine pre-alloyed, chemically activated NiCrY particles with subsequent room temperature isostatic pressing and aluminizing treatments was demonstrated. Electron microprobe scans of the AEP applied NiCrY aluminized coating on directionally solidified Mar M006 showed a NiCrAlY coating with small chromium rich particles dispersed throughout a nickel-chromium-aluminum matrix and minimal interdiffusion with the base metal. Scattered porosity remained in the coating. The yttrium distribution was not determined.

Processing parameters used in application of the AEP NiCrAlY coating were as follows:

- o Fine NiCrY particles in a size range of 1-20 were electro-phoretically deposited.
- o Zein deposited on the NiCrY particles during the electro-phoretic process was removed with a 2 hour cycle at 760 C in argon.
- o Densification of the NiCrY coating was achieved by pressing at room temperature in a hydrostatic press at 54,000 psi with a 15 second cycle.
- o The pressed NiCrY coating was densified, bonded to the base metal and converted to NiCrAlY by pack aluminizing at 1130 C for 2½ hours.

The isostatic pressing cycle was introduced into the processing development near the end of the Task I effort. As a result of this only a limited number of press cycles could be run and corrosion testing of the NiCrAlY coating was not conducted. Previous work concentrated on achievement of coating structures by vacuum and hydrogen sintering plus aluminizing of the MCrY and MCrAlY powders. A number of experiments were conducted however only partial sintering was achieved.

The AEP NiCrAlY process feasibility was demonstrated with the inclusion of the press cycle however the following additional work should be conducted to develop the process.

- o NiCrAlY coating thickness was 2.5 mils. Coatings with increased thickness should be developed by increasing electrophoretic deposition times.
- o Scattered porosity remained in the NiCrAlY coating. This could be eliminated by use of higher pressing pressures and/or improved bagging techniques.
- o Achievement of CoCrAlY and Ni/CoCrAlY composition should be evaluated by pressing and aluminizing AEP deposited CoCrY and Ni/CoCrY powders.
- o Varying aluminum contents of the MCrAlY coatings should be investigated by adjusting pack compositions.
- o Techniques for coating of complex components should be developed by use of press cycle bagging formed by dipping the component or by use of segmented bagging.
- o Hot corrosion, ductility, and oxidation evaluations of the castings and evaluation of their effect on substrate properties should be conducted.
- o An evaluation of the process scale-up should be made.

SECTION I

INTRODUCTION

The objective of the program is the development of a cost-effective process for applying M(Co/Ni) CrAlY coatings to advanced nickel and cobalt base superalloy gas turbine components which will provide protection equivalent to the PVD applied coatings. Further the process shall be applicable to both single blades and complex multiple vane segments producing uniform coating thickness without the line-of-sight limitations inherent in the PVD coating application procedures.

The approach in this investigation is based upon the electrophoretic (AEP) application of pre-alloyed fine powders followed by cold compaction and high temperature treatments to develop MCrAlY type coatings. The AEP coating procedure has previously demonstrated the capability of producing modified aluminide coatings by the deposition of multiple element and/or pre-alloyed fine powders on a variety of nickel and cobalt base superalloy turbine components including complex multiple vane geometries.

The temperatures used in the report are in the SI system. An English unit conversion chart is included in the Appendix. Major equipment used in the program is also described in the Appendix.

The Task I objective is to demonstrate the feasibility of the AEP-MCrAlY process.

TASK I DEVELOPMENT PROGRESS

Program efforts for the contract period (December 1, 1975 to October 1, 1976) have been confined to Task 1 of the development program. The goals being to establish processing parameters for electrophoretic application of MCrAlY type coatings to simple test specimens of directionally solidified (DS) Mar M006 and TDNiCr alloys. The Mar M006 DS specimens were utilized with the approval of the AFML Technical Manager because of their immediate availability and in recognition of the close similarity to the Mar M247 DS alloy which will be used for hot corrosion/ductility test airfoils.

Allison Electrophoretic Process (AEP)

Micron size particles dispersed in solutions especially prepared for electrophoretic deposition will migrate and deposit on work suspended in the solution when voltage is applied between the terminals as shown in Figure 1.

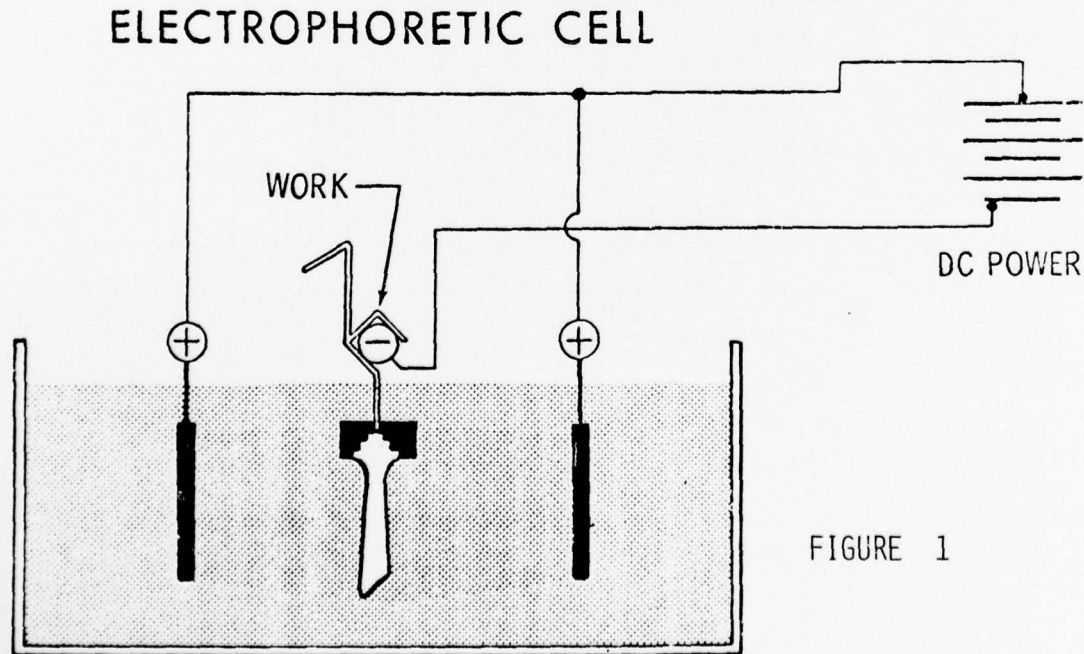


FIGURE 1

Electrophoretic deposition differs from electroplating in that particles are deposited rather than ions.

Solutions for AEP deposition of the fine pre-alloyed powders used in this program are formulated as shown below:

AEP COATING SOLUTION COMPOSITION

Pre-alloyed MCrAlY Powder	-	20.0 gms/liter
Zein	-	2.7 gms/liter
$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	-	.1 gm/liter
60 w/o isopropanol - 40 w/o nitromethane mixture	-	1.0 liter

BATH MAKEUP

Zein Concentrate

Zein concentrate solution for the AEP suspension is prepared by dissolving zein powder (15-20 gms/l) in a 60 w/o isopropanol-40 w/o nitromethane solution. The zein powder is dissolved in the 60-40 solution while stirring the solution vigorously. This solution with the undissolved portion is allowed to settle overnight and the liquid is decanted. Zein concentration of clear liquid concentrate is then determined by evaporation of a 10cc sample of the solution at 100 C. The residue (Zein) is then weighed and the concentration calculated.

Cobalt Nitrate Hexahydrate Concentrate

This solution is prepared by dissolving 2 gms. of Cobalt Nitrate Hexahydrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) in 1 liter of the 60-40 solution.

AEP Coating Solution Preparation

Sufficient quantities of the liquid concentrates are mixed with the 60-40 solution to give the AEP solution composition shown on page 2-3. The weighed pre-alloyed powder is then placed in a Waring blender with the solution poured over it and thoroughly mixed. The AEP coating solution is then poured into the coating beaker or tank.

AEP Anoding

Anoding for these experiments was not configured to the work. Anoding consisted of four equally spaced eleven mesh brass wire screens 1 3/16" wide and 3" long electrically connected. These were placed inside a 600 ml glass beaker next to the glass. Screen anoding permitted better flow and was easier to clean than solid anoding. Work was suspended in the center of the anoding. Anoding was connected to the positive terminal of a constant voltage DC power supply. Work was connected to the negative terminal.

AEP Coating Parameters

Agitation

Agitation was provided by a stirrer adjusted so as to move the solution rapidly enough to keep the particles suspended. Stirring rods were insulate to prevent short circuiting during coating.

Constant DC Voltage

Specimens were coated at 200 volts. This was the maximum voltage that could be used with this anode spacing as slight "treeing" or roughness was noted on sharp edges.

Current

Current is self regulating depending mainly on the applied voltage, distance from anode to cathode and area of the work.

Temperature

Room temperature during coating ranged from approximately 22.2 C to 24.4 C. Solution temperature, due to evaporation, was approximately 1.1 to 1.5 C lower than room temperature.

Program Alloys

Alloys used for specimens were Mar-M006 D, Mar-M247 DS and TDNiCr. Specimens of IN 713 alloy were also used in certain experiments for comparison and evaluation. Nominal compositions of these alloys are shown on the next page.

NOMINAL COMPOSITIONS OF PROGRAM ALLOYS (W/%)

	<u>Al</u>	<u>B</u>	<u>C</u>	<u>Co</u>	<u>Cr</u>	<u>Mo</u>	<u>Ni</u>	<u>Ta</u>	<u>Ti</u>	<u>W</u>	<u>Zr</u>	<u>Hf</u>	<u>ThO₂</u>
Mar-M247	5.5	.015	.15	10.0	9.0	.55	Bal	3.1	1.5	10.0	0.5	1.34	
Mar-M006	5.5	.015	.15	10.0	9.0	2.5	Bal	1.5	1.5	10.0	.05	2.15	
TDNiCr			.04		20.5		Bal						2.2
IN 713C	6.0	.01	.14		14.0	4.0	Bal	(2.3 Cb+ Ta)	.9	.9			

TDNiCr specimens were cut from blocks of TDNiCr into specimens approximately 5/16" x 1/8" x 1 1/2" in size. Mar-M006 DS and Mar-M247 DS specimens were cast in molds used to cast Model T56 1st stage turbine blades. The Mar-M006 DS specimens were to be coated for corrosion/ductility testing.

MCrAlY Powders

Program powders were in a micron size range easily suspended with mild agitation.

Chemical and Coulter analysis of the fine pre-alloyed CoCrAlY, NiCrAlY, CoCrY and NiCrY powders used in this program are shown in Table 1. The yttrium analysis was made by atomic absorption. The chemically activated NiCrY by Universal Cyclops was analyzed by atomic absorption. All other chemical analysis was made by wet chemistry.

Table 1

Chemical and Particle Size Analysis of MCrAlY and MCrY Powder

<u>Powder</u>	<u>Chemical Analysis (W/o)</u>						<u>Particle Analysis (μ)</u>		
							<u>DDA</u>		
							<u>Cerac</u>	<u>Peak of</u>	
						<u>Ave.</u>	<u>Dist.</u>	<u>Range</u>	
CoCrY	--	71.82	15.94	--	4.05	.15	.82	6.4	2.0-25.0
NiCrY	75.6	.031	15.6	--	7.8	.15	.50	7.4	1.5-17.0
CoCrAlY		59.27	16.33	14.13	4.93	.43	.93	3.5	1.0-15.0
NiCrAlY	71.66		17.14	6.13	2.60	.24	.78	16.0	2.0-22
*NiCrY	Bal.		15.0		1.4	.17		7.3	1.5-20.0

*Chemically activated - Universal Cyclops

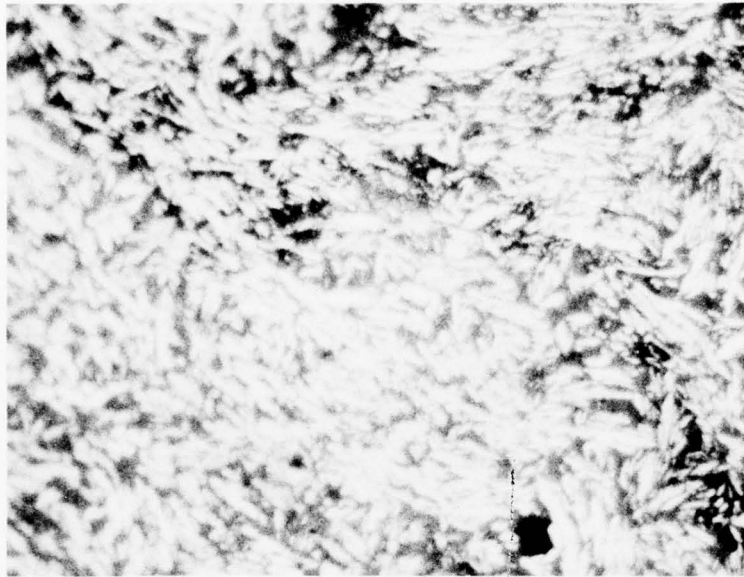


Figure 2

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Pre-alloyed CoCrY Powder

Early efforts to accomplish sinter densification and bonding of MCrAlY type coatings to the DS alloy at the higher temperature ranges revealed unsatisfactory coating density and poor quality interparticle and substrate bonding. This problem was determined to be associated with oxidation effects from the best available hydrogen atmosphere environments. Figure 3 is illustrative of the microstructural and bond zone problems associated with the use of hydrogen atmosphere. Several technique modifications related to the "green" coatings and hydrogen heat treatments were tested without material success. These included thin electrolytic nickel strike on the substrate prior to AEP coating, addition of free aluminum to AEP dispersions, and application of ammonium chloride as a fluxing agent over the AEP deposit.

Coating test specimens of TDNiCr became available for Task I appreciably later than the Mar-M006 DS specimens. As a result of the time lag, efforts to AEP coat this material were benefited by the earlier work in

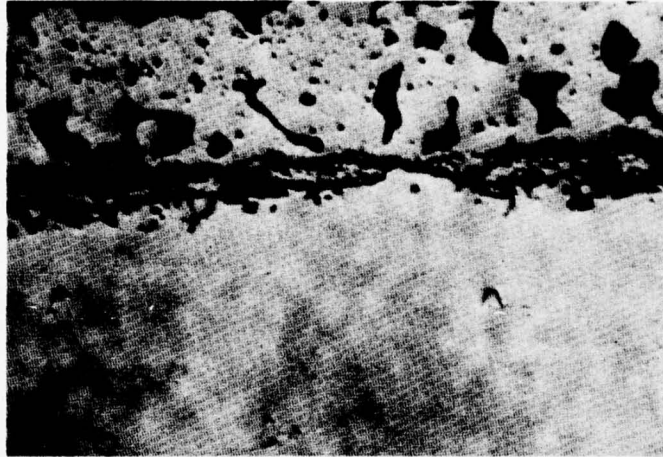


Figure 3

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AEP deposited CoCrY over a thin nickel electrolytic strike on Mar-M006 DS diffused 24 hrs at 1219 C.

coating the DS alloy. This specifically applied to the sinter densification and bonding heat treatments of the green AEP coatings. Limited effort was expended in using the hydrogen atmosphere for heat treatment of coated TDNiCr specimens.

Vacuum Sintering

The decision was made to investigate the use of vacuum sintering in an effort to overcome problems related to use of hydrogen atmospheres. Initial efforts showed an appreciable reduction of interparticle and coating-substrate oxidation interference problems with substantial improvement of interparticle and substrate bonding characteristics.

Vacuum Sintering Cycle at 1149 C

The first vacuum sintering was run with the zein binder in the coated specimens. Specimens of both Mar-M006 DS and TDNiCr base alloys were coated with CoCrY, NiCrY, CoCrAlY and NiCrAlY pre-alloyed powders. Coated specimens were then sintered in a vacuum at 1149 C/4 hours.

TDNiCr

Visual examination of coatings showed a clean metallic luster with no apparent oxidation. Microexamination of the sintered TDNiCr specimens revealed no contamination at the bond line as observed in hydrogen sintering. Less porosity and better bond was observed in the NiCrY coating than in CoCrY coating. Figures 4 and 5 are photomicrographs of NiCrY and CoCrY coatings on TDNiCr.

Mar-M006 DS

Microexamination of the D/S Mar-M006 coated with CoCrY and NiCrY coatings were similar to the TDNiCr samples, only the NiCrY was bonded to the base metal on Mar-M006 DS. Photomicrographs of NiCrY and CoCrY on Mar-M006 DS are shown in Figures 6 and 7, respectively. Surfaces of CoCrY and NiCrY coatings had a metallic luster. NiCrAlY had a slight surface oxidation on visual observation. Microexamination showed that the NiCrAlY coating was not bonded. Co CrAlY exhibited a dark powdery residue on the surface and the coating was not bonded. Photomicrographs of NiCrAlY and CoCrAlY on Mar-M006 are shown in Figures 8 and 9, respectively. Results of the vacuum sintering run indicated the following:

- o With the vacuum sintering no contamination was present at the bond line as was observed in the hydrogen sintering.
- o Only NiCrY coating was bonded on both D/S 006 and TDNiCr.
- o Considerable porosity remained in all the coatings.
- o Aluminum containing powders were susceptible to oxidation with higher aluminum, CoCrAlY, showing the greatest oxidation. CoCrAlY powder contained 14% aluminum to 6% for the NiCrAlY.

Vacuum Sintering Cycle at 1219 C/4 hours

Mar-M006 DS Samples

To improve bonding and sintering the sintering temperature was increased to 1219 C.

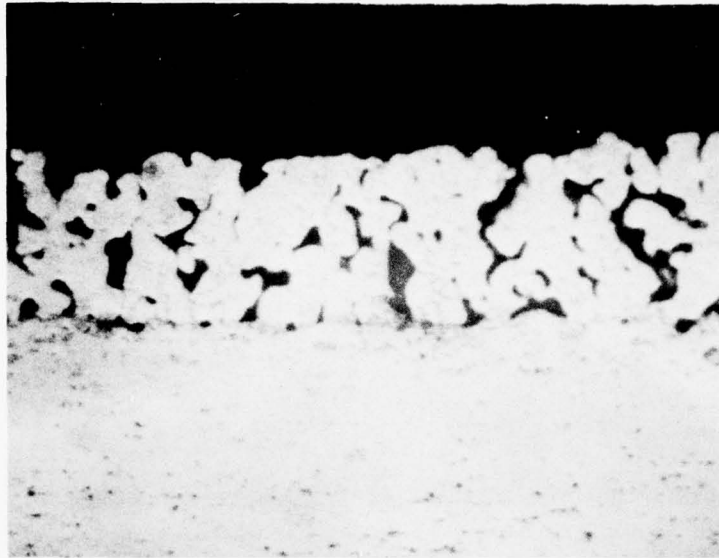


Figure 4

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Pre-alloyed NiCrY coating on TDNiCr sintered at 1149C/4 hrs in a vacuum.



Figure 5

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Pre-alloyed CoCrY coating on TDNiCr sintered at 1149C/4 hrs in a vacuum.

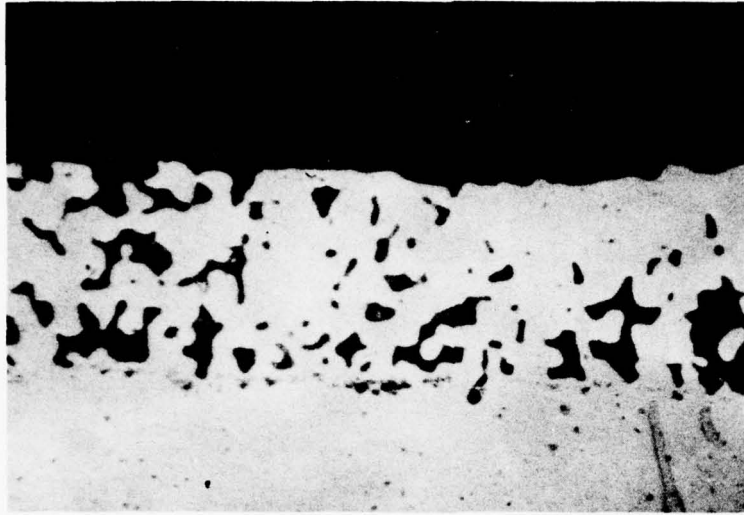


Figure 6

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Pre-alloyed NiCrY coating on D/S Mar-M006 sintered at 2100F(1149C)/4 hrs in a vacuum.

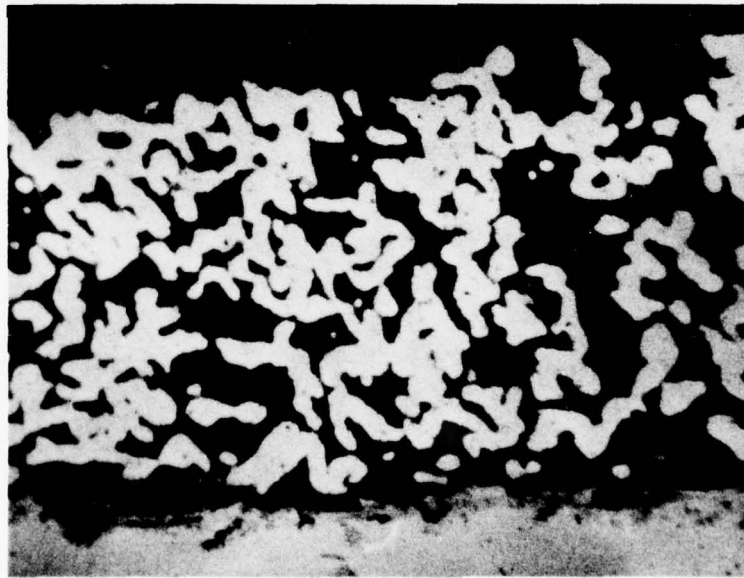


Figure 7

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Pre-alloyed CoCrY on D/S Mar-M006 sintered at 2100F (1149C)/4 hrs. in a vacuum.

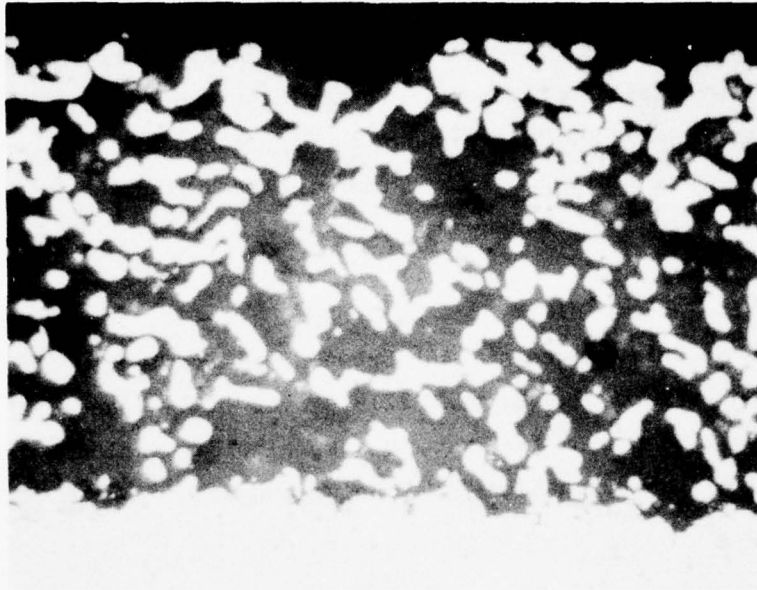


Figure 8

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Pre-alloyed NiCrAlY coating on Mar-M006 DS sintered at 1149 C/4 hours
in a vacuum.

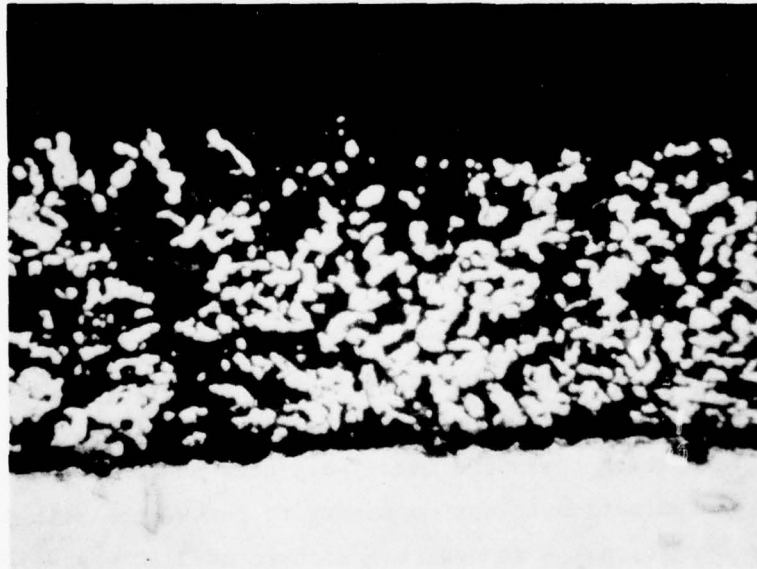


Figure 9

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Pre-alloyed CoCrAlY coating on Mar-M006 DS sintered at 1149 C/4 hours
in a vacuum.

Significant improvements were obtained in bonding and sintering on CoCrAlY, NiCrAlY and CoCrY coatings. NiCrY which was the best at 1149C sintering temperature showed less improvement. Photomicrographs of CoCrAlY and NiCrAlY coating on Mar-M006 DS are shown in Figures 10 and 11, respectively. Coating thickness was reduced, illustrating the need for a double AEP deposition to obtain increased thickness.

Visual examination showed NiCrY and CoCrY had a metallic luster, however, the CoCrAlY and NiCrAlY showed a light brown discoloration on the surface. The reduction in surface oxidation at 1219 C over 1149 C suggests that the oxidation may have been from the ceramic sample holder included in both runs which had less contamination after being outgassed in the first 1149C/4 hour run.

TDNiCr Samples

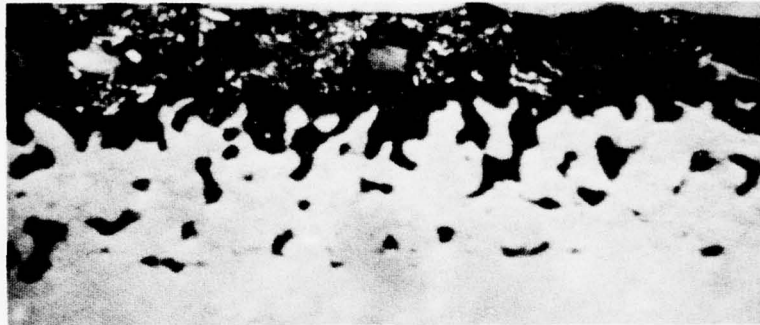
CoCrY coating showed a slight improvement in sintering over the 1149 C run, however, the bonding to the base metal was poor. A NiCrAlY sample also showed poor bonding.

Electron Microprobe Examination

Electron microprobe examination of several of the coated samples showed some depletion of chromium in the coating. Nickel diffusion into the coating and cobalt and aluminum diffusion into the base metal was also observed. EBS scans of CoCrY on TDNiCr and CoCrAlY on TDNiCr and Mar-M006 DS are shown in Figures 12 - 14. The percentage of yttrium in the powders is below the detectability of the electron microprobe used.

Furnace Contamination from Zein

At completion of the 1219C/4 hr run, contamination was found in the vacuum furnace and in the diffusion pump oil. It was concluded this came from the zein in the AEP bath which coats the metal particles and acts as a binder. To prevent this contamination it was necessary to remove the zein in a separate furnace operation. A removal temperature of 371C was selected since previous experiments indicated volatilization of the zein was complete below 371C and all zein odor ceased below this temperature. An initial run was made in hydrogen and subsequent to this, runs were made in vacuum to remove the zein more completely.



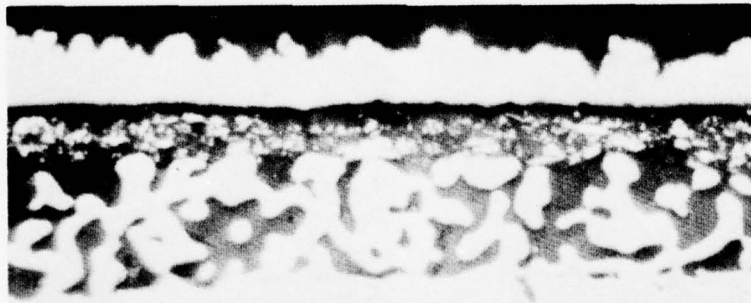
Plating

Coating

Figure 10

Magn: 500X

Photomicrograph of CoCrAlY coating on Mar-M006DS after 1219 C/4 hours vacuum sintering cycle.



Plating

Coating

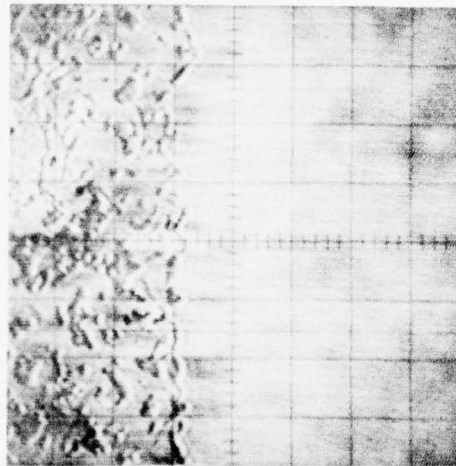
Figure 11

Magn: 500X

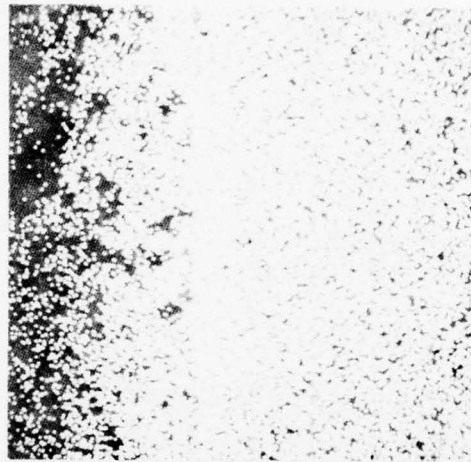
Photomicrograph of NiCrAlY coating on Mar-M006DS after 1219 C/4 hours vacuum sintering cycle.

TDNiCr CoCrY COATING 2225F/4 HOURS

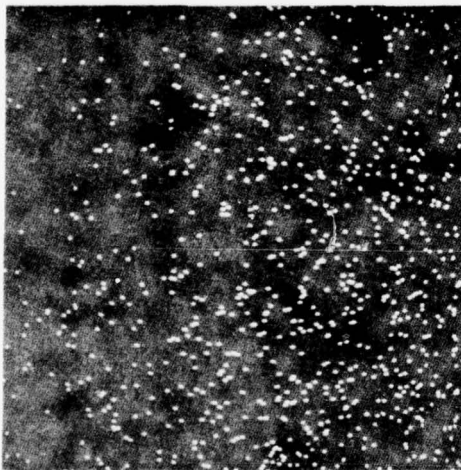
EBS SCANS 250X



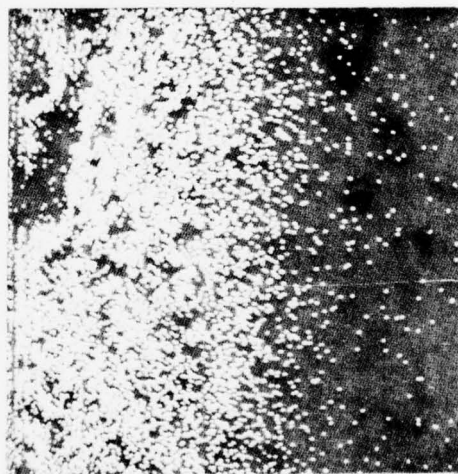
NSC



Ni



CR

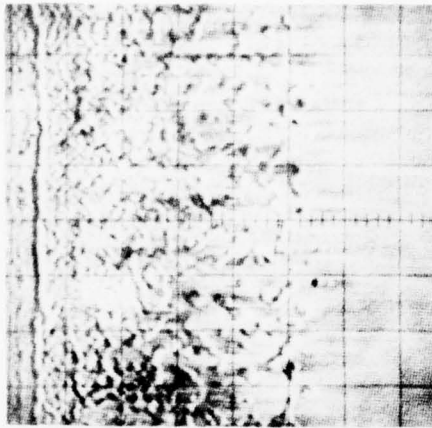


Co

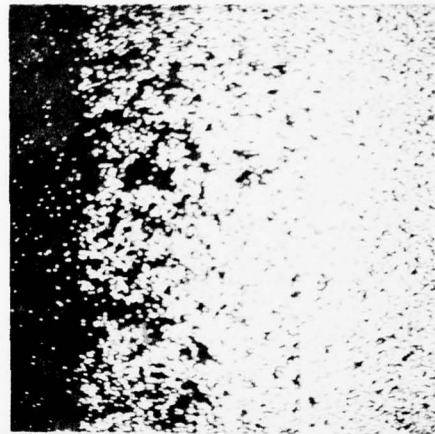
Figure 12 CoCrY coating on TDNiCr

TDNiCr CoCrAlY COATING 2225F/4 HOURS

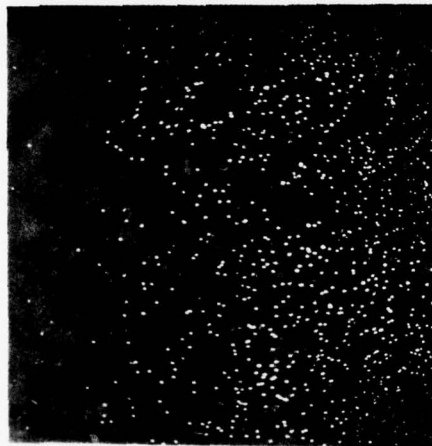
EBS SCANS 250X



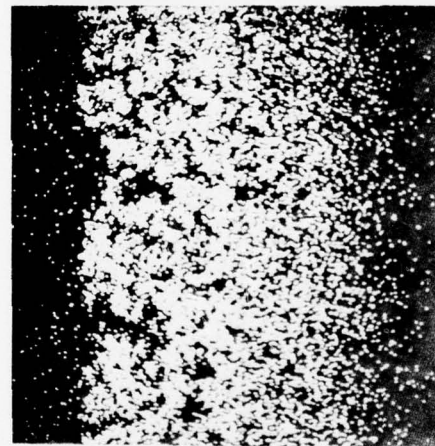
NiSC



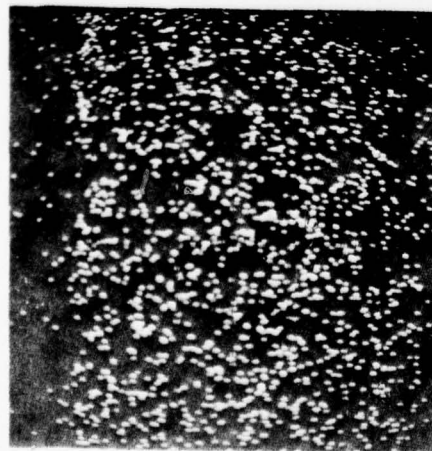
Ni



Cr



Co

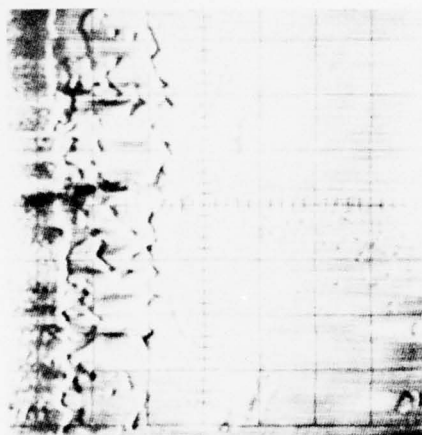


Al

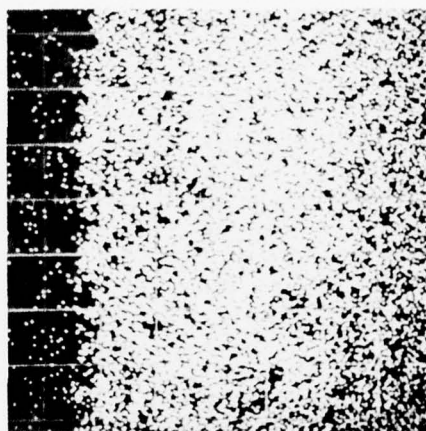
Figure 13 CoCrAlY coating on
TDNiCr

MAR M-006 CoCrAlY COATING 2225F/4 HOURS

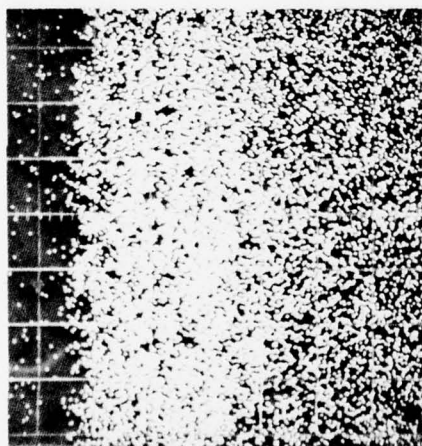
EBS SCANS 250X



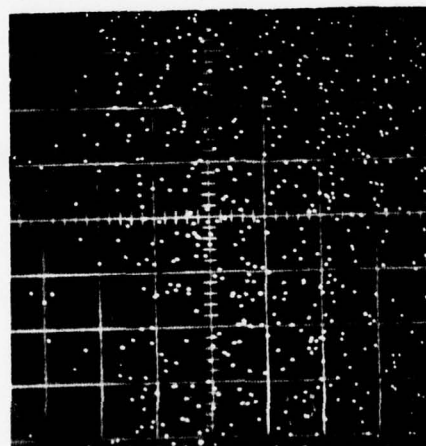
NiSC



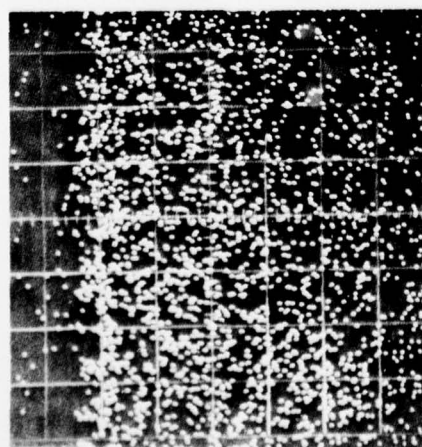
Ni



Co



Cr



Al

Figure 14 CoCrAlY coating
Mar-M006

Vacuum Sintering 371C/2 hour + 704C/2 hours + 1002C/1 hour (500 μ Argon) + 1219C/2 hours (1000 μ Argon)

Sintering time at the 1219C temperature was cut from 4 to 2 hours to reduce coating-base metal interdiffusion and a separate 700F (371C) zein removal cycle was used.

Ten percent (10%) aluminum was added to the NiCrY powder to improve sintering and bonding and obtain a NiCrAlY coating on TDNiCr. Two hours at 704C and an hour at 1002C were added to initiate the formation of nickel-aluminum phases in the NiCrY at a lower temperature. A CoCrAlY coated TDNiCr specimen was also included in the run.

TDNiCr

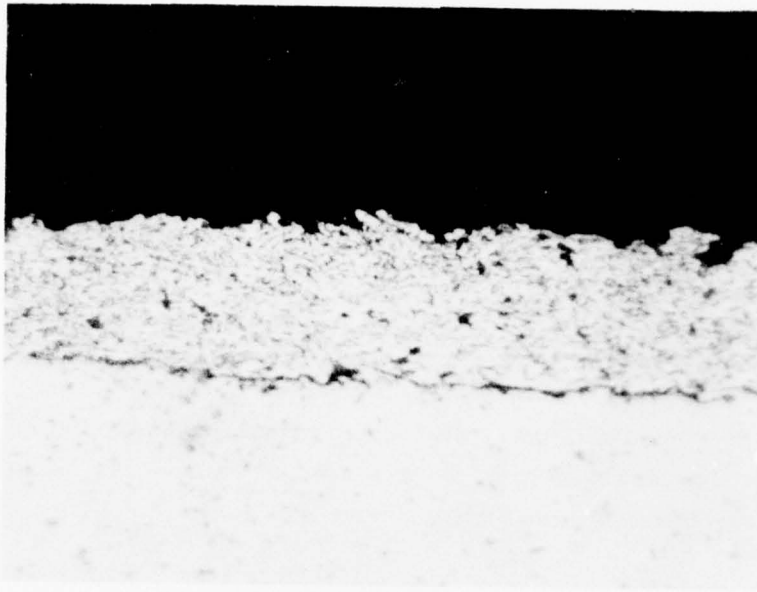
The NiCrY + 10% Al coated TDNiCr specimen showed no visual evidence of oxidation upon removal from the furnace. Microexamination showed a partially bonded coating and no improvement in sintering resulting from the 10% aluminum addition. Microprobe analysis indicated the aluminum had diffused into the inner portion of the coating adjacent to the TDNiCr alloy. CoCrAlY on TDNiCr exhibited an improvement in bonding and sintering over previous vacuum cycles.

Vacuum Sintering 371C/2 hour + 704C/2 hour + 1038C/3 hour (800 μ Argon) + 1038C/16 hour (Vacuum)

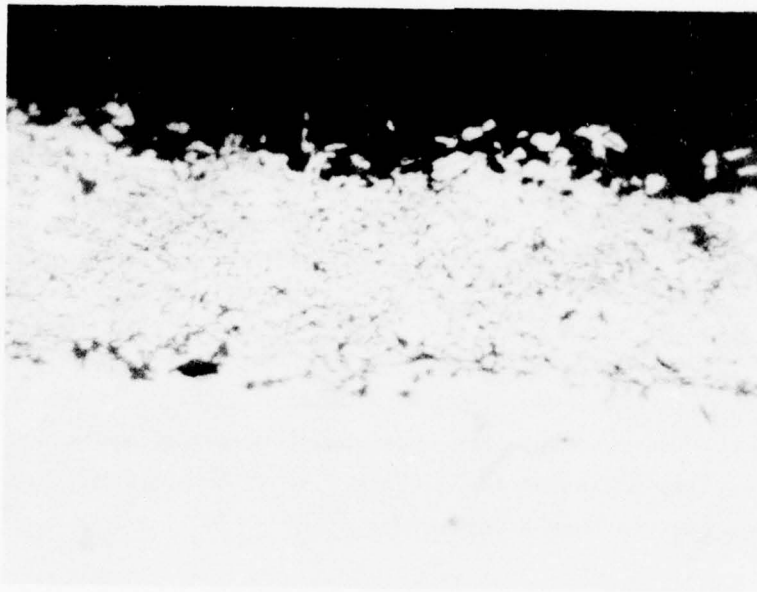
A second vacuum cycle was run with the NiCrY + 10% Al coating on TDNiCr and Mar-M006 DS. To reduce the aluminum diffusion to the base metal encountered on the previous run, the sintering temperature was reduced to 1038C and the time increased to 19 hours. NiCrY and NiCrAlY coatings were included on the two base alloys.

Mar-M006 DS

NiCrY + 10% Al and NiCrAlY coatings showed a significant improvement in consolidation of the coatings compared with previous sintering at 1149C and 1219C. NiCrAlY appeared to have the better bond of the two compositions. These coatings are shown in Figure 15. NiCrY coating showed no improvement in sintering and a poorer bond. See Figure 15.



NiCrY + 10% Al Coated Mar-M006 DS



NiCrAlY Coated Mar-M006 DS

Figure 15

Magn: 500X

Coatings on Mar-M006 DS after 2 371C/2 hr + 704C/2 hr + 1038C (800 μ Argon)
+ 1038C/16 hr vacuum sintering cycle.

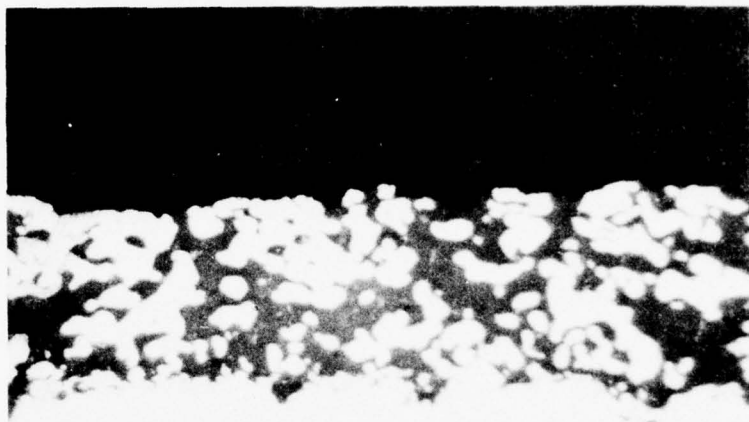


Figure 16

Magn: 500X

NiCrY coated Mar-M006 DS after a 371C/2hr + 704C/2 hr + 1038C/3 hr
(800 μ argon) + 1038C/16 hours vacuum sintering cycle

TDNiCr

Unlike the Mar-M006DS alloy, NiCrY + 10% Al and NiCrAlY coatings on TDNiCr exhibited an increased porosity and reduction in base metal bond with the 1038C sintering temperature. NiCrY coating was approximately equivalent to coatings sintered at the higher temperatures.

Mechanical Densification of Sintered Coatings

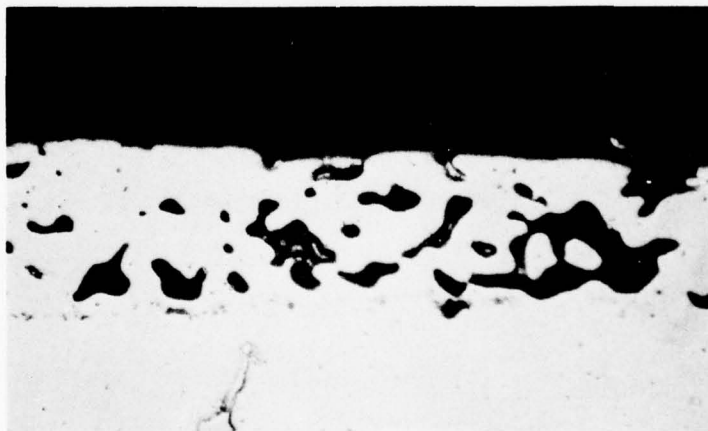
The sintered coatings were glass bead peened to decrease porosity. Coatings peened were from the 1219 C vacuum sintering run. In the initial attempts to peen for densification, number 13 glass beads were used with a pressure of 15 psi for 1 minute. Only slight deformation of the surface resulted. Figure 17 shows the deformation of the NiCrY coating on TDNiCr and the CoCrY coating on Mar-M006DS. An additional glass bead peening step with #13 beads for one minute and the pressure increased to 25 psi resulted in a significant improvement in densification with deformation extending through the coating. Figure 18 shows the NiCrY coated TDNiCr after the double peening. Increasing the glass bead size to the larger #6 bead at 25 psi and reduction of the time to 5 seconds resulted in consolidation and deformation completely through the coating. Figure 19 shows the CoCrAlY coated TDNiCr specimen after peening with No. 6 beads. As a further densification step some of the peened coatings were given a 1066 C for 4 hours treatment in hydrogen. This operation would improve density, however, due to a break in the furnace line some contamination of the coatings occurred. Figure 20 shows the NiCrY + 10% Al coating on TDNiCr after compacting with the #6 beads and after the 1950 F heat treatment.

Vacuum Furnace Contamination

To obtain samples for subsequent densification by peening vacuum sintering runs were made with a zein removal cycle of 371 C/2 hrs followed by vacuum sintering at 1219 C with a partial argon backfill. MCrY and MCrAlY coatings were powdery and oxidized upon removal from the furnace. A fine dark deposit was present on the vacuum chamber walls. It was felt that the zein removal cycle should be further investigated to determine if a portion of the zein removal cycle should be further investigated to determine if a



NiCrY coated TDNiCr-1219C/4 hr #13 glass beads, 15 psi -
1 minute.



CoCrY coated Mar-M006DS-1219 C/4 hr #13 glass beads, 15 psi -
1 minute.

Figure 17

Magn: 500X

Glass bead peened coatings showing deformation on outer surface.



Figure 18

Magn: 500X

NiCrY/TD NiCr - 1219 C/4 hr (vacuum) #13 glass beads, 15 psi 1 minute +
#13 glass beads, 25 psi - 1 minute.

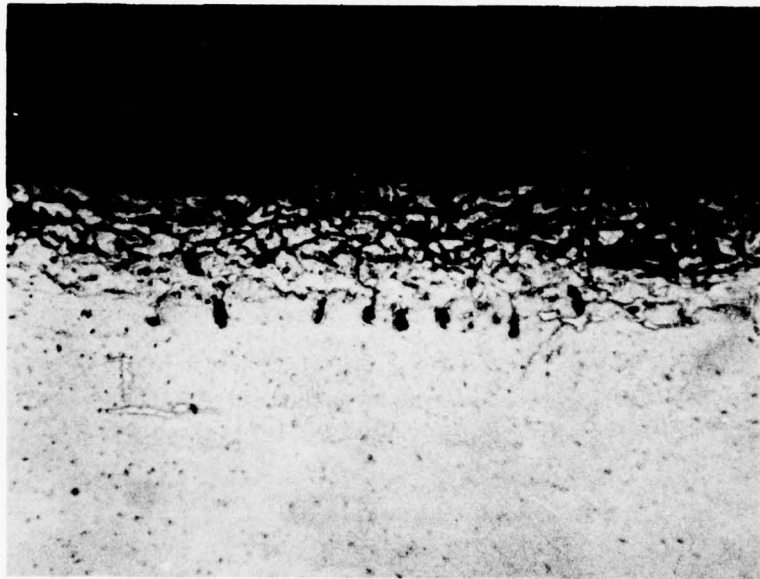
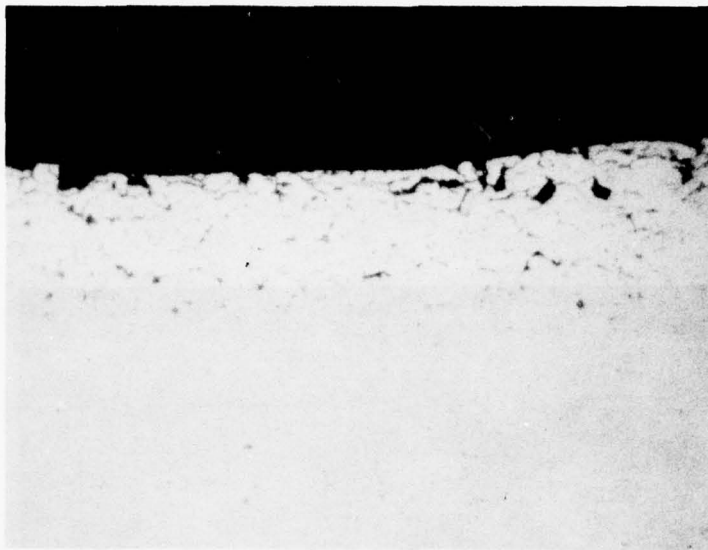


Figure 19

Magn. 500X

CoCrAlY coated TDNiCr 371 C/2 hr + 704 C/2 hr (vac) + 1002 C/1 hr (500 μ)
+ 1219 C 2 hr (1000 μ argon) -- peened #6 glass beads, 25 psi - 5 seconds.



Magn: 500X

NiCrY + 10% Al - coated TDNiCr - 371 C/2 hrs + 704 C/2 hr (vac) + 1002 C/1 hr (500 μ argon) + 1219 C/2 hrs -- glass bead peened #6 beads, 25 psi - 5 seconds

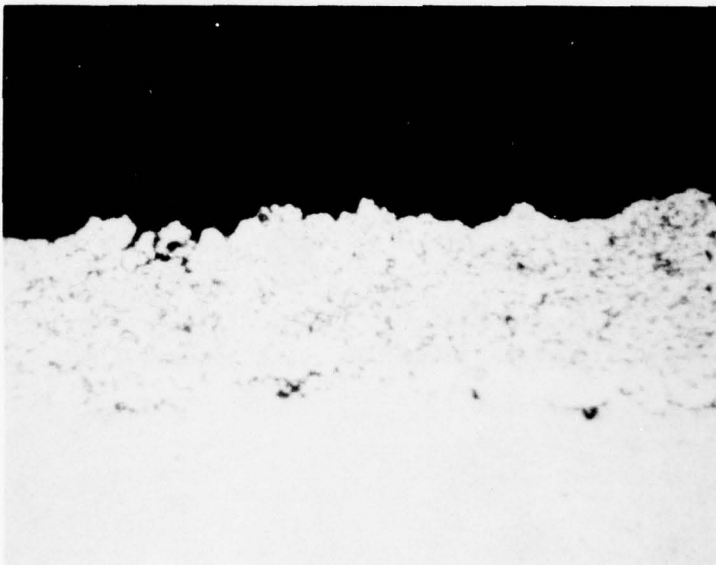


Figure 20

Magn: 500X

NiCrY + 10% Al - coated TDNiCr - 704 C/2 hr (vac) + 1002 C/1 hr (500 μ argon) + 1219 C/2 hr -- glass bead peened, #6 beads at 25 psi, 5 sec - 1066 C/4 hrs (hydrogen)

portion of the zein still remained after 371 C and was contaminating the vacuum furnace. Experiments were conducted in hydrogen and vacuum with zein coated specimens of Mar-M006DS to determine if the zein was completely removed by the 371 C cycles previously used. At 371 C/2 hrs approximately 62% of the zein weight remained and a dark varnish remained on the sample. After 2 additional hours at 538 C/2 hrs the zein weight remained essentially unchanged and the varnish darkened in color. After exposure of the sample for an additional 760 C/2 hrs the weight of the coated specimens indicated complete removal of the zein and the sample was free of the surface varnish. This indicated a temperature in the 760 C range was necessary for zein removal.

In the next experiment two Mar-M006DS specimens were coated with NiCrY on CoCrY pre-alloyed powders. The specimens were taken to 760 C/2 hrs in a vacuum. Observation of the specimens after the 760 C/2 hr treatment revealed that the surface of the prealloyed particles were found oxidized but the basic metal under the coating was clean indicating possible contamination from decomposition of the zein.

In similar experiments Mar-M006DS specimens coated with pre-alloyed NiCrY and CoCrY were heat treated in hydrogen at 760 C/2 hrs. After removal from the furnace, the prealloyed particles had a metallic lustre. However, underneath the particles oxidation was present on the aluminum and titanium containing base metal. In subsequent zein removal experiments titanium and magnesium were both used as getters to prevent oxidation during the zein removal cycle. Titanium foil surrounded two specimens coated with pre-alloyed NiCrY and CoCrY. After two hours at 760 C in an argon atmosphere the powders and base metal were free of oxidation. A specimen of Mar-M006DS was taken to 760 C/2 hrs in hydrogen using magnesium as a getter. No oxide was visible after the cycle, indicating the necessity of gettering the furnace atmosphere during the zein removal cycle to prevent oxidation.

Vacuum Sintering at 1219 C after Zein Removal

Another vacuum sintering cycle at 1219 C/4 hrs was run to see if the pre-alloyed particles would sinter and bond in a vacuum after complete zein removal. Specimens of Mar-M006DS were AEP coated with CoCrY, NiCrY, CoCrAlY

and NiCrAlY. Another specimen of Mar-M006DS was coated with Universal Cyclops chemically activated NiCrY.

Prior to vacuum sintering, zein was removed by heating the specimens in a titanium gettered argon atmosphere at 760 C/2 hours.

After zein removal, the coated specimens were placed in a vacuum furnace with titanium foil for gettering any oxygen which might be in the furnace. The furnace was then taken to 2225 F/2 hours.

Microexamination of the vacuum sintered specimens revealed that the NiCrY particles had only partially bonded and sintered. NiCrY particles that had been chemically activated had the best sintering and bonding. CoCrY particles were sintered with some bonding to the base metal. CoCrAlY particles showed some interparticle sintering with no bond to the base metal. NiCrAlY particles showed sintering but very limited bonding. NiCrY and chemically activated NiCrY coating and chemically activated NiCrY on Mar-M006DS is shown in Figures 21 and 22, respectively. All coatings were porous.

It was concluded that removal of the zein prior to sintering did not improve bonding and sintering over a previous run with zein in the coating. The aluminum bearing pre-alloyed particles did not bond or sinter as well as the non-aluminum bearing powders.

Aluminum Pack Sintering

Pack aluminizing cycles were initiated to densify and aluminize AEP NiCrY and CoCrY deposited pre-alloyed particles. NiCrAlY powders were included in packs 2, 3 and 4 to see if they could be densified. The ammonium chloride and aluminum contents of the pack were varied as shown below:

Pack No.	PACK COMPOSITIONS		
	Al_2O_3	NH_4Cl	Al
1	Bal	5	10
2	Bal	5	5
3	Bal	5	--
4	Bal	1/4	5

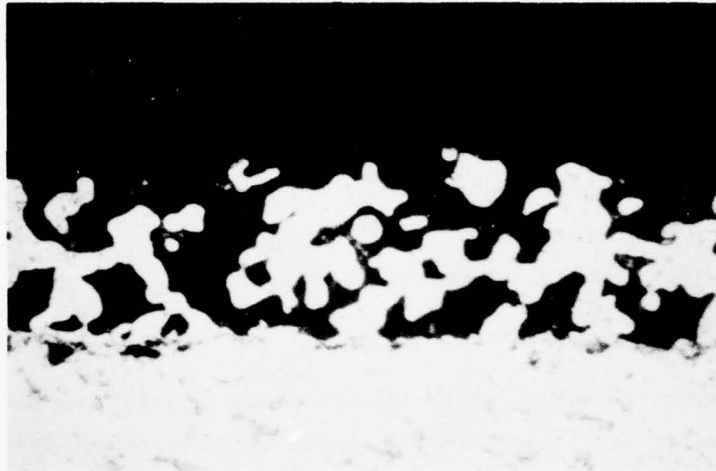


Figure 21

Magn: 500X

Pre-alloyed NiCrY on Mar-M006DS sintered in a vacuum at 1219 C/4 hours

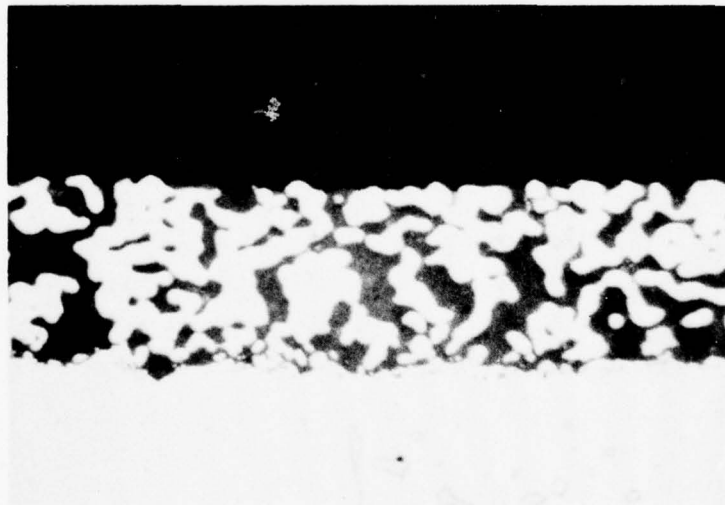


Figure 22

Magn: 500X

Pre-alloyed chemically activated NiCrY on Mar-M006DS sintered in a vacuum at 1219 C/4 hours.

AEP coated specimens in these packs were first heated to 760C/2 hrs in a hydrogen atmosphere for removal of the zein. The pack cycle consisted of 16 hours at 1038 C in a hydrogen atmosphere and cooling to room temperature in hydrogen. Microexamination of the pack specimens revealed aluminum penetration through the pre-alloyed particles into the Mar-M006DS base metal with negligible aluminizing and bonding of the particles. This is shown in Figure 23 showing NiCrY particles on the outside of the aluminum diffusion coating formed. This was typical of all four pack runs.

Modified Packs

The modified packs were modeled after the prototype aluminizing pack number 2 reported in Air Force Materials Laboratory Contract F33615-75-C-5268 by the Solar Division of International Harvester. Makeup composition is shown below:

COMPOSITION (W/o)

<u>Co</u>	<u>Cr</u>	<u>Al</u>	<u>Al₂O₃</u>	<u>NH₄Cl</u>
16	5	4	74.75	0.25

Mixed pack material was placed in a Haynes 188 cobalt base alloy retort and a sleeve containing titanium chips was placed upstream from the pack for oxygen getting.

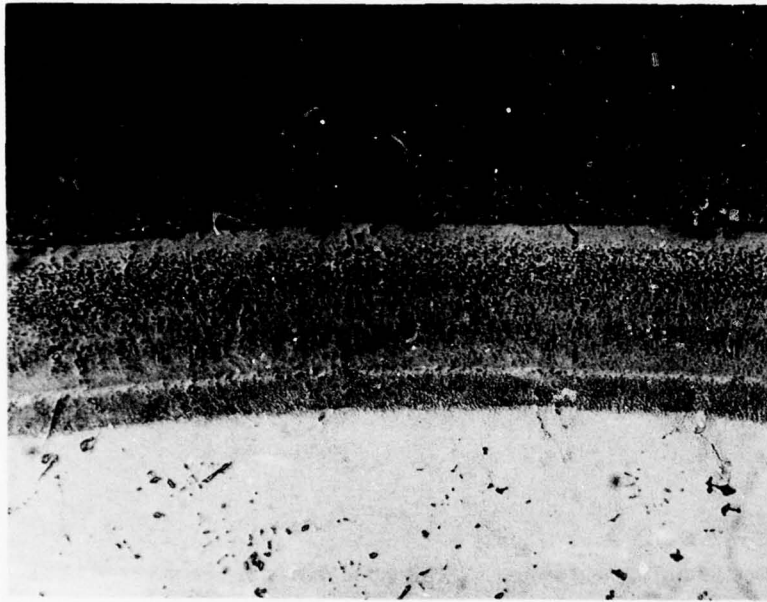
Argon (-50 dewpoint) was flowed through the furnace for 20 minutes and the furnace was then vacuum evacuated for 15 minutes. This cycle was repeated three times. With argon flowing, the packs were taken to 907 C for 16 hrs for Pack No. 1 and 1130 C for 16 hours for Pack Nos. 2-5 and then cooled in argon.

Modified Pack Analysis

Pack analysis was made by atomic absorption from the aqua regia soluble portion of the conditioned pack. Results are shown in Table 2.

Zein Removal from AEP Coating Prior to Modified Sintering Pack

All specimens used in the modified pack sintering experiments were heated in titanium "gettered" argon atmospheres to 760 C for 2 hrs for zein removal.



Pre-alloyed powder

Aluminide coating

Figure 23

Magn: 200X

Pre-alloyed NiCrY particles on Mar-M006DS base alloy after being pack cycled through 704 C and sintered for a 1038 C/16 hours.

Table 2

Modified Pack Analysis (W/o)

<u>Pack No.</u>	<u>Co</u>	<u>Cr</u>	<u>Al</u>	<u>Fe</u>	<u>Ni</u>	<u>Al₂O₃</u>
1 Start	15.40	4.70	3.80	0.4	0.1	Bal
1 Conditioned	14.50	3.20	3.40	0.3	---	Bal
2 Conditioned	15.65	4.80	3.15	0.2	---	Bal
3 Start	16.20	5.10	4.00	0.2	---	Bal
3 Conditioned	16.00	4.70	3.20	0.2	---	Bal
3 After Testing	15.80	4.80	3.15	0.2	---	Bal
4 Conditioned	12.3	3.70	2.65	0.3	.4	Bal
4 After Coating	15.1	4.55	3.28	0.4	.4	Bal
5 Start	15.20	4.85	3.70	0.3	---	Bal
5 Conditioned	15.10	4.70	3.20	0.4	---	Bal
5 After Coating	15.10	4.55	3.00	0.4	---	Bal
5 After Replenishing	18.3	7.00	5.50	0.6	---	Bal
5 After Coating	18.6	7.00	5.40	0.6	---	Bal

Modified Pack 1

Specimens Mar-M006DS AEP coated with CoCrY and NiCrY after zein removal at 704°C/2 hr. were placed in the conditioned pack material in the retort used to condition the pack. The furnace atmosphere and purging cycle were the same as used to condition the packs. The specimens were then taken to 1030°C in 2 1/2 hours. The pack was held at this temperature for 2 1/4 hours and after the power was turned off, the argon flow was continued until the pack was cooled to room temperature.

Microexamination of the specimens showed no bonding of the particles to the base metal or among the particles. On the bare fastener end which had not been coated with particles, microexamination revealed only .0003" of diffused aluminum. Because of the thin coating found in the previously bare area, temperature of the pack was too low to adequately coat specimens.

Modified Pack 2

To get more efficient aluminizing the pack temperature was increased to 1130°C. Pre-alloyed NiCrY and CoCrY were deposited on both Mar-M006DS and IN 713C alloys which had been etched for 15 minutes with a 13.7% HCl, 1.0% HF, 4.1% H₂O₂ and Balance H₂O etchant. After zein removal, specimens were run in a conditioned pack at 1130°C/2 1/4 hours. Microexamination of the coated specimens revealed no bonding or densification on either alloy.

Modified Pack 3

The purpose of this experiment was to determine if the pre-alloyed particles would bond without having been AEP coated or through a zein removal cycle. It was thought possible that oxidation of the particles might have occurred during this cycle and prevented bonding. Specimens of IN 713C alloy were used in this experiment and were etched 15 minutes as in the preceding pack run. NiCrY, CoCrY, NiCrAlY and CoCrAlY were placed on the concave side of the etched specimens and leveled. A pack was conditioned as before at a temperature of 1130°C. The specimens with the powders were then placed in the conditioned pack cycled 2 1/2 hours to 1130°C and held at temperature 2 1/4 hours as before.

Modified Pack 4

To determine if chemically activated particles would bond and densify in a modified pack, pre-alloyed NiCrY chemically activated with a proprietary process by Universal Cyclops was used. Chemically activated NiCrY was placed on an IN 713C specimen and the powder leveled. Another IN 713C specimen was AEP coated with NiCrY (UC). The specimens were then diffused in a modified pack No. 4 at 1130°C for 2 1/4 hours.

Microexamination of the specimens revealed no bonding or densification of the specimen which was electrophoretically alloy coated. The specimen with the powders placed on the specimen and leveled showed interparticle bonding and consolidation of the chemically activated particles but no bond to the base alloy.

The chemically activated NiCrY showed improvement in interparticle densification over the standard NiCrY run in pack No. 2 when the AEP coating and zein removal cycle was omitted, however a bond was still not achieved. With the electrophoretic coating and zein removal cycle, the chemically activated NiCrY showed no advantage over the standard powder.

Modified Pack 5

Pack 5 was mixed in the same manner and with the composition of the preceding packs. This pack was used to sinter and densify the following isostatically pressed runs.

Isostatic Pressing

An isostatic press built by Auto Clave Engineers with a pressure capacity of up to 80,000 psi was used in the isostatic pressing experiments. The press is shown on page 59 in the Appendix. Isostatic pressing was used to densify the AEP deposited specimens after the removal of zein. Isostatic pressing was done in a cylinder approximately 4 inches in diameter and 20 inches long. Specimens were pressed at room temperature in water soluble oil. The pressure used was 54,000 psi. Pieces were pressed for approximately 15 seconds. Zein was removed prior to pressing.

All alloys used in the isostatic press experiments were etched in the etch previously reported prior to AEP coating for 1 1/2 minutes, except in the first experiment where samples were etched for fifteen minutes.

Isostatic Press 1

Pre-alloyed chemically activated NiCrY was AEP deposited on a specimen of IN 713C alloy. The as-deposited coating had a grey metallic luster and was quite adherent in the as-deposited condition.

After zein removal, the coated specimen was bagged with GE 664 RTV synthetic rubber by dipping the heated specimen (325°F) in the synthetic rubber. Curing of the rubber was hastened by holding the specimen in hot air from a hair dryer. The bagged specimen was then cold isostatic pressed at 54,000 psi for 15 seconds. After pressing, the bag was removed from the specimen. Pre-alloyed powder locally adhered to the bag with approximately 15% of the area of the blade left bare. Figure 24 shows the densification of the coating after pressing.

One portion of the specimen was sintered in hydrogen and the other was sintered in a vacuum at 1093°C/1 hour. The temperature was raised slowly over a two hour period to 1000°F to reduce the effect of escaping gases from opening up porosity. Figure 25 shows the coating after hydrogen sintering. The coating showed considerable porosity.

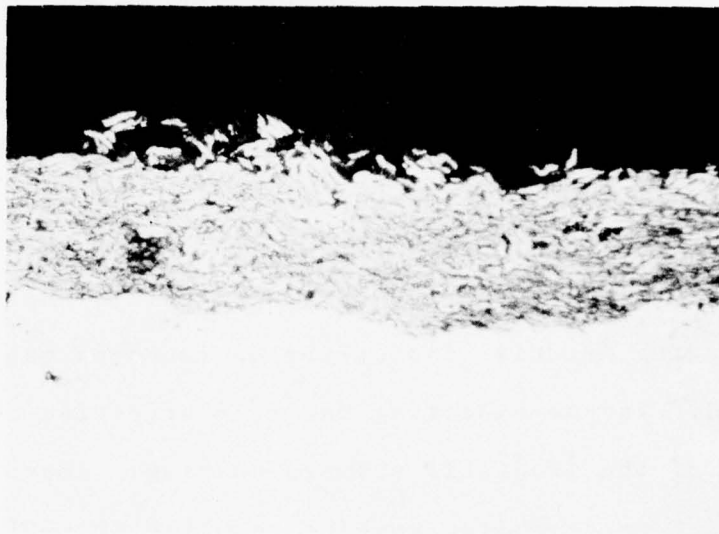


Figure 24

Magn: 1000X

NiCrY chemically activated coating on IN 713C after zein removal
and cold isostatic pressing at 54 ksi

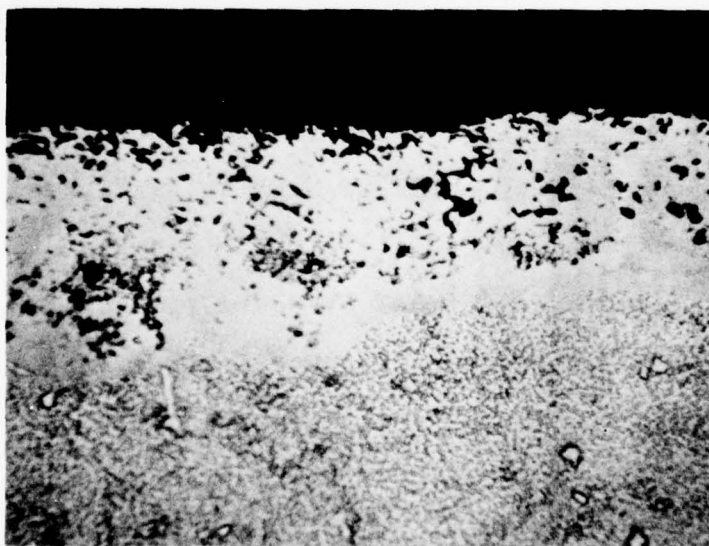


Figure 25

Magn: 1000X

NiCrY chemically activated coating of IN 713C pressed and sintered
in a hydrogen at 1093°C/1 hour

The vacuum sintered specimen exhibited a much denser coating. Figure 26 shows two airfoil locations on the vacuum sintered specimen. A much brighter metallic luster was noted on the vacuum sintered specimen. A coating thickness of 1 to 2 mils was achieved from an original deposit of 19 mgs/cm².

Isostatic pressing the electrophoretic coatings significantly increased coating density however some powder was pulled off by the bagging material indicating an improved bagging technique was needed. Vacuum sintering was more effective than hydrogen sintering of the isostatic pressed coating. Approximately 19mgs/cm² of as-deposited coating resulted in .001 inch to .002 inch of sintered dense coating.

Isostatic Press 2

This run was made to compare coating bond and densification on Mar M006DS with the previously coated IN 713C alloy and to investigate the effectiveness of Cerac Fluorocarbon, SP 102, sprayed on the coating prior to bagging, as a release agent to aid in a bag removal.

After zein removal, specimens of CoCrAlY, NiCrAlY and NiCrY on Mar M006DS were bagged with GE 664/RTV synthetic rubber. Additional specimens of NiCrY, CoCrY, and CoCrAlY with the zein removed were sprayed with Cerac Fluorocarbon SP 102 which was intended to act as a release agent for the synthetic rubber bagging. The specimens were then bagged with GE 664 RTV synthetic rubber. All specimens were isostatically pressed at 54,000 psi for 15 seconds.



Lower Airfoil

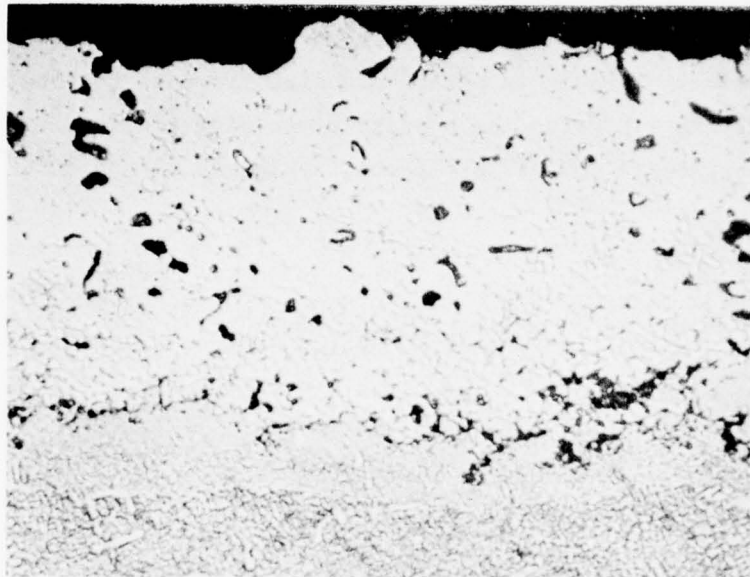


Figure 26 Upper Airfoil Magn: 1000X

NiCrY chemically activated coating on IN 713C pressed and sintered in a vacuum at 1093 C/1 hour.

Coatings on the CoCrAlY, NiCrAlY and NiCrY coated Mar-M006DS specimens had the coating adhere to the GE 664 RTV rubber during bag removal and almost all the coating came off with the bags. Apparently, the pre-alloyed coating had not bonded as well to the Mar-M006DS alloy as the NiCrY coating had on IN 713C alloy in the previous press run No. 1

Removal of bags from the fluorocarbon sprayed NiCrY and CoCrAlY resulted in only a small amount of coating coming off with the bags. The CoCrY bag developed a leak during the press cycle and did not press. The fluorocarbon spray was an effective release agent for bag removal. Microexamination of the pressed NiCrY and CoCrAlY samples showed porosity indicating the fluorocarbon may have penetrated the coating and retarded pressing. After pressing the CoCrAlY and NiCrY coatings were sintered in a vacuum with a 2 hour rise time to 538 C followed by 1 hour at 1093 C.

Microexamination revealed bonding and partial sintering of the NiCrY coating. The coating was porous. Figures 27 and 28 show the as-pressed and the sintered NiCrY coating. No bonding or sintering was observed in the CoCrAlY coating. This is in accordance with previous results which showed difficulty in sintering the powders containing aluminum. The as-pressed and the sintered CoCrAlY are shown in Figures 29 and 30.

Isostatic Press 3

The purpose of this experiment was to press and sinter chemically activated NiCrY on Mar-M247DS alloy and to compare another bagging material, Turco TFM-5580-6 with GE 664.

Two Mar-M247DS alloy test blades were coated with chemically activated NiCrY. Coating deposition of the two specimens were 18 mgs/cm² each. After zein removal at 760 C/2 hours, one specimen was bagged with GE 664 and the other with Turco TFM-5580-6.

The Turco bagging was removed at room temperature with perchloroethylene. G.E. 664 RTV was peeled off. Some coating came off with the GE 664. Almost all the NiCrY coating remained after removal of the Turco bagging.

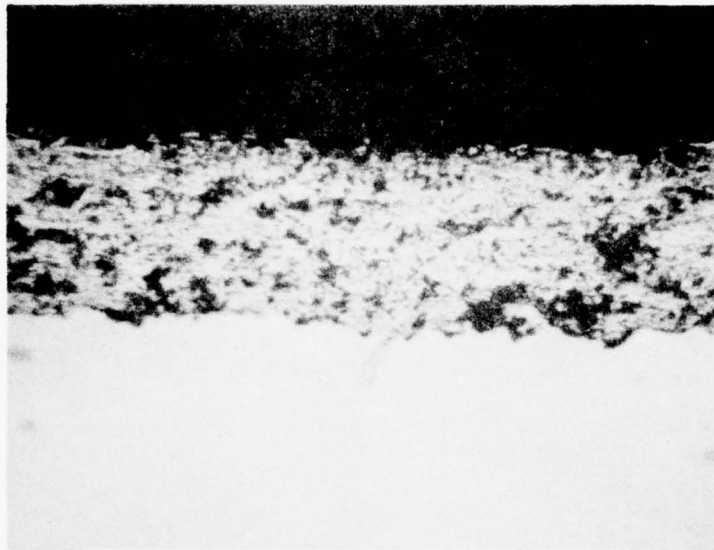


Figure 27

Magn: 500X

Pre-alloyed NiCrY coating on Mar-M006DS alloy sprayed with Cerac Fluoro-carbon SP 102, bagged with GE 664 RTV and cold isostatically pressed.

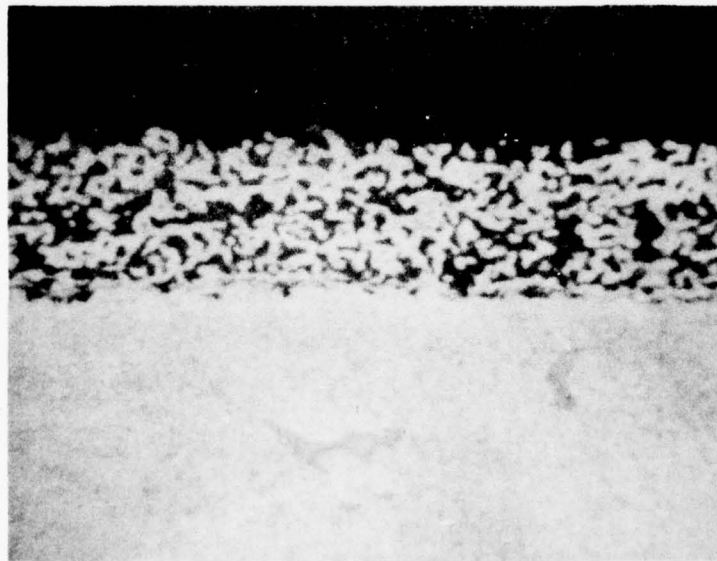


Figure 28

Magn: 500X

Pre-alloyed NiCrY coating on Mar-M006DS sprayed with Cerac Fluorocarbon SP 102 and bagged with GE 664 RTV, room temperature isostatic pressed at 54 ksi and sintered in a vacuum at 1093 C/1 hour in vacuum.

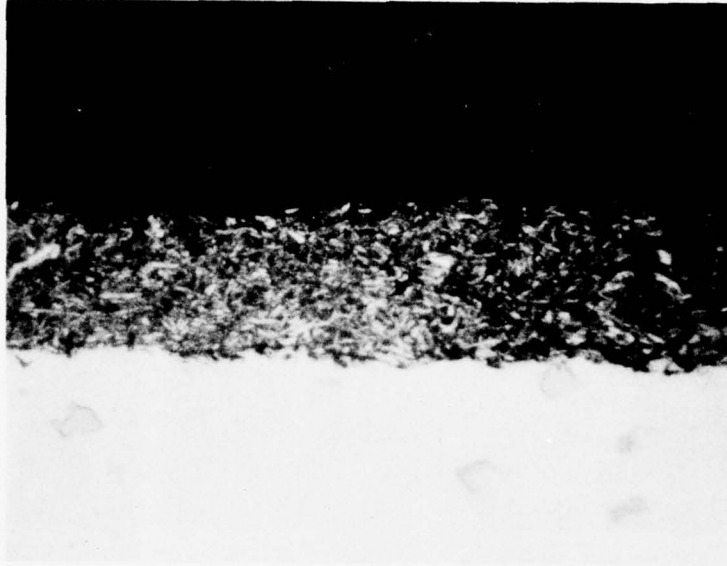


Figure 29

Magn: 500X

Pre-alloyed CoCrAlY coating on Mar-M006DS sprayed with Cerac Fluorocarbon and bagged with GE 664 RTV cold isostatic pressed at 54 ksi.

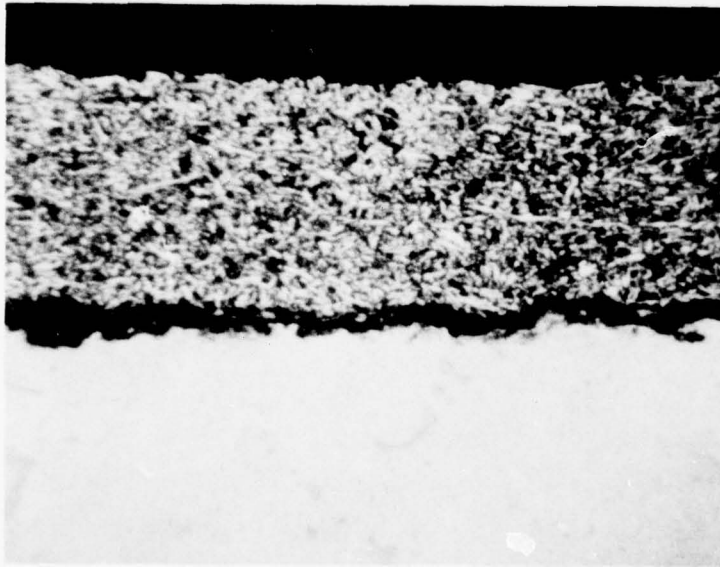


Figure 30

Magn: 500X

Pre-alloyed CoCrAlY coating on Mar-M006DS sprayed with Cerac Fluorocarbon SP 102 and bagged with GE 664 RTV, room temperature isostatic pressed at 54 ksi after 1093 C/1 hour.

Microexamination of the specimens showed the GE 664 RTV bagging produced the best density in the as-pressed coating. The specimen bagged with Turco was not nearly as dense. Figure 31 and 32 shows the pressed coatings bagged in GE 664 RTV and Turco, respectively.

The as-pressed specimens were sintered in a vacuum at 1093 C/1 hour. The specimen bagged in GE 664 was far less porous than the specimen bagged in Turco. Figures 33 and 34 show the two specimens, respectively.

Isostatic Press 4

This experiment was run to develop improved bagging techniques. The GE 664 bag has caused some loss of coating material during removal. The zein removal temperature was increased to promote better green strength to aid in removal of the bag after pressing. Chemically activated NiCrY was AEP deposited on a Mar-M247DS test blade and two specimens of Mar-M006DS. After zein removal at 871 C, the coated Mar-M247DS specimen was bagged with Ambi Polyvinyl Chloride evacuated and sealed with a rubber band. The second specimen, coated with activated NiCrY on Mar-M006DS was bagged with latex rubber, evacuated and sealed with a rubber band. The third specimen was wrapped with aluminum foil, bagged with latex rubber and sealed with a rubber band. The specimens were then isostatic pressed at 54,000 psi for 15 seconds at room temperature. After pressing, the bags were removed. The Ambi Polyvinyl Chloride bag was the easiest to remove, leaving the coating totally intact. It was noted that the bag had leaked. The latex rubber bag was easy to remove and no coating adhered to the rubber bag. The specimen wrapped in aluminum foil had the smoothest appearance of these specimens. The aluminum came off the pressed coating without any coating adhering and was easy to remove.

Microexamination of the pressed NiCrY (UC) coating showed the aluminum foil bagged and the latex bagged specimens had significantly improved density compared with the previous press runs. These are shown in Figures 35 and 36. The polyvinyl chloride bagged specimen showed considerable porosity due to leakage during the press run.

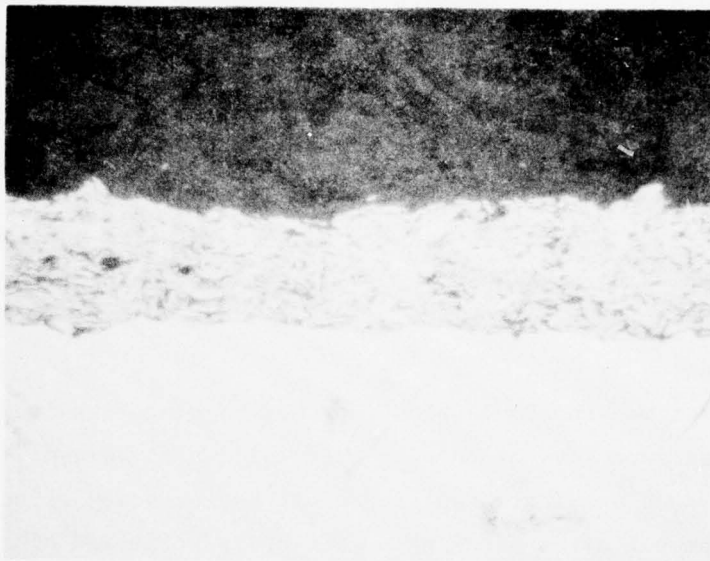


Figure 31

Magn: 500X

NiCrY chemically activated coating on Mar-M247DS bagged with GE 664 and pressed at 54 ksi/15 seconds.

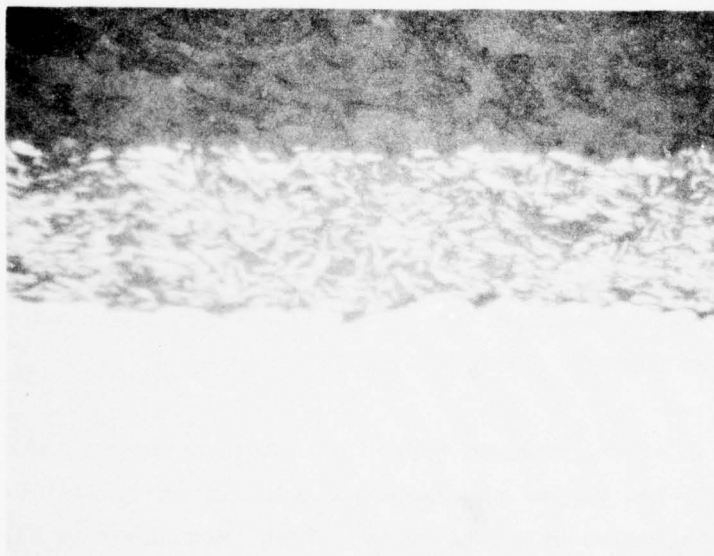


Figure 32

Magn: 500X

NiCrY chemically activated coating on Mar-M247DS bagged with Turco TFM5580-6 as pressed at 54 ksi.

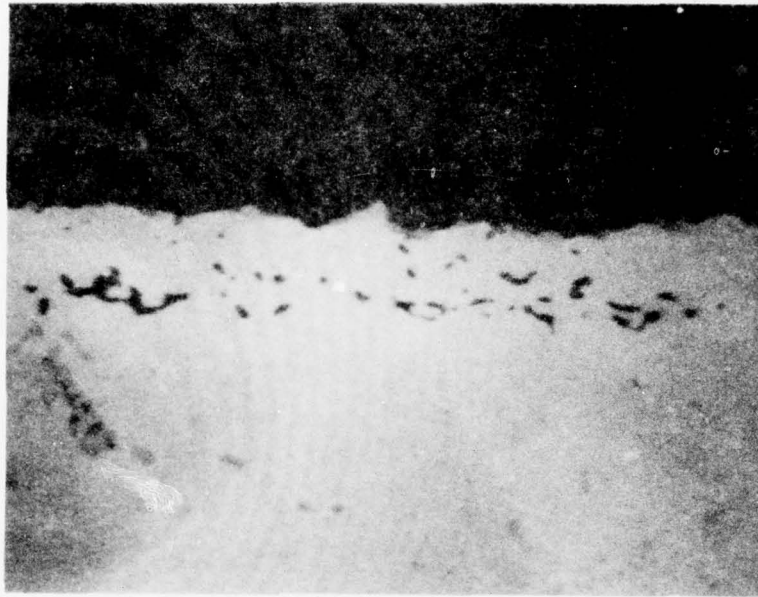


Figure 33

Magn: 500X

NiCrY chemically activated coating on Mar-M247 bagged with GE 664 and pressed at 54 ksi/15 sec. after 1093 C/1 hour in a vacuum.

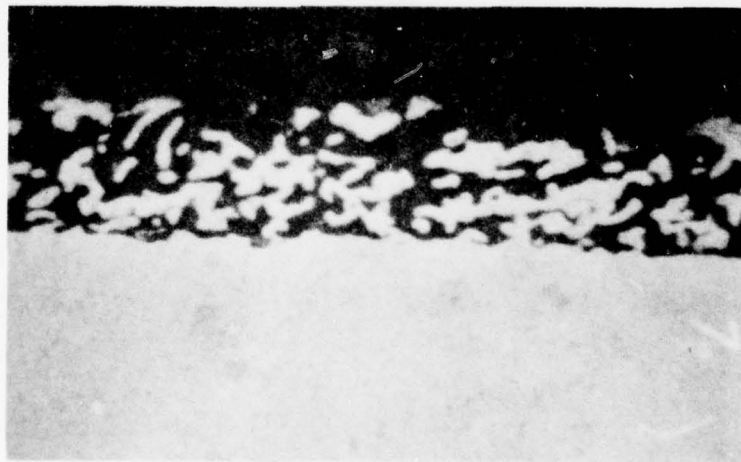


Figure 34

Magn: 500X

NiCrY chemically activated coating on Mar-M247DS bagged with Turco TFM 5580-6 pressed at 54 ksi after 1093 C/1 hour in a vacuum.

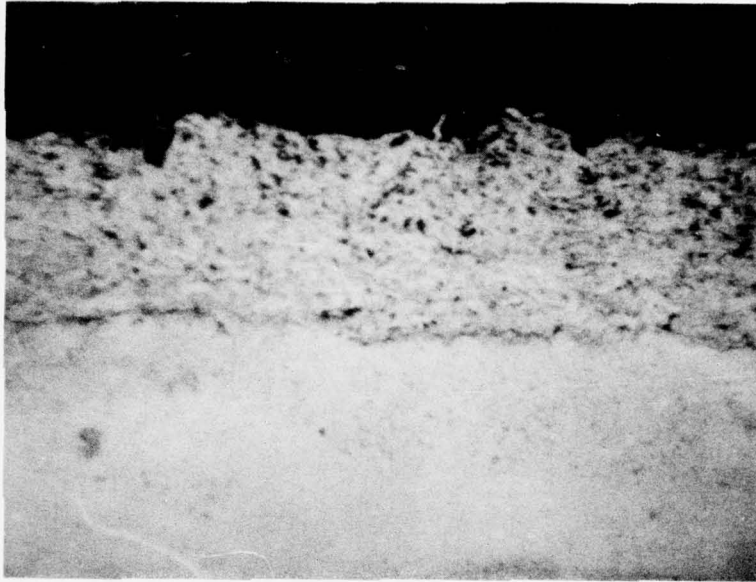


Figure 35

Magn: 500X

Chemically activated NiCrY coating on Mar-M006DS bagged with latex rubber and pressed at 54 ksi.

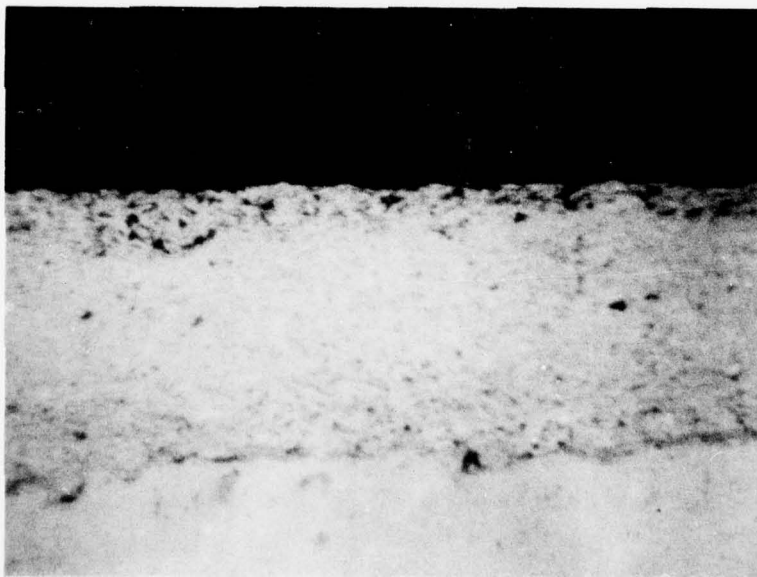


Figure 36

Magn: 500X

Chemically activated NiCrY coating on Mar-M006DS wrapped with aluminum foil bagged with latex rubber and pressed at 54 ksi.

Portions of the pressed specimens were vacuum sintered at 1093 C for 1 hr. Both the specimens bagged in latex rubber and the specimens bagged in aluminum foil showed dense coatings. See Figures 37 and 38.

Isostatic Press Sinter - Aluminize

Isostatic pressed NiCrY specimens from the previous experiments were aluminized in modified packs to see if the pressed coating could be aluminized and sintered and converted to a NiCrAlY composition. Modified pack 5 analysis used in the aluminizing runs are shown below Table 3; 25% NH₄Cl was added to the pack composition but is not shown in the AA analysis.

Table 3

Modified Pack 5 Composition (W/o)

	<u>Co</u>	<u>Cr</u>	<u>Al</u>	<u>Fe</u>	<u>Al₂O₃</u>
Start	15.20	4.85	3.70	.3	Bal
Conditioned	15.10	4.70	3.20	.4	Bal
After Coating	15.10	4.55	3.00	.4	Bal
After Replenishing	18.3	7.00	5.50	.6	Bal
After Coating	18.6	7.00	5.40	.6	Bal

The specimen from isostatic press No. 1, coated with chemically activated NiCrY on IN 713C alloy, was diffused in pack No. 5 in a titanium gettered argon atmosphere for 2 ½ hours at 1130 C.

Microexamination of the coated specimen revealed a dense coating 2 to 3 mils thick. Microprobe analysis of the coating showed an outer layer of NiAl coating with an inner chromium rich layer which is apparently aluminized NiCrY material. A photomicrograph of the coating structure is shown in Figure 39.

Isostatic Press plus Aluminize

After the pack 5 diffusion, the pack was analyzed and replenished. Another experiment was then conducted using this replenished pack. The purpose of the experiment was to achieve a NiCrAlY coating by directly aluminizing the NiCrY coated specimens after isostatic pressing and zein removal. The vacuum sintering cycle after pressing was eliminated.

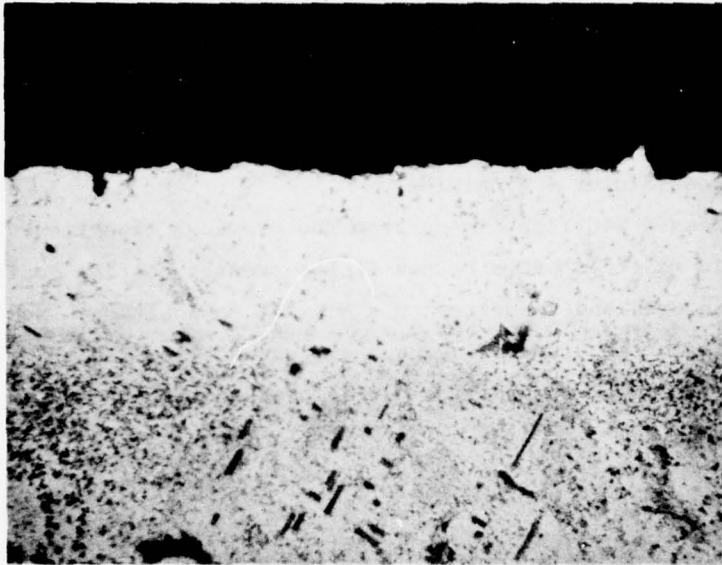


Figure 37

Magn: 500X

Chemically activated NiCrY coating on Mar-M006 bagged with latex rubber, pressed at 54 ksi and sintered at 1093 C/1 hr in a vacuum. Etched in aqueous solution of 14% sodium hydroxide and 14% potassium permanganate.

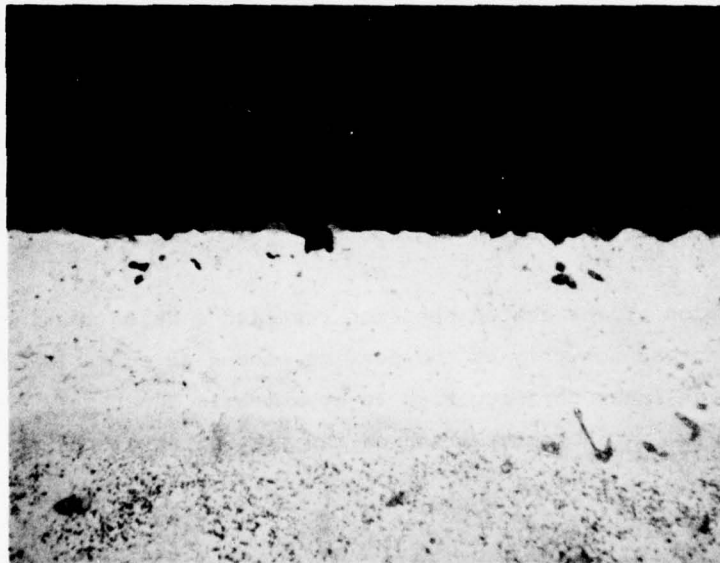


Figure 38

Magn: 500X

Chemically activated NiCrY coating on Mar-M006DS wrapped with aluminum foil, bagged with latex rubber, pressed at 54 ksi and sintered at 1093 C/1 hour. Etched with an aqueous solution of 14% sodium hydroxide and 14% potassium permanganate.

Aluminide
coating

Aluminized
NiCrY Region

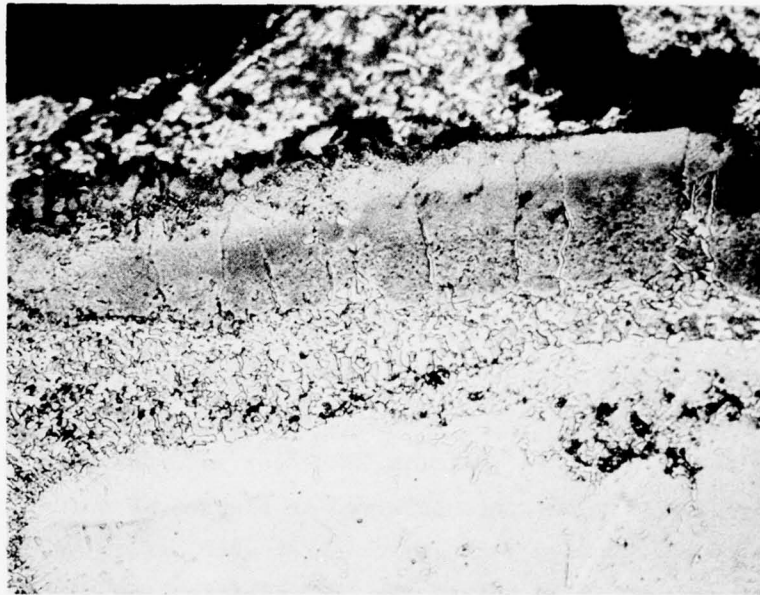


Figure 39

Magn: 500X

Chemically activated NiCrY coating on IN 713C bagged with GE 664
pressed at 54 ksi sintered at 1093 C/1 hour and aluminized in Pack 5.

- o Mar-M006DS coated NiCrY with and without an aluminum wrap under a latex bag prior to pressing had a relatively dense coating of approximately 2 mils. Slight porosity existed in the coating. Figures 40 and 41 show the chromium rich areas dispersed in a nickel aluminide matrix and the thin interdiffusion layer at the interface.

Microprobe

Microprobe analysis scans were performed on the pressed and aluminized specimens coated in the replenished pack 5. Relative concentrations of the NiCrAlY coating elements was determined, except for the yttrium, which at this low concentration (0.17%) is not within the detectability limits of the instrumentation. A microprobe scan of the NiCrAlY coating structure is shown in Figure 42.

The coatings have a narrow outer region of nickel aluminide. The major portion of the coatings consist of chromium rich particles in a nickel-chromium-aluminum matrix. In addition, SEM spot mode analysis of the white particles and gray matrices observed in Figures 40 and 41 showed the particles to be chromium rich and the gray matrices to be nickel-chromium-aluminum. As noted previously, the distribution of the yttrium was not detectable in the microprobe scans. The coating aluminum content ranges from 15 to 22 percent.

The coatings are primarily an overlay type with limited interdiffusion with the base alloy as indicated in Figure 43 by almost no Mo and W in the coating structure. Base alloy carbides do not extend over 0.5 mil into the coating. Microprobe scans of a non-NiCrY coated specimen area showed a typical aluminum diffusion coating with base elements in the coating.

Isostatic pressing followed by aluminizing of the AEP deposited MCrY pre-alloyed powders appears to be the most promising procedure for producing the desired MCrAlY overlay coatings.

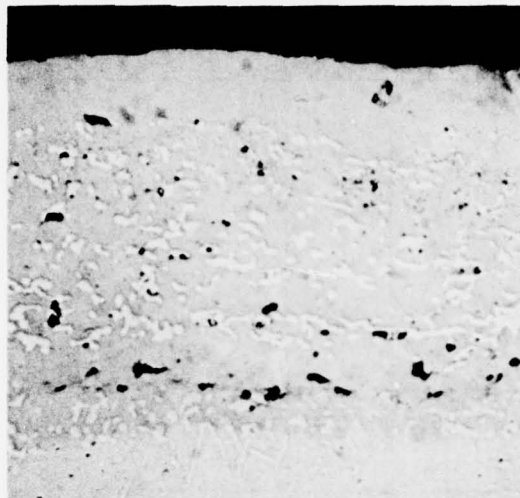


Figure 40

Magn: 1000X

Pre-alloyed NiCrY chemically activated coating on Mar-M006DS bagged with latex rubber over aluminum foil, pressed at 54 ksi and sintered in replenished Pack 5.

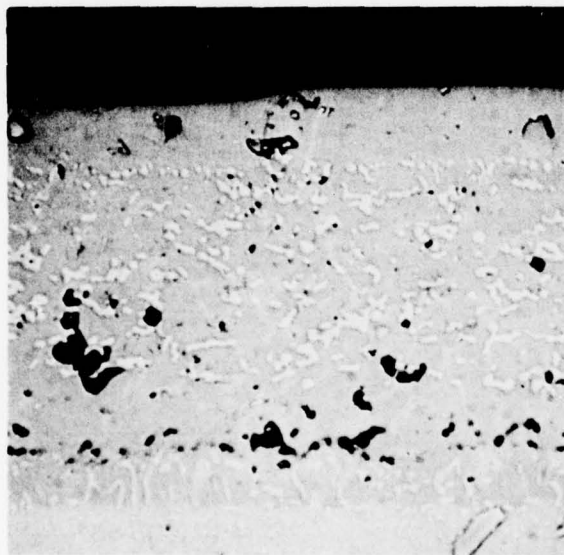


Figure 41

Magn: 1000X

Pre-alloyed NiCrY chemically activated coating on Mar-M006DS bagged with latex rubber pressed at 54 ksi and aluminized in replenished Pack 5.

Electron Microprobe Scan of Overlay Coating Showing Distribution of Coating Elements

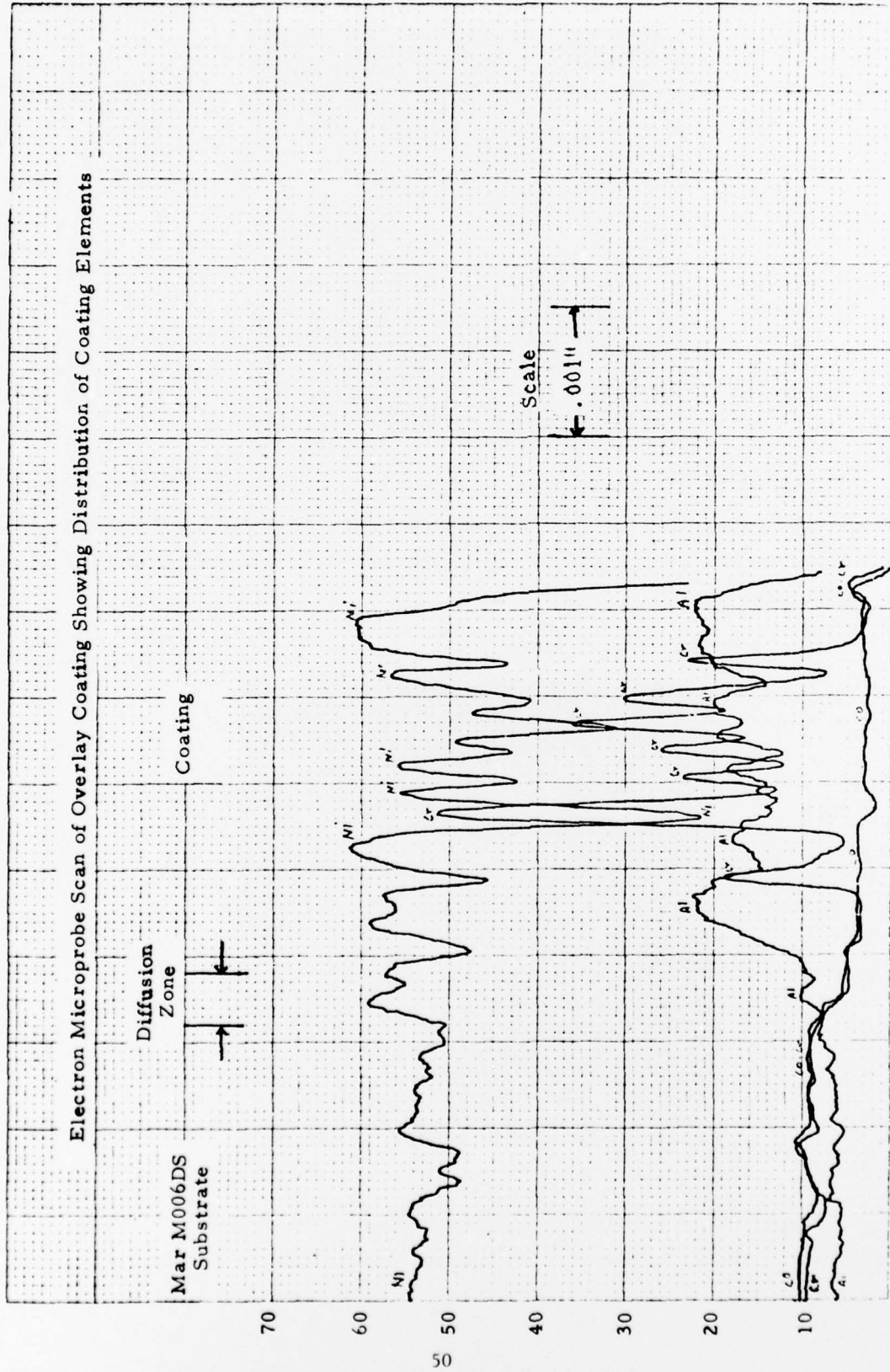


Figure 42

Electron Microprobe Scan Illustrating the Minimum Diffusion in an NiCrAlY Overlay Type Coating as Shown by the Molybdenum and Tungsten Lines

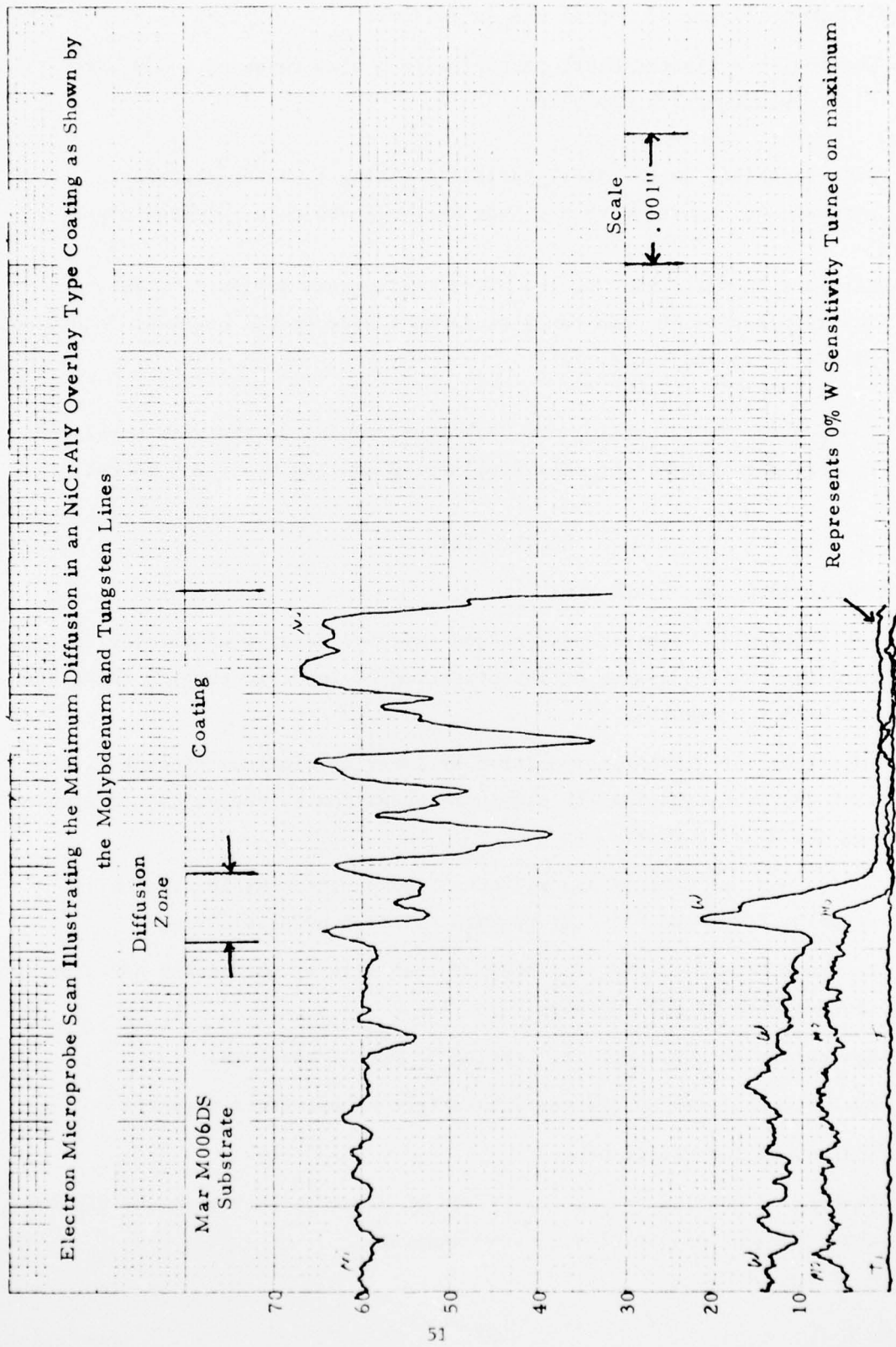


Figure 43

Processing employed to develop the pressed and aluminized NiCrAlY coatings shown in Figure 40 and 41 was as follows:

- o Chemically activated NiCrY particles in a size range of 1-20 μ were electrophoretically deposited.
- o Zein deposited on the NiCrY particles during the electrophoretic process was removed with a 2 hour cycle at 760 C in gettered argon.
- o Particle densification of the NiCrY coating was achieved by isostatically pressing at room temperature in a hydrostatic press at 54,000 psi with a 15 second cycle.
- o The pressed NiCrY coating was densified, bonded to the base metal and converted to NiCrAlY by pack aluminizing at 1130 C for 2 $\frac{1}{2}$ hours.

SECTION III

RECOMMENDATIONS FOR FUTURE WORK

- o Achievement of higher densities and improved coating uniformity during cold isostatic pressing by investigation of improved bagging techniques and higher pressures.
- o Adjustment of NiCrAlY composition by lowering aluminum content of the pack and investigating AEP aluminum deposition before and after pressing the NiCrY powder.
- o Achievement of CoCrAlY and Ni/CoCrAlY composition by isostatically pressing CoCrY and Ni/CoCrY powders followed by aluminizing.
- o Increase MCrAlY coating thickness to 3-6 mils by increasing AEP deposition weights of MCrY powders.
- o Hot corrosion and ductility testing of MCrAlY coatings.
- o Evaluation of AEP MCrAlY coatings effect on substrate properties.
- o Scaleup of AEP MCrAlY process.
- o Coating of complex components by use of press cycle bagging by dipping the component or use of segmented bagging.

APPENDIX

Coulter Counter scans of the particle size distribution of the prealloyed particles used in this program are shown in pages 54 to 58. Equipment used in the program is shown on pages 59 through 62. A temperature conversion table of the commonly used temperatures is given on page 63.

LIST OF ILLUSTRATIONS

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SCANS I

COULTER COUNTER Model T & TA PARTICLE SIZE ANALYSIS		COULTER ELECTRONICS INC. 575 N. 11th FALCON, WIS. 53120	
OPERATOR DDAD	SAMPLE Cerac 14Cr-0.2Y, Bal. Co Lot 4645-D 0.82	MODEL F. N. Lea	15-200-A 1 PERCENT
EQUIPMENT DDAD	SERIAL	PART NO. 51A	SAMPLE SETTINGS 51A
SAMPLE SIZE	FOR MODEL T $A_1 = \frac{(d_1)^2}{4}$	FOR MODEL TA $A_2 = \frac{(d_2)^2}{4}$	SAMPLE WEIGHT $W = \frac{A_1}{A_2} \times W_2$

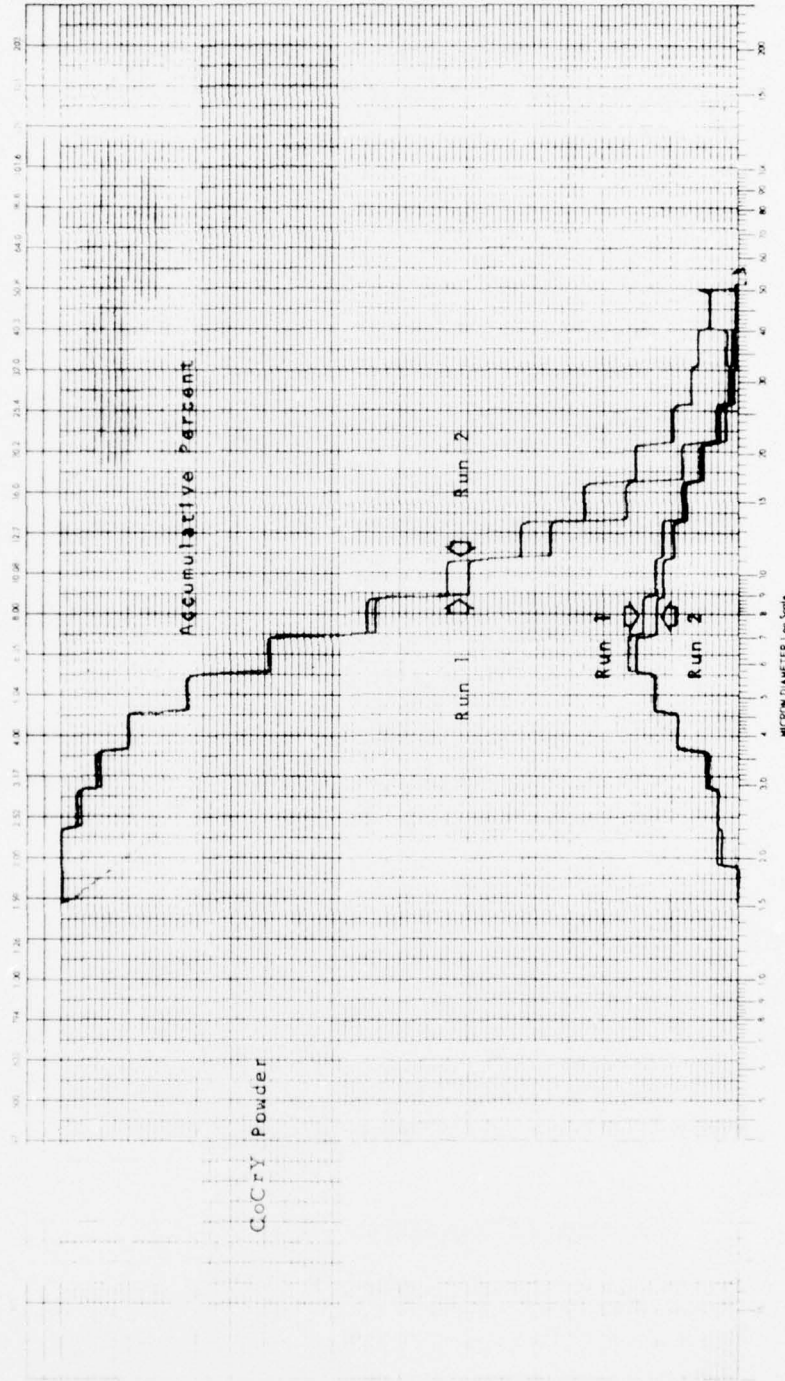


Figure 1 MICRON DIAMETER LOG SCALE

451110

SCANS 11

COULTER COUNTER® Model T & TA		PARTICLE SIZE ANALYSIS	
ORGANIZATION OPERATOR EQUIPMENT	DDAD F. K. Lea	COULTER ELECTRONICS INC. 900 N 95 ST. MESA, FLA. 32950	15 - 200 - X PERCENT
SAMPLE Cerac Lot 4646-B (18Cr 15Al 0.5Y balance Co) (0.93μ)		FOR MODEL T $A_2 = \left(\frac{d_2}{d_1}\right)^3$ when $W_2 = W_1$ $A_1 = \left(\frac{d_1}{d_2}\right)^3$ when $W_1 = W_2$	
ELECTROLYTE DISPERSANT		FOR MODEL TA $A_2 = \left(\frac{d_2}{d_1}\right)^3$ when $W_2 = W_1$ $A_1 = \left(\frac{d_1}{d_2}\right)^3$ when $W_1 = W_2$	
APERTURE SIZE SERIAL		DIA. W ± LA A DIA. W ± LA A	

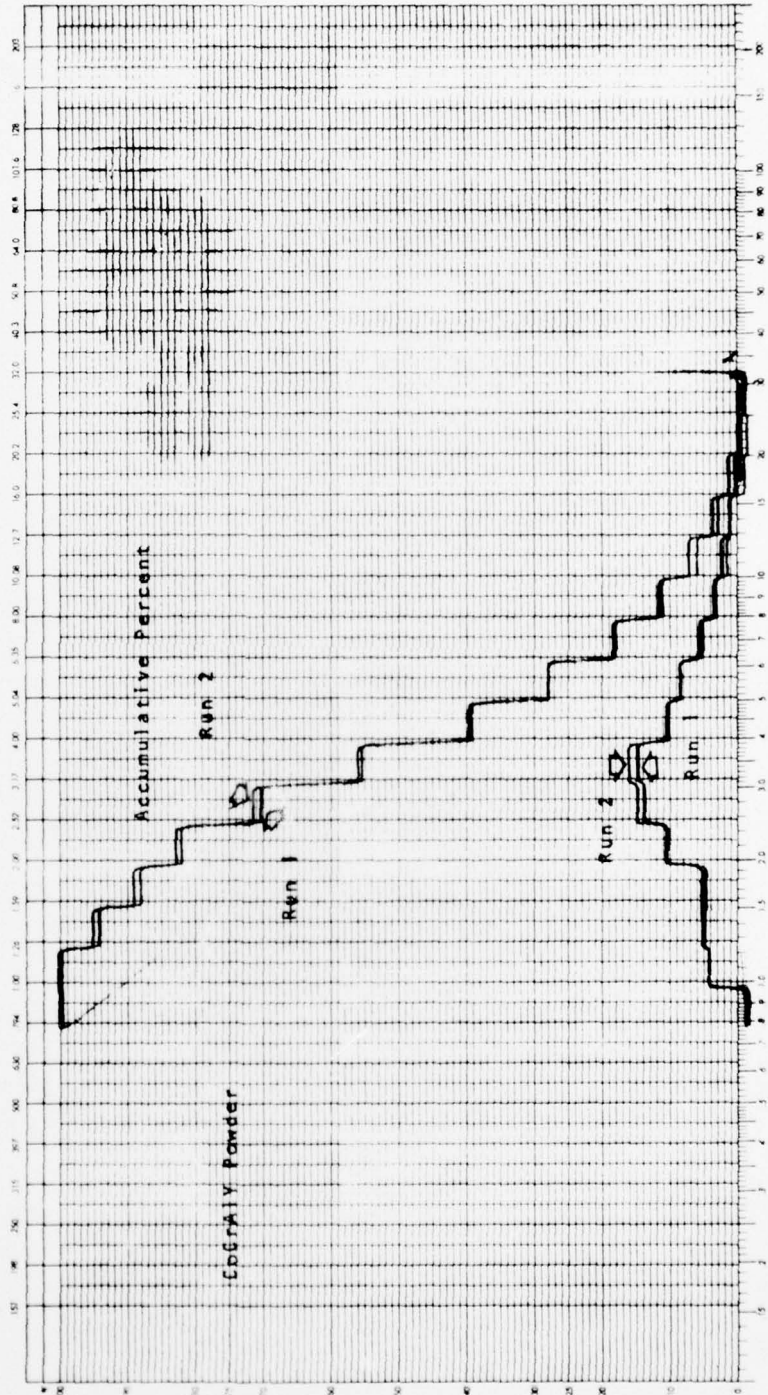


Figure 2 MICRON DIAMETER LOG SCALE

SCANS III

COULTER COUNTER Model T & TA		PARTICLE SIZE ANALYSIS		COULTER ELECTRONICS INC. 195 W 25 ST HIALEAH, FLA. 33010	
ORGANIZATION OPERATOR EQUIPMENT	DDAD F. K. Lea	AMPL. SETTINGS FOR MODEL TA $A_2 = \left(\frac{d_1}{d_2}\right)^3$ when $W_2 = W_1$ $A_1 = \left(\frac{d_2}{d_1}\right)^3$ when $W_2 = W_1$	PART DIA W 2 1A A DIA W 1 1A A		
SAMPLE MICRALY Cerac Lot #4375C-3	ELECTROLYTE DISPERSANT	AREA SIZE SERIAL			

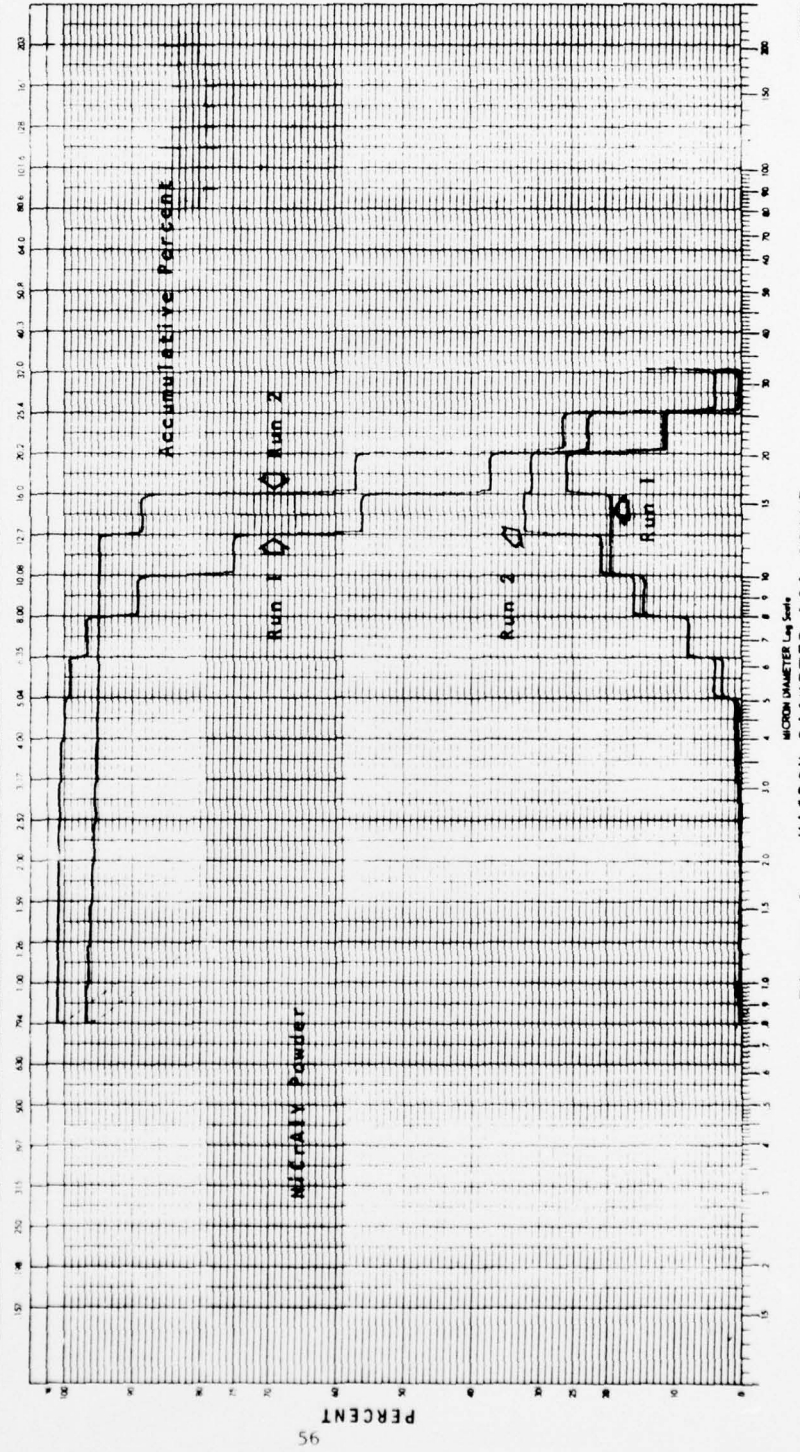


Figure 3 MICRON DIAMETER LOG SCALE

SCANS IV

COULTER COUNTER Model T & TA		PARTICLE SIZE ANALYSIS	
ORGANIZATION DDAD		15, 200- 2 PERCENT	
OPERATOR F. K. Lea		COULTER ELECTRONICS INC. 590 W. 31st. HIALEAH, FLA. 33010	
EQUIPMENT Gerac Ni-Cr (16%) Y (0.2%) Lot #4646-C-RR (0.50μ)		SAMPLE SETTINGS $\frac{A_1}{A} = \left(\frac{d_1}{d}\right)^3$ when $W_1 = W$ $\frac{A_2}{A} = \left(\frac{d_2}{d}\right)^3$ when $W_2 = W$	
SAMPLE		FOR MODEL TA PART DIA # DIA A DIA W DIA A	
APERTURE SIZE SERIAL		FOR MODEL T PART DIA # DIA A DIA W DIA A	

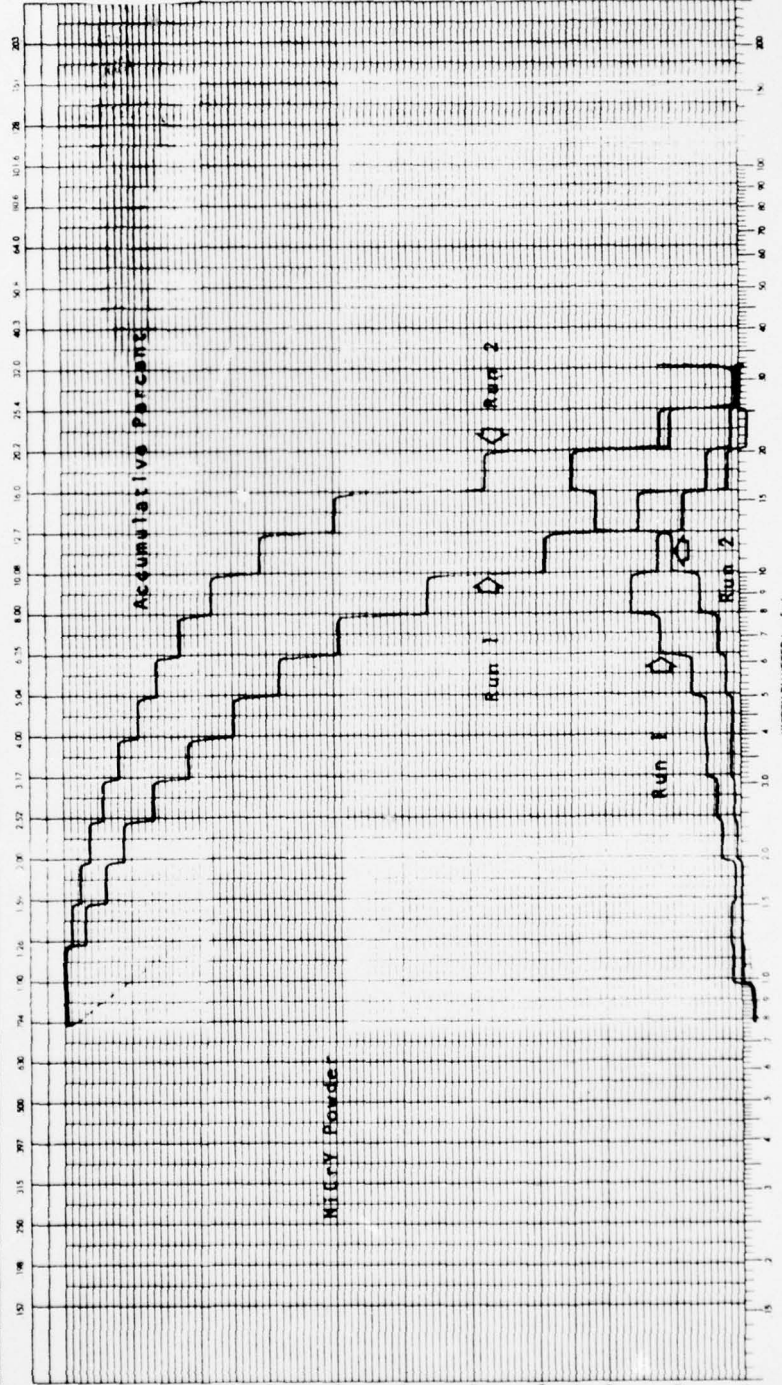


Figure 4 MICRON DIAMETER LOG SCALE

SCANS V

COULTER COUNTER Model T & TA		PARTICLE SIZE ANALYSIS		COULTER ELECTRONICS INC. 990 W 20 ST MIAMI FLA 33136			
ORGANIZATION OPERATOR EQUIPMENT		15-200% X PERCENT		SAMPLE #			
DDAD F. K. Lea		$1 + \frac{1}{2} \frac{V}{V_0}$ FOR MODEL T		$A_2 = \left(\frac{d_1}{d_2} \right)^2 \frac{W_1}{W_2}$ FOR MODEL TA			
SAMPLE ELECTROLYTE		AFTER SIZE SERIAL		START DIA. A B C D E F G H I J K L M N O P Q R S T U V W X Y Z			
SUPERNATANT		1.00 1.25 1.50 1.75 2.00 2.50 3.00 3.50 4.00 4.50 5.00 6.00 7.00 8.00 10.00 12.5 15.0 20.0 25.0 30.0 35.0 40.0 50.0 60.0 70.0 80.0 100.0 125 150 200		1.00 1.25 1.50 1.75 2.00 2.50 3.00 3.50 4.00 4.50 5.00 6.00 7.00 8.00 10.00 12.5 15.0 20.0 25.0 30.0 35.0 40.0 50.0 60.0 70.0 80.0 100.0 125 150 200		1.00 1.25 1.50 1.75 2.00 2.50 3.00 3.50 4.00 4.50 5.00 6.00 7.00 8.00 10.00 12.5 15.0 20.0 25.0 30.0 35.0 40.0 50.0 60.0 70.0 80.0 100.0 125 150 200	

Cerac Ni, Cr (16%), Y(0.2%)
 Chemically activated by Universal Cyclops

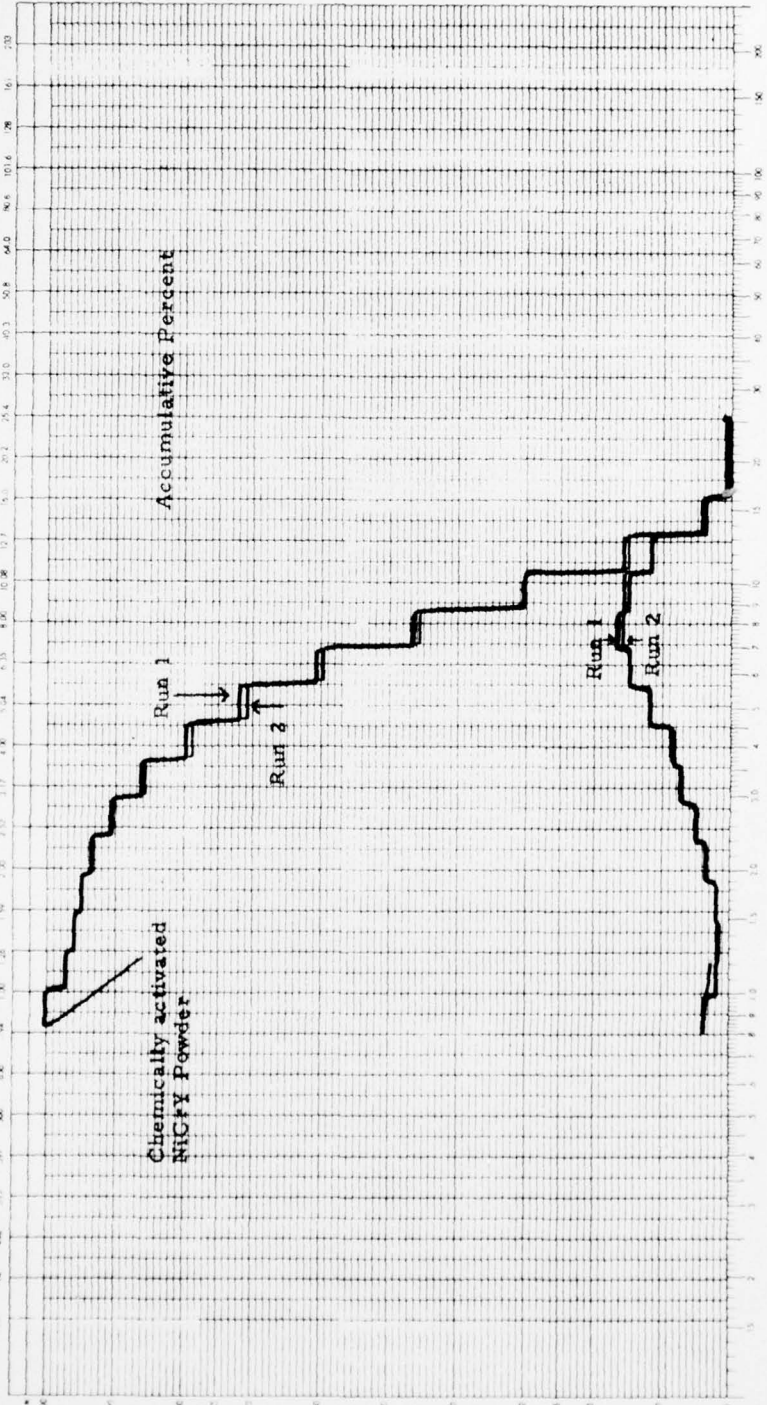


Figure 5 Micron Diameter Log Scale



Figure 6 Isostatic Press

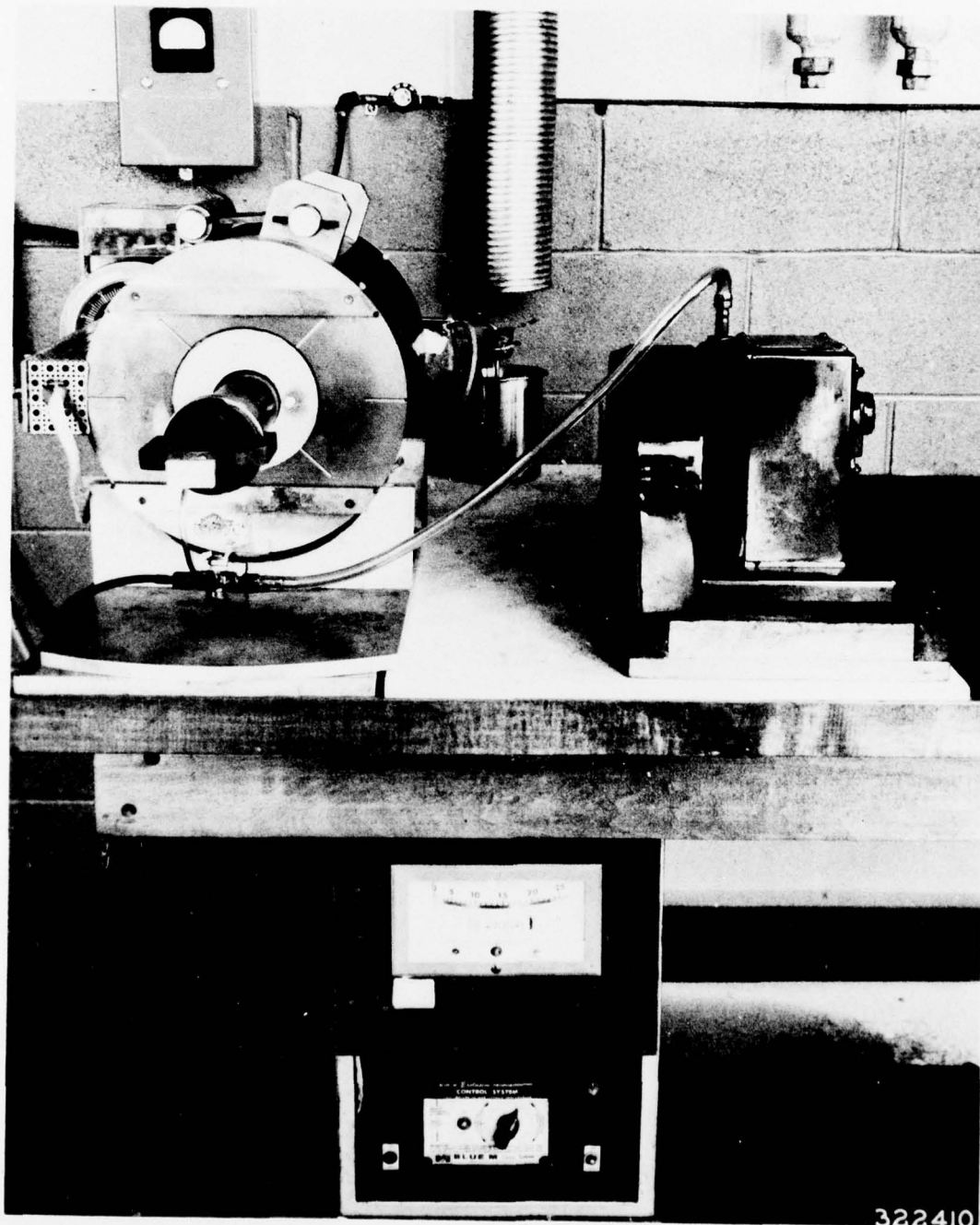


Figure 7 Tube Furnace



Figure 8 Vacuum Furnace

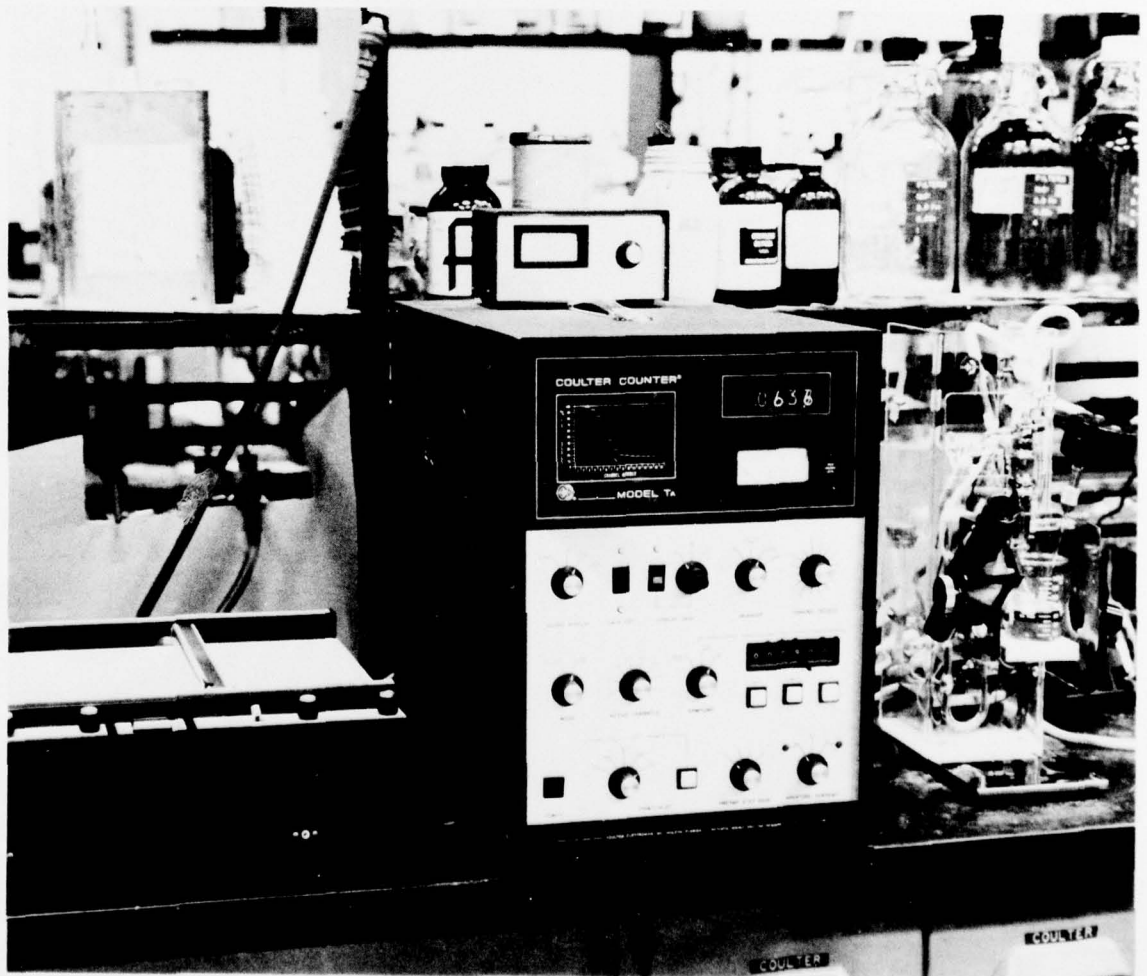


Figure 9 Coulter Counter

Table 1

UNIT CONVERSION

Temperatures in this report are presented in SI units. Common conversions to English units are presented below:

<u>METRIC</u>	<u>ENGLISH</u>
371 C	700 F
704 C	1300 F
760 C	1400 F
1021 C	1870 F
1093 C	2000 F
1130 C	2066 F
1149 C	2100 F
1219 C	2225 F