Refractomet Division

UNIVERSAL-CYCLOPS STEEL CORPORATION

Technical Report

CONTRACT NOw 60-0041-c

FINAL REPORT
VOLUME I OF SEVEN VOLUMES

Bridgeville, Pennsylvania

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TABLE OF CONTENTS
Final Report

Page

VOLUME I

TABLE OF CONTENTS (cont'd)

TABLE OF CONTENTS (cont'd)

LIST OF TABLES

LIST OF FIGURES

LIST OF FIGURES (cont'd)

LIST OF FIGURES (cont'd)

ABSTRACT

This final report summarizes all of the work accomplished under Contract NOw 60-0041-c. Portions of the report are devoted to operator training, development of basic facility operations, translation of operational knowledge to basic metallurgical processes, and the establishment of initial standards for operation. Conclusions regarding the results obtained and recommendations regarding future actions are clearly set forth and discussed.

I. Introduction

Contract NOw 60-0041-c was commenced on March 16, 1960 by and between the Department of Navy, Bureau of Naval Weapons and Universal-Cyclops Steel Corporation. The basic contract was then amended by Supplement Agreement No. 1, which extended the contract completion date to 30 June 1961. Supplement Agreement No. 2 extended the contract date to "during the month of January 1962" and increased the total estimated cost from $175,000 to $315,000. In addition to these supplements, a letter was received from the Bureau of Naval Weapons dated 1 February 1962, which extended the contract to 30 June 1962. This report is being issued as the final report under this contract.

The objectives of this contract were for the contractor to use its best efforts as permitted by the operational limits of the pilot line plant to

1) Provide for operator training.
2) Develop basic facility operation.
3) Translate operational knowledge derived from Item (2) to basic metallurgical processes.
4) Establish initial product and process standards.

The objectives have been met to the extent permitted by the pilot line plant. However, there is much additional information to be gained from this pilot line plant and recommendations to this effect will be set forth.

An outline of the process development program to be undertaken is shown as follows.

A. Training of Personnel

  1. Definition of Basic Characteristics and Design Aspects of the Facility

    a. The facilities contract (NOa 55-006-c) will provide information pertaining to the purification system as set forth below.

      (1) Complete construction drawings and flow sheets defining operation of the InFab purification system.

      (2) Operating data derived during initial start-up as performed by Air Products' personnel.

        (3) Operating procedures developed through experience gained during start-up and via technical literature provided by Air Products, Inc.

    b. Extend basic information to assist in the training of operating and supervisory personnel in the theory of the room design and operation.

        (1) Basic room design.

        (2) Pressure balance system.

        (3) Equipment and material locks.

        (4) Man door and emergency doors.

        (5) Lighting and other major electrical circuits.

        (6) Room air-conditioning system.

        (7) Relation of room and purification system as complete circuit.

        (8) Areas that require close attention in regard to leakage, rupture, or other malfunctioning.

    c. Develop proper use of man locks to enable minimum contamination and expense of exit and entry into room when under atmosphere.

2. Emergency Operational Procedures

    a. Develop emergency repair methods in the event of damage to windows or other portions of the system that could result in expensive loss of room atmosphere or equipment.

    b. Set up emergency condition in the event of power failure primarily as related to the purification system.

3. Safety Features as Related to Personnel and Equipment

    a. Train personnel in first aid as may be required for man stricken in room.

b. Provide safety equipment required for in-room rescue work and revival of personnel suffering from anoxia. All people related to the operation of the room should be familiar with the use of this equipment.

c. Set-up routine daily safety checks of suits and breathing apparatus.

d. Safety check all operating procedures to insure safety of man within the room enclosure.

4. Complete Communications Procedure

    a. Establish procedure to enable complete communication between men in the room and the outside operators.

    b. Establish system of hand signals in the event of failure of communication system.

5. Definition of Basic Mill-Type Equipment Operation

    a. Have operators study theory of basic mill-type equipment operation from equipment manuals and verbal explanation.

    b. Observation of initial tests as conducted by contractor and representatives of equipment manufacturers.

    c. Plan training of basic operations that must be carried out by personnel within the enclosure.

    d. Set up preliminary operating instructions prior to use of the equipment.

6. Cold Operation of all Equipment

    a. Use of logs and planks.

B. Develop Basic Facility Operation (Debugging)

1. Establish the Functional Operation of Specific Equipments

    a. Development of techniques related to the heating, rolling, shearing, and other material fabricating processes that are directly related to the 14" x 16" experimental rolling mill and its associated equipment.

b. Development of techniques related to forging utilizing the Chambersburg No. 15 impactor and associated furnaces, hoists, and manipulator.

c. Development of procedures for handling of materials within the InFab enclosure.

  (1) Provide handling equipment to enable the optimum use of the overhead crane in the handling of materials and equipment.

    (a) Provide and develop required equipment to provide flexibility of crane operations and loading.

  (2) Develop requirements such as dollies, manipulating hooks, tongs, etc. that will permit flexible operations and , yet, provide adequate protection to operators.

d. Establish operational practice for hydrostatic compaction equipment.

  (1) Develop satisfactory process for the compaction of metallic powders and sponge.

  (2) Investigate pressure density variable.

e. Establish operational practice for hydrogen sintering furnace.

  (1) Establish proper sintering cycles for various types of compacted materials.

  (2) Establish and insure safe operating procedure for utilization of this equipment.

2. Develop Preliminary Values and If They Exist Limitations of All Equipments

a. Rolling Mill and Associated Equipment

  (1) Gain technology on heating of material to minimize any contamination by environmental materials.

  (2) Develop rolling procedures and techniques.

    (a) Develop roll pass designs.

    - (b) Develop handling equipment and methods to minimize human effort required within the room.
  - (3) Determination of physical limitations of equipment.
    - (a) Since application is unique, it must be determined to what limits of temperature equipment can be used.
    - (b) Determine most applicable starting and finishing sizes to permit maximum utilization of equipment.

b. Impactor and Associated Equipment

  - (1) Develop most desirable shape of basic ingot and sprue to enable the best handling and forging conditions.
    - (a) Study castable shapes for sprue requirements.
    - (b) Study sprue weldments.
  - (2) Provide simple handling equipment for loading and unloading of manipulator.
  - (3) Determine practical limitations of material sizes to be forged, and possibilities of avoiding procedures presently encountered in the production of refractory metals that are not desirable as to economy or yield.

c. Hydrostatic Compaction Equipment

  - (1) Develop equipment for handling of compaction assemblies.
  - (2) Develop material specification best suited for compaction techniques.
  - (3) Establish scope of practical compaction operations.

d. Hydrogen Sintering Furnace

  - (1) Determine operating capacity of the furnace.
  - (2) Conduct study on gas consumption and quality requirements.
  - (3) Determine maximum allowable sintering temperatures.

3. Redesign and Modification, As Required, and Develop Under "1" and "2".

   (A RECYCLING REQUIREMENT UNTIL ALL FEATURES ARE KNOWN)

   a. Incorporate designs required to make equipment perform a given operating function.

      (1) Make required design changes to insure safety of operations that are presently borderline or unsafe.

      (2) Make necessary design changes to reduce human effort within the enclosure to a minimum.

      (3) Make required modification or design changes required to provide a practical operation with sufficient capacity to warrant operation of the facility.

   b. If necessary, initiate new equipment designs to replace or augment existing equipment.

   c. Compile design and operational data on equipment for further process and operating specifications.

C. <u>Translation of Operational Knowledge to Basic Metallurgical Processes</u>

1. Evaluation of Thermal Effects During Forging and Rolling

   a. Material Considerations

      (1) Maximum and minimum die temperatures

      (2) Rate and duration of reheat cycles

   b. Equipment Considerations

      (1) Time-temperature service limitations

      (2) Construction material and product interactions

      (3) Inductive coupling effects

2. Evaluation of Environmental Effects During Forging and Rolling

   a. Material Considerations

      (1) Atmospheric impurities

      (2) Purposeful atmospheric additions

   b. Equipment Considerations

      (1) Atmospheric - electrical interactions

      (2) Atmospheric - lubricant interactions

3. Evaluation of Rate Effects During Forging and Rolling

   a. Material Considerations

      (1) Rates of deformation

      (2) Rates of heating and cooling

   b. Equipment Considerations

      (1) Loading effects

      (2) Thermal transfer effects

4. Evaluation of Degree Consideration During Forging and Rolling

   a. Material Considerations

      (1) Percentage reductions - forging

      (2) Percentage reductions - rolling

      (3) Interaction effects of forging and rolling

   b. Equipment Considerations

      (1) Gage and size limitations induced by separating forces, handling and manipulation.

5. Other Aspects

   a. Material structural requirements

   b. Process contribution to properties

   c. Shape considerations

D. Establish Preliminary Product and Process Specification

1. As a function of a given material type, establish preliminary forging specifications defining:

   a. Starting sizes

   b. Finished sizes

   c. Heating rates and times

   d. Deformation cycle

   e. Shape considerations

   f. Finishes and tolerances

   g. Production rates

2. As a function of a given material type, establish preliminary rolling specifications defining:

   a. Starting sizes

   b. Finished sizes

   c. Heating rates and times

   d. Roll pass design

   e. Finishes and tolerances

   f. Production rates

3. As a function of a given material type and particle size distribution, establish preliminary pressing specifications defining:

a. Particle sizes

b. Bag-filling operations

c. Pre-and post-pressing densities

d. Compaction cycle

e. Dimensional changes and density factors

f. Product sizes and relative strength

g. Tolerances and finishes

h. Production rates

4. As a function of a given material type, establish preliminary sintering specifications defining:

   a. Starting sizes

   b. Finished sizes

   c. Density and related strength

   d. Purity vs. atmosphere

   e. Purity vs. temperature

   f. Dimensional variations caused by sintering

   g. Production rates

The unique thermal and environmental conditions associated with facilities covered by Contract NOa 55-006-c and amendments have established the need for a process development program to prove out the integrated operation of these facilities. In determining the content and estimated cost of a process development proposal, it became apparent that continuous operation is synonymous with economy. To permit continuous operation of the facility, it is absolutely necessary to establish a sound maintenance program providing for preventative, routine, and corrective maintenance.

Routine and preventative maintenance are, for all practical purposes, the areas covered by this proposal. Corrective maintenance which represents malfunction of equipment or poor construction practice falls within the responsibility of the subcontractor or equipment vendor. Subcontract AUTH 10, to prime contract NOa 55-006-c, sets forth subcontractor responsibility in this regard.

Corrective maintenance which constitutes inadequacy of design or specification, therefore not the responsibility of the subcontractor, will require additional design, engineering, and development in order to achieve the objectives as set forth in the facilities contract. When this situation is involved, it is recommended that such work be performed within and under the scope of the Facilities Contract NOa 55-006-c and amendments.

This outline has been completed to the extent possible with the present equipment capabilities. Thus, we can state that objectives have been met. In attaining these objectives, there was of necessity an overlap, and in some cases, no clean cut means of determining when the objectives were attained. However, in Appendix A is a chart with identifying circles indicating the report period when the objective was attained.

II. Argon Purification Plant

The argon purification plant is a complex operation requiring extensive training of operators to learn the intricacies of keeping this plant in operation on a twenty-four hour, seven-day a week basis. An attempt will be made to treat the various pieces of equipment associated with the purification plant individually so an appreciation of the complete system can be realized.

A. Nitrogen Compressors

When the purification system was first put into operation, there was a continuing problem with short valve life due to carbon build-up. This carbon build-up was directly related to the breakdown of the oil used in the force feed lubricators. The oil originally used in these compressors was DTE105. This oil was recommended by Ingersoll-Rand, the builders of the compressor, and Air Products, Inc.

After long consultations with Ingersoll-Rand and Air Products, Inc. personnel, the oil was changed to Solnus 500 a naptha base oil. It was found, after running the compressors using this new oil, that sludge and carbon which had been formed by the previous oil were loosened and the compressors began to clean up. During the break-in period with this new oil, the compressors were operated with a 1500 psi output pressure and, after running for six days, they were torn down and all valves and traps cleaned of sludge and old oil. Subsequent operation for a nine month period caused no deterioration of valve life. Thus, the use of naptha base oils had, for all practical purposes, eliminated the valve problems in the Ingersoll Rand nitrogen compressors.

During July, 1962, Air Products, Inc. sent a notice to all users of Solnus 500 that compressor temperatures must be held below 365 °F, the flash point of the oil. We had been operating at a discharge pressure of 400°F, while operating at pressures around 2500 pounds. When the temperature and pressures are dropped, a loss in cooling efficiency results and, as a consequence, a new oil with a higher flash point was desired.

Representatives of both Ingersoll-Rand and Air Products, Inc. were called in to witness the operation of these compressors. After considerable consultation and actual trial runs by Air Products, Inc. at other locations, the oil was changed to Cellulube 300, which is a high detergent oil with a flash point of 570°F. This oil should not change the operation of the compressors, and it is anticipated that no further difficulties will be encountered.

B. Argon Compressors

The argon compressors are the prime movers in the system and control the pressure and flow of the argon through the purification system and circulation through the InFab enclosure. These compressors are built by Nash Engineering Company as standard units. However, in the InFab operation, the compressors are operated under water to prevent contamination of the argon gas stream. Operation under water results in corrosion of the compressor and chamber components. Thus, periodically, the chamber and pumps are scraped to remove all rust and grease, and then painted with water resistant and rust inhibiting type paints. When this clean-up is effected, the supply lines are flushed to remove any residual sludge that may have accumulated.

During December, 1961, there was increasing concern with the fact that these compressors were becoming noisy and restricting the output pressure to the argon process system. Consultations with Nash Engineering and Air Products, Inc. indicated that the problem of wear may have been due to corrosion resulting from the use of distilled water.

One of the units was dismantled and shipped to Nash Engineering for rebuilding. Nash indicated that a large portion of the wear was caused by misalignment of the pump shaft and motor shaft. The second compressor was rebuilt on site and both units are presently working satisfactorily. However, there is still the problem of sludge build-up and we are continually working on this problem even though it is of a minor nature and can be taken care of by routine cleaning. It is hoped that we can eventually obtain a water softener that will allow the sludge to be self cleaning.

C. Catalytic Reactor

The catalytic reactor is a heated palladium catalyst which combines hydrogen and oxygen to form water vapor. This unit is the means by which oxygen is removed from the argon gas. When the purification system was first operated, difficulties were encountered with this unit because the operating temperature could not be maintained at high gas flows. Installation of a higher BTU heater resolved this problem and it has been operating excellently ever since.

D. Instruments and Instrument Air Lines

1. Beckman Recorders and Analyzers

a. H_2RC-5 Hydrogen in Argon

This recorder is periodically checked and mechanically and electrically zeroed and spanned. It is calibrated for a full span deflection of 1.25 MV. The battery in the standardization circuit is also maintained in top running order by periodical checks and replacement.

The thermo-conductivity analyzer is calibrated with standard gases to assure its accuracy. Potentiometers are adjusted to correct cell voltage and facilitate span calibration.

b. AR-4-Argon Analyzer

This recorder is electrically and mechanically calibrated on a routine basis. Adjustments to potentiometers are made to perform accurate calibrations.

c. O_2R_2 Oxygen Analyzer

This unit requires only minor adjustments to the normal zero and span potentiometers.

d. AR-3 Argon Purity Analyzer

Occasionally the slide wire contactors are not in contact electrically with the slide wire. This calls for slight adjustments. The balancing motor is noisy and rough in operation and has been replaced.

The analyzer portion of this instrument is the most difficult to align of all the instruments. The stability of the instrument is affected greatly by temperature.

Any temperature change on the components on the inner door of the analyzer changes the reading as much as 100%. If the inner door was opened, the temperature change would drive the reading downscale and would not return until the door was closed and the temperature reached its point of control. This is inherent to this low sensitivity instrument.

Zero millivolts from the thermoconductivity cell indicates pure argon. At this low voltage, the sensitivity is at its greatest; therefore, the reading varies as much as 9% off the scale. This instrument should be more stable at the pure argon (100%) end of the scale.

A solution to this problem is being looked into and that is to revise the action of the analyzer. By replacing the pure argon reference gas with the downscale gas, a zero would be established at the downscale end of the recorders. The span gas would then be pure argon or 100%. This problem will be taken care of in the near future.

e. O_2R-1 Oxygen in PPM

A new analyzer cell has been installed in this unit and is working excellently. The recorder is mechanically and electrically zeroed and spanned to maintain continual calibration.

2. Foxboro Consotrol Recorders

These instruments are serviced by the Foxboro Instrument Company under a scheduled service contract. A Foxboro representative comes in once a year to check and clean the instruments.

Listed below are some of the points covered by the last service call.

a. Checked calibration to the signal pressure range 3-15 pounds.

b. Checked and calibrated zero and span adjustments.

c. Checked pen stops.

d. Checked chart drive mechanism.

e. Adjusted reaction time of the recorders.

f. Changed seal valves on liquid level recorders LRC-1 and LRC-3.

g. Cleaned all instruments.

In general, the instruments were in good condition except for the fact that there was some oil carried over to the instruments by the air lines. The changeover from plant air to the stand-by air compressor is the first step in remedying this condition. If the problem still exists, it may be necessary to switch to a Teflon ring compressor.

3. Instrument Air Lines

All air supply lines are now supplied by the InFab auxiliary compressor rather than house-air. This change-over was made to minimize oil carry-over into the process lines. There are two by-pass lines installed in the instrument air line. One by-pass was installed in the Hankison filter line and the other was installed in the pressure regulator line. These lines permit the removal of filter and regulator for cleaning or replacing without shutting down the plant.

E. Low Level Freon Unit

This unit is used to cool compressed argon to -80°F to its being fed into the cold box for expansion and liquefaction. This unit requires periodic maintenance by refrigeration engineers and Heagy Electric has been retained to perform this service work. It was thought that we had an undue amount of maintenance on this unit but, when checking with other plants of this size and complexity, we were gratified to learn that our problems had been minimal.

Normal maintenance that is performed on this unit includes the following:

1. Clean crankcase and replace oil change.

2. Recalibrate and clean oil separator float valve assembly.

3. Leak test and check entire system.

F. Distilled Water System

This unit has worked satisfactorily except for a build-up of sludge in the lines, condensor, and evaporator. The use of water softeners should resolve this problem and it is anticipated that a resolution will be obtained in the next six months.

G. Miscellaneous Equipment

All other units such as hydrocarbon absorber vacuum pump, argon process lines, storage tanks, chilled water units, etc. are periodically checked to insure efficient operation of the system. Spare parts that are required for all equipment are kept on hand to insure continuous operation of the entire unit. No other problems of importance are presently known except those mentioned above.

H. Start-Up Procedures

1. Defrost

Normally the defrost cycle is used only to clear out frozen contaminants that collect in the process lines of the purification system. However, in all cases, it is deemed advisable to have a complete defrost and purge when the system has been idle for a month or more.

The defrost of process lines is accomplished by flowing hot air through the lines and then venting to the atmosphere until the vent gas is warm to the touch. The air for the defrost is produced by running the nitrogen compressors on air service and drying the gas in the nitrogen adsorbers. The heating is accomplished with chromalox elements in the defrost heaters.

The system is purged with dry nitrogen immediately after defrost to replace the air and to prevent water vapor from entering the lines.

The total time for defrosting the plant is approximately twenty-seven hours for argon process lines and ten hours for the nitrogen process lines.

2. Burn-Out of Oxygen

The oxygen content of the room is decreased by burning hydrogen in the room. Water vapor produced by this burning is removed by the process driers and the gas returned to the room so as to prevent a large build-up of water in the room. Nitrogen is added from the balance tank to make up for the volume of gas lost in combustion.

The circulation cycle, while burning, is accomplished by passing the gas through the compressors, then the driers and back to the room. The recycle line back to the room must always be regulated so as to maintain a pressure of 50 psig at the drier in service.

Combustion is continued in the room until an oxygen level of 10% is reached. The total burning time required to reach this level is approximately forty hours with 38, 000 cubic feet of hydrogen used to effect the reduction. The rate of flow used is 900 scfh, with the final four hours of the process using a rate of 600 scfh.

3. Purge Room with Argon

When an oxygen level of 10% is reached, all operations connected with the room are ceased to allow the room to be dormant for purging. Purging is effected by adding 95% pure argon to the bottom of the room and the top vented so there is a displacement of oxygen and nitrogen by argon. This procedure is continued until the oxygen level in the vent gas reaches 5%, at which time purging is stopped.

4. Recycle to Remove Oxygen

With an oxygen level of 5%, the catalytic reactor is started and removal of oxygen in this manner is continued until the level of oxygen in the room is approximately 2%. It should be noted that while operating the reactor, a recycle stream must be maintained to keep the oxygen content to the reactor below 3%. The reaction is continued until the oxygen content of the room is approximately 1% before the process stream to the liquification and distillation column is started.

5. Start Plant

When the oxygen content of the room reaches 1%, the nitrogen process system is started and the nitrogen refrigeration unit is put into operation to cool the nitrogen. Once the nitrogen and argon process streams are directed to the cold box, the operators must maintain a vigil on pressures and wait until liquids begin to form in the columns.

6. Gas Consumption

During the start-up of the purification system - from August 22 to August 31, 1961 - when purity was reached, the following quantities of gas were used:

Nitrogen	61, 446 cubic feet
Argon	141, 898 cubic feet
Hydrogen	38, 340 cubic feet

I. Shut Down Procedures

Incorporated into the purification system is a "pump to storage" system for recovering argon from the room. This system simply involves pumping liquid argon from the purification plant to the liquid storage tanks. In the first attempt to accomplish this task, various problems were encountered. It was obvious from the outset that when pumping liquid, it would be necessary to maintain proper temperature balance and liquid levels. As pumping is continued, this becomesincreasingly difficult to accomplish until finally argon liquid levels are lost and pumping is necessarily discontinued.

On the first attempt to recover argon during shutdown, only 12, 000 cubic feet was recovered. Subsequent shutdowns resulted in 23, 000 cubic feet, 42, 000 cubic feet, and 62, 000 cubic feet recoveries. It is anticipated that approximately 80, 000 cubic feet can be recovered on the next trial and this will be the leveling point of recovery. Of course, continued practice may allow even better recoveries than have been anticipated to date.

J. Routine Operations and Personnel Training

During the routine operation of the InFab purification system, the operators record the information shown in Figure 1. If any changes in the operating characteristics are noted, corrective action can be quickly initiated. With this comprehensive list of information, the operators can determine trends of operation and, in many cases, corrections are made before any unbalance of the system is attained. It is planned that this information will continue to be collected whenever the purification system is operated.

There are presently six qualified purification operators and a supervisor. They are E. Lelik - Supervisor and G. Wagner, L. Gill, J. Malinky, T. Raineri, W. Mehalic, and S. Heiser. All of them can perform the routine operations of the facility on a continuing basis. Whenever problems of a major nature occur, experts and consultants are called in to help determine the necessary corrective action.

III. In-Room Equipment Operation

The operation of the equipment associated with the InFab enclosure has involved considerable development effort because of the uniqueness of the facility. This is the first room of its size, where attempts have been made to process refractory metals on a pilot plant basis. As a consequence, all of the problems associated with being first have been encountered. An attempt will be made to outline these problems individually with respect to each piece of equipment.

A. Rolling Mill and Related Items

The description of the rolling mill and its operation will include the furnaces, rolls, and all associated items of concern to the rolling operation. Each problem will be handled separately and discussed.

1. Rolling Mill Furnace

The furnaces used for rolling are two graphite susceptor units capable of 4000°F. These units have the temperature capability and have performed quite satisfactorily. However, because of the use of graphite in conjunction with the magnesium oxide insulation, a reaction takes place at elevated temperatures which contaminates the argon atmosphere as well as the atmosphere within the furnaces. Large amounts of carbon monoxide and carbon dioxide are given off by the furnace.

FIGURE I

IN-FAB

UNIVERSAL CYCLOPS STEEL CORPORATION
BRIDGEVILLE PA.

600 SCFM ARGON PURIFICATION & RECOVERY SYSTEM

DAILY LOG SHEET
NORMAL OPERATION SHEET NO.1

Time		12M/1AM	2AM/3AM	4AM/5AM	6AM/7AM	8AM/9AM	10AM/11AM	12N/1PM	2PM/3PM	4PM/5PM	6PM/7PM	8PM/9PM	10PM/11PM	
Nitrogen Compressor	A/B													
Suction Pressure	PI-92/93													
1st Stage Inlet Temp.														
1st Stage Discharge Press.	PI-74/71													
1st Stage Disch. Temp.														
2nd Stage Inlet Temp.	TI-24/21													
2nd Stage Discharge Press.	PI-75/72													
2nd Stage Discharge Temp.														
3rd Stage Inlet Temp.														
3rd Stage Discharge Press.	PI-76/73													
3rd Stage Discharge Temp.														
Aftercooler Temp.														
Crankcase Oil Added														
Lubricator Check, Oil Added														
Nitrogen Oil Adsorber														
NOA in Service														
Hrs. in Service														
Reactivation Temp. In	TI-19													
Reactivation Temp. Out	TI-18													
Nitrogen Low Level Freon Unit														
Suction Press.														
Intermediate Press.														
Discharge Pressure														
Oil Pressure														
Oil Level Check														
Nitrogen to Freon Unit	TI-1-2													
Nitrogen out Freon Unit	TI-1-3													
	TI-1-25													
Chilled Water Freon Unit	A/B													
Suction Pressure														
Discharge Pressure														
Oil Pressure														
Oil Level Check														
Argon Drier Precooler Temp.	TI-9													
Argon Driers														
Driers in Service														
Hrs. in Service														
Driers on Reactivation														
Hrs. on Reactivation														
Reactivation Temp. In														
Reactivation Temp. Out														
Drier Outlet	T-1-15													
Cooling Water Still Check														
Remarks														

FIGURE I (cont'd)

IN-FAB

UNIVERSAL CYCLOPS STEEL CORPORATION
BRIDGEVILLE PA.

600 SCFM ARGON PURIFICATION & RECOVERY SYSTEM

DAILY LOG SHEET
NORMAL OPERATION SHEET NO2

Time		12M / 1AM	2AM / 3AM	4AM / 5AM	6AM / 7AM	8AM / 9AM	10AM / 11AM	12N / 1PM	2PM / 3PM	4PM / 5PM	6PM / 7PM	8PM / 9PM	10PM / 11PM
Hours Since Defrost													
Flows													
Argon to In Fab Room	PI-1												
Argon Feed	FRC-												
Recycle Argon	FRC-												
Waste Gas	PI-4												
Nitrogen Non-Condensible	PI-												
Hydrogen Flow	PI-8, 9 or 10												
Purities													
Hydrogen After Reactor	H_2XC-5												
Oxygen, Argon After Reactor	O_2XR-1												
Oxygen, Argon to In Fab Room	O_2XR-1												
Oxygen, Argon out In Fab Room	O_2XR-2												
A. Argon to In Fab Room	AR-3												
Argon in Waste Gas	AR-4												
A. Argon out In Fab Room	AR-3												
.													
Liquid Levels													
Reboiler Argon	LRC-1												
Reboiler Nitrogen	LRC-2												
Product Argon Column	LRC-3												
Aux. Condenser, Nitrogen	LRC-4												
Argon Condenser, Argon	LI-5												
Temperatures													
Nitrogen													
H.E. Tube Inlet	TI-1-1												
H.E. Shell Outlet													
Freon Unit Inlet	TI-1-2												
Freon Unit Outlet	TI-1-3												
H.E. Tube Outlet	TI-1-4												
H.E. Shell Outlet	TI-1-9												
H.E. 5.11 Nitrogen Outlet	TI-1-10												
	TI-1-19												
	TI-1-25												
Argon													
Heater Inlet	TI-4												
Reactor Inlet	TI-5												
Reactor Outlet	TI-6												
37.4 Condensate Trap Inlet	TI-8												
Drier Inlet	TI-9												
H.E. Feed Inlet	TI-1-5												
H.E. Recycle Outlet	TI-1-8												
H.E. Product Outlet	TI-1-11												
H.E. Argon Product Inlet	TI-1-12												
H.E. Recycle Inlet	TI-1-7												
Defrost Temperature In	TI-20												
	TI-6												
Pressures													
Nitrogen Head Pressure	PRC-1												
Nitrogen to Reboiler	PI-69												
Nitrogen out Condenser 5.24	PI-67												
In Fab Room	PI-91												
Argon Feed to Plant	PI-42												
Argon H.P. Column	PI-65												
Argon Column 8.10	PI-66												
Argon Δ P across Hx. 5.11	PDI-60												
Argon Product	PI-64												
Bank Pressures													
Hydrogen	PI-3												
Nitrogen	PI-12												
Argon Makeup	PI-16												
Argon Recovery	PI-18												
Liquid Argon													
Liquid Nitrogen													
Argon Recovery													
Argon Pump Inlet	TI-1-21												
Argon out H.E. 5.12	TI-1-22												
Argon to Recovery Storage	PI-50												
Pump Speed RPM													
Pump Blowby Check													
Hrs. on Packing													
Remarks:													

To circumvent the difficulties inside the furnace, a removable stainless steel liner was constructed for insertion into the furnace. This liner is used for heat treating and rolling at temperatures up to 2200°F. At temperatures above 2200°F, molybdenum liners are used to minimize contamination of the work piece.

Because of the persistent problem of contamination from using these units, specifications E100 and E101, for a new rolling furnace and mill tables, were drawn up. Copies of these specifications are shown in Appendix D. The units were purchased from Ipsen Industries and will be installed in the first quarter of 1963. This should eliminate the problem of contamination from the rolling operation.

2. Rolling Mill Rolls

When the rolling mill was originally ordered, it was planned that it would be a bar and rod mill. As time progressed, the requirements for bar and rod became less and sheet was the predominant shape required in refractory metals. The original 14" diameter rolls did not have sufficient strength for sheet rolling and a problem of roll breakage occurred.

To circumvent this problem, cast steel rolls - 16" in diameter - were designed and these worked quite satisfactorily from a strength standpoint. However, when rolling at elevated temperatures, the problem of iron pick-up from the rolls developed. To minimize this problem, double poured rolls with a cast iron case and a cast steel core were designed. These rolls are presently available, but have not been used to date. It is felt that these rolls will minimize the iron pick-up and give much longer roll surface life.

3. Rolling Mill Emergency Stops

As work with the rolling mill progressed, it was found necessary to install emergency stop buttons at various locations. This allows the operating technicians to stop the mill from inside the room. This was done to give the in-room operator control of the mill in the event of an emergency connected with either material in the mill or the operators themselves.

4. Hydraulic Roll Lifters

When the new rolls were installed in the rolling mill, they were much heavier than the original set and, thus adjustments had to be made on the hydraulic roll lifters. This procedure was necessary due to the fact that after installation, the new rolls would not lift properly upon trying to set the mill. The hydraulic lifter pressure was set at 1, 550 pounds. While setting the pressure, it was necessary to add some hydraulic oil to the sump. Also, during the course of repairs the accumulating pressure was checked out at 850 pounds, while the specified pressure is 900 pounds. This pressure was subsequently adjusted and is operating properly.

5. Positive Control of Roll Separator

The magnetic brake on the screwdown has caused considerable difficulty. When attempting to operate the screwdown, a positive stop is not present because the wiring to the magnetic brake fails periodically. We have had electrical personnel working on this unit and believe that the problem has been resolved. If not, we will continue to search for the solution.

It is necessary to resolve this problem because in attempting to produce a given thickness of material, the gage is determined by the stop on the screwdown. If an overrun occurs, time is lost in resetting the mill or the material is rolled to the wrong thickness. Since the problem resolved itself to a short in the wiring and this has been repaired, we do not encounter any more difficulty.

6. Furnace Ram

When the rolling mill was originally conceived and designed for bar rolling, a ram was installed for pushing the material from the furnace to the rolls of the mill. Since the majority of rolling is now resolved to sheet, this ram is no longer required. In addition, the new furnace is of the roller-hearth type and thus the material rolls automatically from the furnace to the mill. For these reasons, the furnace ram device has been dismantled from the room and will be placed in storage.

7. Power Requirements

As work with the rolling mill progressed, it became obvious that only narrow pieces could be rolled at large reductions. The 200 h. p. mill motor is not sufficient to produce large reductions on wide sheet. For the rolling of TZM at normal reductions, only 12" wide material can be produced. Wider sheet can be made if the rolling reductions are lowered. For other types of material with lower strengths, it is possible to roll wider material and the width capability of any material must be individually calculated.

8. Rolling Mill Tables

When the original mill tables were installed, they could not be used because of the cumbersome design. Thus, they were removed from the room and all rolling was done by hand. The most severe difficulty with these original tables was the difficulty of removing material from the furnace to the tables. If bar were to be rolled, the ram could have been used. But since the majority of work was on sheet, this could not be accomplished. With the new roller hearth furnace, the problem of using the mill tables should be resolved. Thus, the tables were revamped and are being re-installed in the same room. With this new set up, material will automatically move from the furnace right into the rolling mill rolls. Thus, a major improvement in operation should be realized.

B. Impacter and Related Items

The impacter was built by Chambersburg and is used for horizontal forging. The capacity of the unit is 15, 000 foot/ pounds and it is driven by high pressure argon. An attempt will be made to describe some of the problems associated with the operation of this unit and their eventual resolution.

1. Oil Leakage

The problem of oil leaking from this unit has been persistent since the original installation. The main cause of the problem appears to be related to the loosening of fittings from the vibrations of the impacter. Periodically, these fittings must be tightened to minimize the oil leakage problem and the resultant contamination. Upon recommendation of the manufacture, all fittings have been loosened and teflon seals incorporated. It is felt that this change will completely eliminate the problem and result in little or no leakage.

2. Control Lines

In the original installation, the control lines were quite long and the response of the impacter to the manipulation of the controls outside the room was very slow. In order to circumvent this problem, the old control lines were dismantled and new, shortened lines were installed. This increased the sensitivity of the unit and resulted in greater control of the forging operation.

3. Removal of Dies

During the first years operation, all forging was accomplished on flat faced dies. When these dies required removal for refacing operations, no problems were encountered. However, when the V-dies were placed in the impacter for forging rounds, one of the dies was knocked off center and was rubbing on the frame of the impacter. This difficulty necessitated removal of the dies and a re-alignment of same. However, during the attempted removal, it was found that the dies were wedged in the impeller. These dies had to be removed by mill machinists and it took several days to accomplish the task. After removing the dies, changes were made in the design of the alignment wedges so that this problem would not reoccur. The "V" dies have since been used and no problems have been encountered with their installation or removal so that this problem has been corrected.

4. Cleaning of Impacter Pit

On several occasions during forging, oil vapor was seen rising from the coil cart pit and contaminating the atmosphere. When this originally occurred, it was felt that it was due to a large quantity of oil present on the pit. Upon heating, this oil would vaporize and contaminate the room. Upon removing the cooling plate in the pit, a large reservoir of oil was found and subsequently removed. No further problems of excessive oil vapor in the room were encountered. However, it was determined that clearing of the pit had to be accomplished periodically to minimize oil accumulation in the pit. This accumulation is due to oil leakage as previously described and as the leakage problems are corrected, less and less of a problem will be encountered with oil accumulation in the impacter pit.

5. Coil Cart

The coil cart is located underneath the impacter and contains the three induction furnaces used for heating billets for forging. The original one-third h. p. motor for moving the coil cart to different positions to use different induction units was found to be too small to do the job effectively. To remedy this situation, a three-quarter h. p. motor was installed and it was found that this traverses the coil cart much more easily with no apparent overloading.

During impact forging, various pieces of material have fallen on top of the ceramic lining of the coil carts. This requires periodic replacement of the top ceramic liner of the induction units. If one plans on periodic maintenance in this area, no operating difficulties are encountered. Additional routine maintenance includes cleaning of bus bars, limit switches, and flow switches to insure proper operation.

6. Argon Leakage Through Packing

There have been problems of argon leakage past the piston on the impacter. When this occurs, there is a decrease in the energy available for forging. The problem can be and is temporarily solved by removing packing shims. However, the only real solution is the periodic replacement of new packing. This has become a routine operation.

7. Cooling Coil Lines in Impacter Heating Pit

To prevent excessive heating of the floor of the impacter heating pit housing the induction coils, there is a tubular cooling coil to remove heat. This plate coil provides only a very small clearance between the coil cart and cooling plate. Occasionally, a piece of refractory unknowingly drops upon the cooling plate and, upon attempting to reposition the coil cart, the connecting water lines to the cooling plate are bent, due to the small clearance present. We have reviewed whether a cooling chamber can be welded to the underside of the pit and find that this area of the pit sets on a concrete foundation. After a review of all other possible techniques, it was determined that the best solution to this problem was to simply clean this area periodically and make sure that all pieces of refractory or metal were removed from the top of the cooling coil so as to prevent any binding of the cooling coil and cart when they move by each other. This type of preventative maintenance has proved quite successful and no additional problems are foreseen.

8. Facing of Dies

When attempting to produce sheet bar in the impacter, considerable difficulty was encountered with mishapen bars. Chambersburg Engineering personnel indicated that this problem could be alleviated by dressing the faces of the dies. By facing the flat dies so as to eliminate any distortion that may have occurred, it was found that a considerable improvement in the quality of sheet bar resulted. Thus, two sets of dies are now kept on hand so that in the future we will be capable of maintaining a good die surface by interchanging dies and facing worn ones.

9. Dowel Pin Breakage

Due to the high levels of lateral force set up when using the V-dies, we have been plagued with shifting of dies and breakage of centering dowels. To minimize the shifting effect, the dies are now tightened between each series of blows. Also, to prevent the possibility of down time due to breakage of dowels, four additional sets are available and this will provide protection in the event of a future occurrence of this difficulty.

10. Manipulator Hoist Speed

During conversations with Chambersburg Engineering, we requested information on how to increase the manipulator hoist speed. We were advised that this could be done by substituting a different cartridge in the Vickers hydraulic pump. This cartridge has been installed and increased the speed from 120 in./min. to 185 in./min.

11. Impacter Mist Collectors

The mist collectors are located on the argon release side of the impacter to minimize the oil mist from entering the room on the release stroke of the impacter. These mist collectors are electrostatic precipitators built by Dollinger Corporation. These precipitators were not equipped with a means of collecting the oil and it simply accumulated on the floor of the room. To remedy this problem, an oil reservoir was constructed and piped to the basement so that there will be no accumulation in the room. Another problem encountered in the operation of the mist collectors was arcing in the precipitator cells making the units inoperative. This problem was exaggerated in the argon atmosphere. Dollinger Corporation recommended the installation of a 30 ohm, 10 watt wire wound valve resistor to be put in series with the 120 volt line in the power pack service on the mist collectors to drop the voltage on the cell to prevent arcing. This was accomplished and improved the operation of these units considerably.

12. Other Items

All other items of the impacter installation have operated successfully when the proper corrective and preventive maintenance is performed. It must be understood, however, that this equipment requires considerable maintenance to function in a routine manner.

C. In-Room Argon Compressor

This compressor is used to maintain proper room pressure, operate the Chambersburg Impacter, supply argon pressure for door seals, provide the compressed argon used to drive the circulating fan in the man-air system used by in-room operators, and, generally, to act as a supply of high pressure argon. Periodically, all valves, lubricators, filters, fillings, and piping are cleaned, tightened, and checked. Crankcase oil is drained, cleaned, and refilled on a periodic maintenance schedule. No abnormal operation or maintenance has been required on this unit and it has performed exceptionally well.

D. Cooling Fans

The cooling fans are cleaned, greased, and generally inspected, periodically. In connection with the fans, there is a water system for cooling. This water system is cleaned and the automatic valve checked and adjusted to insure proper operation. Along with this part of the water system, the rest of the water system, including the water recovery tank and pumps, is periodically cleaned and inspected. No problems have been encountered with this unit and it is working perfectly.

E. M. G. Sets and Motors

The mechanical and electrical inspection of these units is a routine operation. Also greasing and oil changes are performed per the manufacturers instructions. No problems have been encountered with these units and they are operating perfectly.

F. Leaks in Room

On March 21, 1961, the room developed a leak which resulted in a substantial loss of argon. Every effort was made to locate and stop this leak as quickly as possible and the following is a description of all methods investigated and attempted to find the leak.

1. Soap Checking

Using a mixture of soap and water, which will bubble on the low pressure side when applied to a leak, all door seals, weld seams, windows, welding ports, and piping connections were checked with negative results.

2. Helium Additions

When the soap checking failed to reveal the leak, the use of helium in conjunction with Consolidated Electrodynamics Corporation's helium leak detection equipment was attempted. This method proved unfruitful also, but in using it a very important fact was brought to light. This fact was that helium causes all instruments to react the same as they do toward hydrogen, so that all electrically operated hydrogen valves must be hand operated when helium is present in the gas stream. The reason for this behavior is the high conductivity of helium gas which is very similar to hydrogen.

3. Freon 12 Additions

After using the aforementioned detection methods for six days with no results, the decision was made to shut down the system to conserve gas and allow for the use of other detection methods. The Freon 12 method for leak detection could not be used while the purification plant was running because this refrigerant gas would be permanently frozen out and trapped in the fractionating columns and result in a complete shutdown and defrost of the purification system.

With the use of approximately one-half tank of Freon 12 in air and an electronic detection device, the leak was located in two hours and has since been repaired. The leak that was present was below floor level and was also enclosed in a water pump system; hence, the reason for the failure of soap checking and the light helium gas detection method in finding the leak.

Other methods were suggested and investigated during the course of leak detection, and these are below with their respective disadvantages.

1. Use of Colored Smoke

These compounds are generally CO_2 and such compounds would freeze up the purification plant in much the same manner as Freon 12. Also, colored smoke would dissipate too rapidly in an area as large as the InFab enclosure and would not allow for pinpointing of the leak.

2. Odoriferous Gas Additions

The possibility of using an addition such as that used in natural gas was suggested and would have been useful except for one hazardous compound present in the gas. The compound is sulfurous in nature and presented the possibility of formation of sulfuric acid in the purification system and this would be disadvantageous for a corrosion standpoint.

When leaks develop in the room in the future and cannot be found, it is suggested that the argon be removed and Freon 12 used for leak detection. This will minimize down-time and lead to more efficient operation of the room.

G. Magnetic Brake on In-Room Crane

The magnetic brake on the ten-ton crane was damaged when a locking key came loose inside the motor unit. Another brake coil was ordered and placed into service. While the unit was being repaired, it could still be operated except compensation had to be made for the action of the brake. The installation of the new coil has allowed the complete use of this unit once again.

H. Painting of Room Floor

Periodically, it is required to paint the floor of the InFab enclosure because of the accumulation of oil and dirt. This accumulation allows contaminants to collect and be given off when the floor becomes warm. It is necessary to paint this floor everytime the room is shut down to minimize the collection of oil and dirt.

I. Water Leakage

On two occasions, water leakage into the InFab facility has occurred. On both occasions, the reason for this leakage was a build-up of pressure when the balance of the plant shut down and a resultant breakage of rubber connecting lines on induction coils. The water can be cleaned from the room without encountering serious problems but there is the difficulty of lost time because of these occurrences. To prevent excessive amounts of water from collecting, a water activated alarm system has been installed. This system has been installed in the drain valve of the impacter pit (the lowest point in the room) to afford early detection of any water in this pit. Water in contact with this unit automatically triggers an alarm in the vicinity of the room and in the purification area to insure early detection of this condition.

J. Man Locks

All man locks associated with the InFab enclosure have fittings on such items as the purge system, the door seals, etc. All of these items must be continually checked to insure proper operation of the lock and assure that leakage to and from the lock is not occurring. There has not been any difficulty with the man locks that cannot be taken care of with normal preventive and corrective maintenance.

IV. Personnel Suits

The personnel suits can be divided into three distinct categories, i.e., man-air breathing system, suit enclosure, and communications system. An attempt will be made to treat each of these items separately and list their problems and solutions.

A. Man-Air Breathing System

One of the first problems that developed in this system was the periodic burn-up of electric motors which act to circulate the atmosphere within the suit. When it was finally determined that these motors would not sustain continued operation in the argon atmosphere, other means of circulating the internal air were investigated. Since electric motors also interfered with the communications, attempts were made to locate a device which would not cause electrical interference. The device finally settled upon was an air driven motor that would operate from high pressure argon. Since the high pressure argon was already available from the argon compressor used in conjunction with the impacter, this posed no problem. All suits were subsequently changed over to the argon motors and no problems with these suits have occurred after two years of use.

As the work load in the InFab facility increased, several deficiencies in the man-air breathing system became apparent.

1. The time of operation of the men in a suit was inadequate by a factor of four.

2. The time required for servicing back packs was too long for efficient operation.

3. Back pack cooling was inadequate and resulted in heat prostration on several occasions.

In discussions with the Firewel Corporation, manufacturers of the suit, it was discovered that they had developed a leak-proof system for Los Alamos Scientific Lab and Savannah River. This system continuously supplies dry, refrigerated air to a person totally enclosed. Quotes were obtained on this system and costs submitted to the Navy. Approval was received and the installation will be made to c er the three items listed above. This will now allow a man to operate in an enclosed suit for as long a period as personal hygiene will allow him. It is anticipated that we will operate on four hour shifts.

There was one explosion that occurred during operation that was associated with the back packs. With an operator fully suited, he was placed on the man-air breathing system by turning on the oxygen valve. As the switch was thrown, a considerable amount of sparks, smoke, and a loud bang occurred. The operator smelled something burning and immediately stepped from the suit. It was at first thought that oil or grease had gotten into the oxygen supply line. To confirm our suspicions, the entire suit was sent to Firewel Corporation for repair and investigation of the cause of failure. The investigation revealed that the explosion resulted from a minute high pressure leak of oxygen. The leak occurred at the Kel-F seal used in the control system of the back pack and was known to have occurred twice before - in a high altitude pressure suit and a ground level contamination suit. Firewel indicated that there is no way to predict such an occurrence and the only possible solution is more rigid control of their inspection operation of materials. Firewel assured Universal-Cyclops that more rigorous controls would be used in the future.

The emergency oxygen supply bottles used in conjunction with the man-air breathing system have functioned satisfactorily with a minimum of maintenance and repair. These units will continue to be used when the new system is installed.

B. Suit Enclosure

As in the case of the man-air breathing system, continual use of the suits pointed out deficiencies of operation as follows:

1. Internal suit connections were clumsy and inadequate for continuous usage.

2. The helmet visor was an annoying problem from both the standpoint of location and ease of positioning.

3. The outside of the suit required redesign so that it could be more easily and quickly removable when working the rubber and nylon parts of the suit.

The David Clark Company was contacted for a resolution to these problems and they submitted a quotation which was subsequently approved and the new system is being incorporated into the suit enclosures.

Problems have been encountered with leaking gloves. The original gloves received with the suits were made in two pieces. The defect appeared to be due to aging of the sealing cement and subsequent parting of the seam. These old gloves have been repaired and no subsequent problems have been encountered. However, all gloves that have been ordered since that time are of one piece construction so that this problem should not arise again.

All other units of the suit enclosure have performed excellently with a minimum of maintenance. However, considerable care must be exercised to insure continued maintenance-free operation of the suits. We have requested funds from the Navy for specialized cleaning and airing facilities to minimize any deterioration of the suits. We feel that this facility is absolutely required to obtain maximum efficiency and long life for the units.

C. Communications System

The communication system head sets have been a constant source of difficulty. The system worked perfectly after removal of the interference caused by the electric motor used to move air about the suit. However, constant repair was required because of the light construction of the units. They were built for an operation where light or very fine work was to be performed. Because of the heavy abuse associated with InFab operations the handling of these units posed a continuous problem.

Since the design of these units, the David Clark Company has had considerably more experience in this type of communication and a new head set tried. The results were very good and a quotation was obtained. This information was forwarded to the Navy and they approved new head sets which are presently being incorporated into the system.

This new system will utilize the present amplification system in conjunction with an improved receiving head set and improved carbon microphone. This system will be much more rugged and is more suited for the InFab application.

D. Safety Procedures

The most important consideration in the operation of the personnel suits is the safety of the operator. The first procedure is to select competent personnel. The means of selecting personnel is outlined in Appendix B. It should be noted that detailed physical and mental testing is required before an applicant is considered qualified to be an in-room operator. After this detailed selection procedure is followed, a man must put in thirty hours outside the room before he is allowed to enter the room and perform any operations.

Upon each entry into the room, the in-room operator must go through a check out procedure as shown in Figure 2. This procedure involves being checked by two observers to insure that an observation not made by one would be picked up by the other. Since this procedure was initiated, no difficulties of forgetting or not properly checking have been encountered. We presently feel that the safety procedures associated with the in-room operations are adequate and no further changes in procedure are anticipated.

Presently there are four qualified InFab operators - L. Gill, F. Peduto, T. Raineri, and G. Winnette. All operators have considerable in-room time and work harmoniously as a team. We hope that we can keep this combination together in the future.

V. InFab Activities

In order to promote the availability of the InFab room and its capabilities, a Fact Sheet (Appendix E) was prepared. This fact sheet has been distributed, along with the InFab Brochure (Appendix F), to thoroughly acquaint the technical community with the InFab facility. Approximately 10,000 of the Fact Sheets and InFab Brochures have been distributed to engineers and scientists throughout the country.

Before the InFab personnel will perform any work within the InFab enclosure, they require that an InFab Activity Report, Figure 3, be filled out. This report indicates the work to be accomplished and all other pertinent information. The InFab operators then fill out the balance of Figure 3 and Figure 4 as the work progresses. This system has resulted in a complete compilation of all InFab operations. From these sheets, the engineers will calculate all of the required information for subsequent issuance of technical reports.

FIGURE 2

PRE IN-FAB ENTRY NORMAL CHECK OUT PROCEDURE

NAME__________ DATE________ TIME IN________

OBSERVERS; 1.__________ 2.__________

CHECK OUT LIST

ITEM NO.	DISCRIPTION	CHECK
1	VISUAL INSPECTION OF SUIT	
2	CHECK EMERGENCY O_2 SUPPLY	
3	CHECK CO_2 ABSORBENT	
4	INSPECT O_2 SUPPLY CYLINDER	
5	MECHANICALLY CHECK BACK PAK	
6	PUT ON SUIT	
7	CHECK MID SEAL	
8	ADD ICE TO BACK PAK	
9	CHECK HELMET PASSAGES & CONNECTIONS	
10	PLACE PAK & CONNECT COOLING SUPPLY HOSE	
11	CONNECT & TEST COMMUNICATIONS	
12	PLACE HELMET	
13	CONNECT O_2 HOSE	
14	CONNECT EMERGENCY O_2 HOSE	
15	CONNECT EXHAUST HOSE	
16	OPEN O_2 SUPPLY, PRESSURE SUIT	
17	PLUG IN POWER TO PAK	
18	INSPECT SUIT FOR LOSS OF PRESSURE	
19	OPERATE FOR 3 MINUTES OUTSIDE ROOM	
20	GULP TEST CHECK REGULATOR	
21	PSI OXYGEN AT ENTRY TIME	

TIME OUT________

REMARKS --

FIGURE 3

UNIVERSAL-CYCLOPS STEEL CORPORATION
REFRACTOMET DIVISION

IN-FAB ACTIVITY REPORT

Date ________

ORDER INFORMATION

Customer ________ Customer Order No. ________ Mill Order No. ________

Date Received ________ Date Promised ________

IDENTIFICATION

Job No. ________ Project No. ________ Material ________

Heat No. ________

REQUIREMENTS & ACTIVITY

Roll ☐ Forge ☐ Welding ☐ Heat Treatment ☐ Other ☐ Date of Working ________

Starting Size ________ Finish Size ________

Time in Room ________ Time Out of Room ________

Time in Furnace ________ Time Out of Furnace ________

Remarks: ________

____

____

In-Room Operators ________ ________ ________

____
Engineer in Charge

FIGURE 4
INFAB DATA COLLECTION

InFab Gas Composition	Start	1st Hour	2nd Hour	3rd Hour	4th Hour	5th Hour	6th Hour	7th Hour	8th Hour
Oxygen									
Nitrogen									
Other									
Argon									

FORGING RECORD

Reheat No.	KW	Temp.	Time		Working Temperature				Finish		Remarks
					Start		Finish				
			In	Out	Optical	True	Optical	True	Time	Size	

ROLLING RECORD

Pass No.	KW	Temperature	Time		Size		Mill Speed	Remarks
			In	Out	In	Out		

A. Powder Metallurgy

1. Pressing and Sintering Characteristics

Work has been accomplished on pressing and sintering in conformance with this contract and the following information is supplied.

As the initial stage in the evaluation of pressing and sintering characteristics of three powders, a number of isostatic pressings were made in 1-3/8" diameter cans. The bag material was .006" Visten. The results of these initial pressing experiments and the size bars produced are shown in Table I.

Since the starting can size was constant, it is readily apparent from the diameter, length, and pounds, the differences in apparent density among the various materials. The M & R powder, which had the lowest apparent density, produced the smallest bars from the same can. Also, the least weight could be placed in the can when using the M & R Powder.

The green densities shown in Table I were measured after presintering the pressed compacts for one hour at 1800°F. This presintering was necessitated because the as-pressed compacts were too fragile to handle. The presinter at 1800°F did not change the physical dimensions of the bars, and, therefore, should not have changed the density. However, the presinter did allow the bars to be more readily handled without the fear of breakage. Apparently, there is micro sintering that takes place during this operation but not macro sintering.

It should be noted from the data in Table II, that the Sylvania powder attained the highest presinter density of the three powders evaluated. Thus, at 30,000 psi, it apparently compacts more readily than either the General Electric or M & R powder. The M & R powder consistently had the lowest presinter density while the General Electric powder was slightly higher.

Typical pressed bars after presintering are shown in Figure 5. The differences in diameter are readily discernible even though the bars came from the same pressing

TABLE I

RESULTS OF INITIAL PRESSING EXPERIMENTS ON MOLYBDENUM POWDERS

Powder Supplier	Pressing No.	Pressure (psi)	Diameter (in.)	Length (in.)	Weight (lbs.)	Green Density Percent of Theoretical (1hr./1800)
G. E. Cr2966	IP 48 A	30,000	1-3/32	13-3/32	2.87	61.8
	B					62.7
	C					62.7
	IP 51 A	30,000	1-3/32	13-3/8	2.96	63.8
	B					62.9
	C					63.0
	D					64.9
	E					62.9
	F					62.2
	IP 55 A	30,000	-	-	-	62.8
	B					62.7
	C					62.7
	D					62.5
	E					62.6
	F					62.9
Sylvania Mo 530	IP 50-1 A	30,000	1-3/32	11-1/4	2.14	65.6
	B					65.2
	C					65.3
	D					65.3
	E					65.3
	IP 50-2 F	30,000	1	11-5/8	2.19	68.6
	G					65.5
	H					65.6
	I					65.5
	IP 55 A	30,000	-	-	-	65.46
	B					65.43
	C					65.61
	D					65.60
	E					65.59
M & R Assay 5411	IP 51-1 A	30,000	7/8	10-13/16	1.6	61.8
	B					62.0
	C					62.0
	D					62.0
	E					62.1
	IP 51-2 F	30,000	7/8	10-5/8	1.58	61.8
	G					62.1
	H					61.7
	I					61.8
	IP 55 A	30,000	-	-	-	61.9
	B					61.2
	C					61.9
	D					62.0
	E					62.2

TABLE II

RESULTS OF INITIAL SINTERING EXPERIMENTS ON MOLYBDENUM POWDERS

Powder Supplier	Sintering Temperature (°F)	Sintering Time Hours	Density Percent of Theoretical
G. E.	2750	4	87.6
Sylvania	2750	4	86.7
M & R	2750	4	92.1
G. E.	2750	8	89.7
Sylvania	2750	8	89.4
M & R	2750	8	94.7
G. E.	2750	24	93.2
Sylvania	2750	24	92.4
M & R	2750	24	96.4
G. E.	2850	4	89.1
Sylvania	2850	4	89.8
M & R	2850	4	96.0
G. E.	2850	8	91.7
Sylvania	2850	8	91.1
M & R	2850	8	96.8
G. E.	2850	24	94.3
Sylvania	2850	24	93.5
M & R	2850	24	96.6
G. E.	2950	1	84.9
Sylvania	2950	1	84.9
M & R	2950	1	91.0
G. E.	2950	2	87.4
Sylvania	2950	2	87.2
M & R	2950	2	94.0
G. E.	2950	4	91.5
Sylvania	2950	4	91.0
M & R	2950	4	96.0
G. E.	2950	8	91.7
Sylvania	2950	8	92.1
M & R	2950	8	96.7
G. E.	2950	24	95.6
Sylvania	2950	24	94.2
M & R	2950	24	96.8
G. E.	3100	4	91.9
Sylvania	3100	4	91.3
M & R	3100	4	95.8
G. E.	3100	8	93.5
Sylvania	3100	8	91.6
M & R	3100	8	96.0
G. E.	3100	24	95.3
Sylvania	3100	24	93.3
M & R	3100	24	97.0

FIGURE 5 - Pressed and Presintered Molybdenum Bars, 0.4X

can. Of special interest is the relative straightness of these bars on the initial trials.

After the presinter operation, the pressed bars were machined to a uniform diameter and cut into lengths of approximately two inches. These pieces were then full sintered for various times at various temperatures. The densities attained after the full sinter are shown in Table II and several plots of the data are shown in Figures 6, 7, 8, and 9. Additional means of plotting the data could be used depending upon the information desired.

Figure 9 is of particular significance for it shows the results of an eight hour full sinter of the material at various temperatures. This is the maximum time desired for this operation on a production basis. At 2850°F, for example, the M & R powder will sinter to 96.8% of theoretical density while the General Electric and Sylvania powder will only attain 91.7% and 91.1% respectively. From the standpoint of attaining high density for further fabrication, this is undoubtedly advantageous. However, the disadvantages incorporated by a low apparent density and the problem of obtaining low oxygen values in material of larger sizes still must be resolved.

Figure 10 shows the machined cylindrical bars after the full sintering treatment. More readily apparent at this stage is the difference in size produced from the same pressing can. These sizes are directly comparable to the apparent density of the starting material.

The microstructures of the full-sintered material after sintering for eight hours at 2850°F, 2950°F, and 3100°F and twenty-four hours at 3100°F are shown in Figures 11, 12, 13, and 14, respectively. It will be noted that the three powders give sintered grain sizes that are approximately the same after each sintering cycle examined.

However, the density achieved in the M & R samples is higher than that of the other two powders in all cases. This can readily be seen by the amount and size of voids present in all of the full-sintered samples. After an eight hour sinter at 2850°F, the M & R powder gives a density approximately 5% greater than the other two powders.

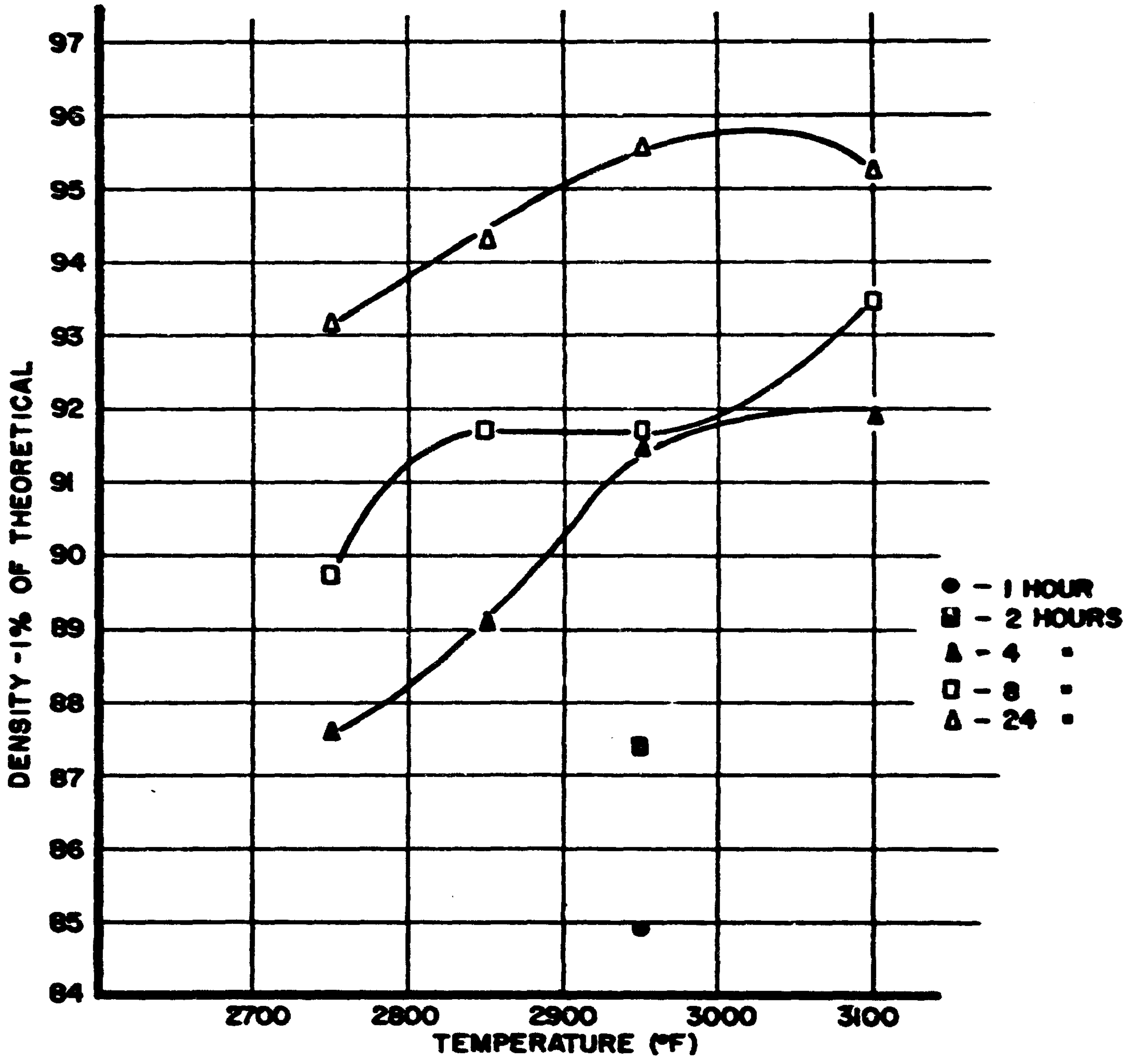


FIGURE 6

SINTERING CHARACTERISTICS OF GENERAL ELECTRIC MOLYBDENUM POWDER – LOT Cr 2966

RTA-2179

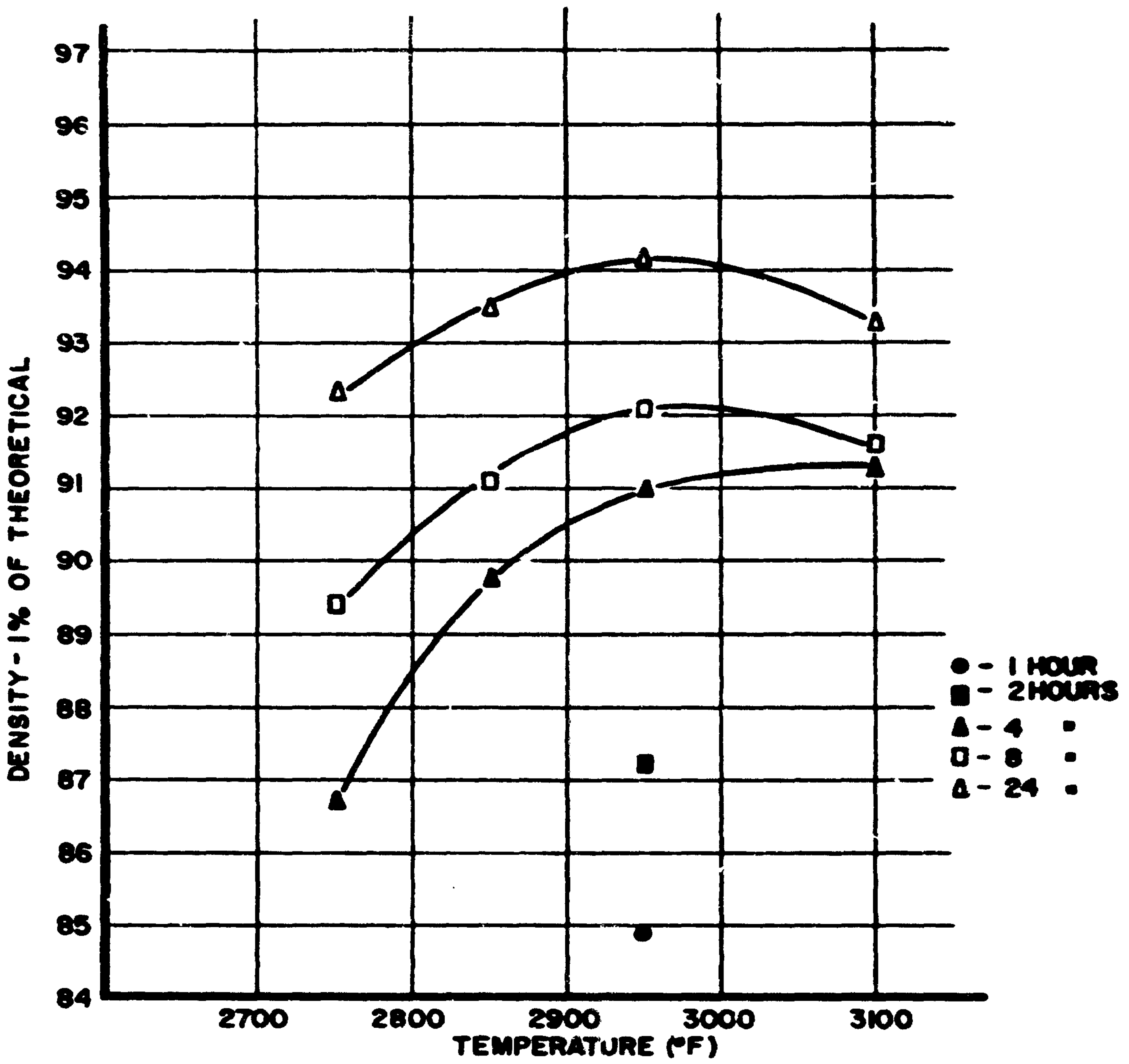


FIGURE 7

SINTERING CHARACTERISTICS OF SYLVANIA ELECTRIC MOLYBDENUM POWDER — LOT Mo 530

RTA-0180

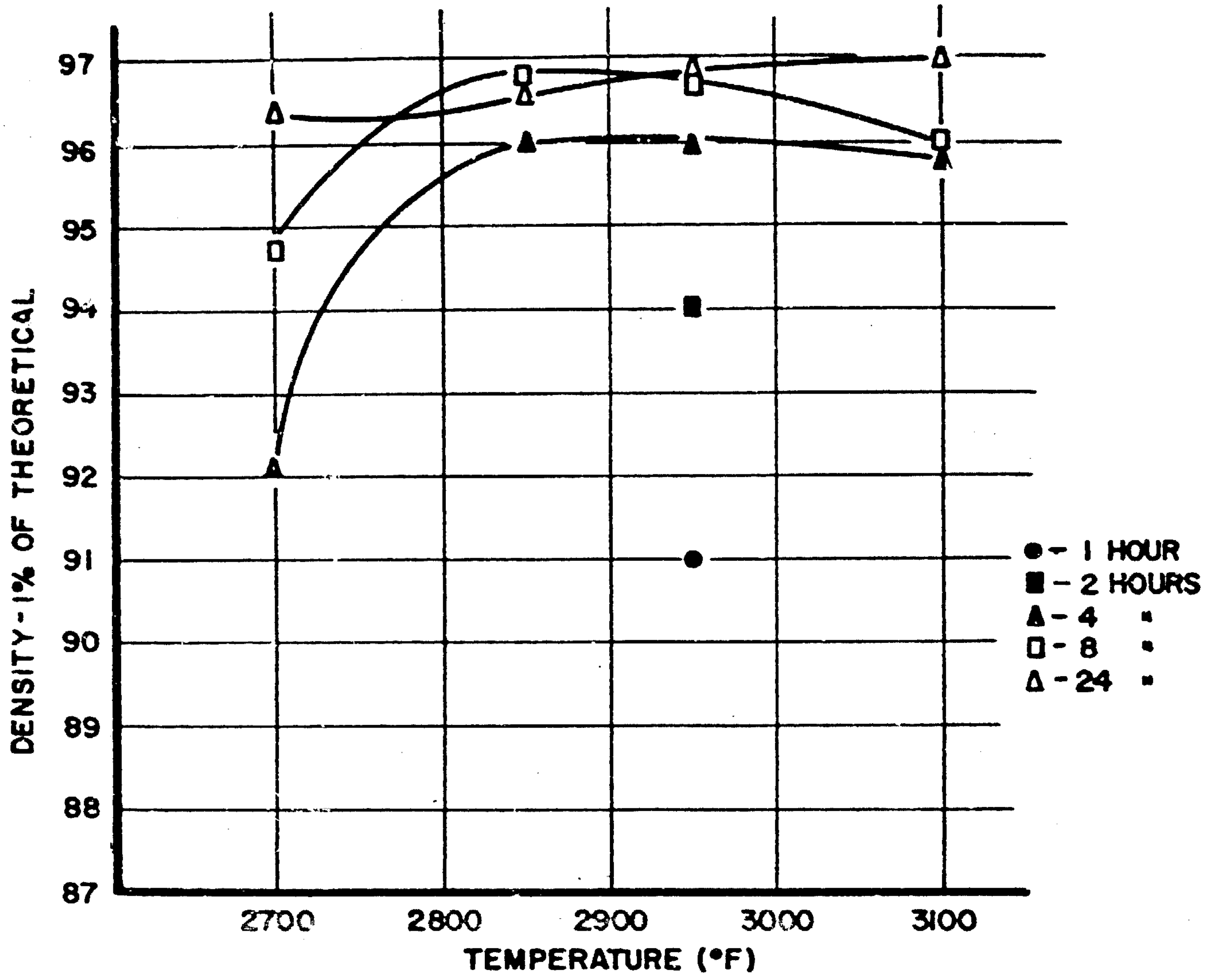


FIGURE 8

SINTERING CHARACTERISTICS OF METALS & RESIDUES
MOLYBDENUM POWDER - LOT ASSAY 5411

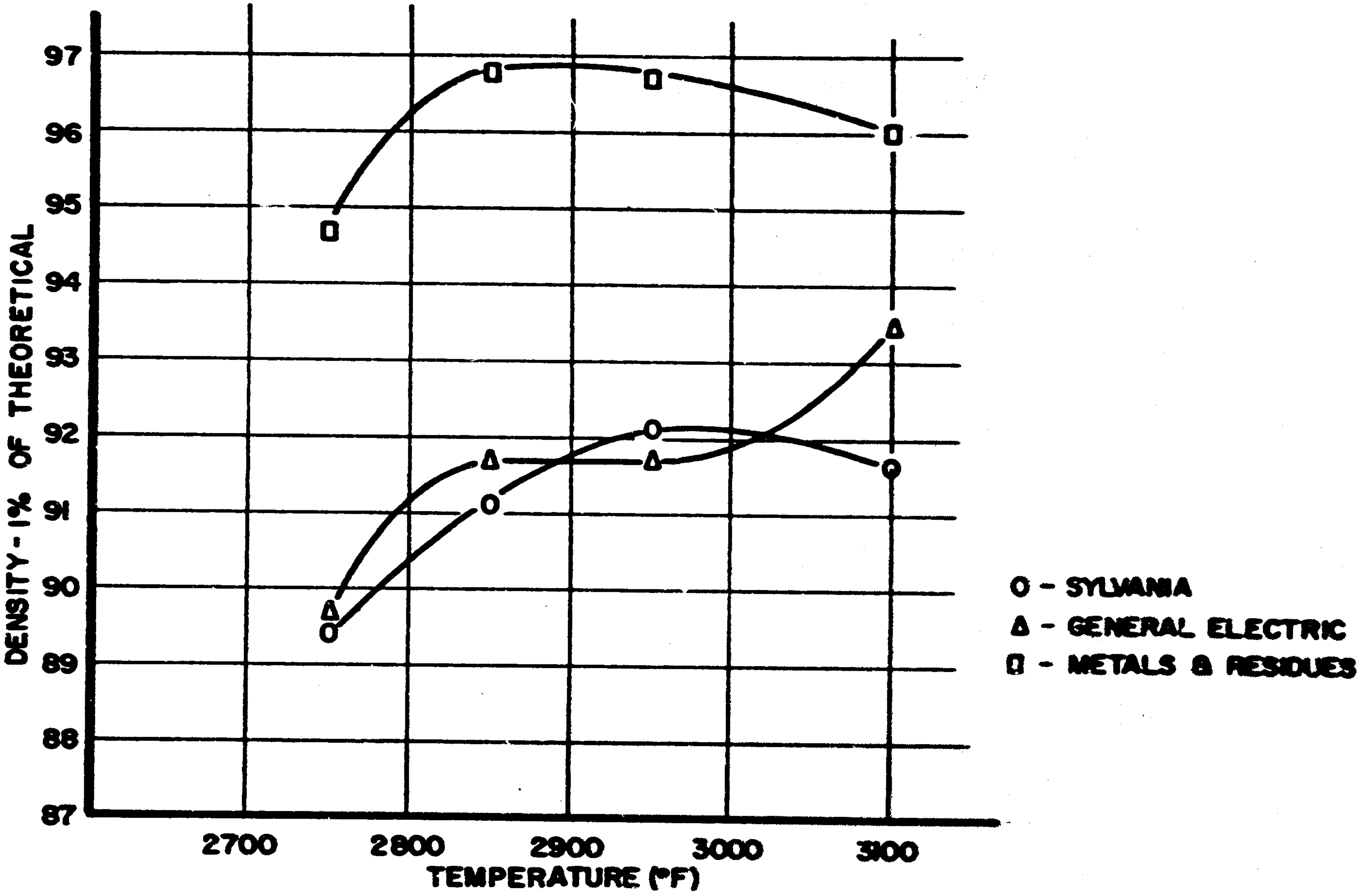


FIGURE 9

COMPARISON OF SINTERING CHARACTERISTICS OF GENERAL ELECTRIC, SYLVANIA AND METALS & RESIDUES MOLYBDENUM POWDER FOR AN 8 HOUR SINTER

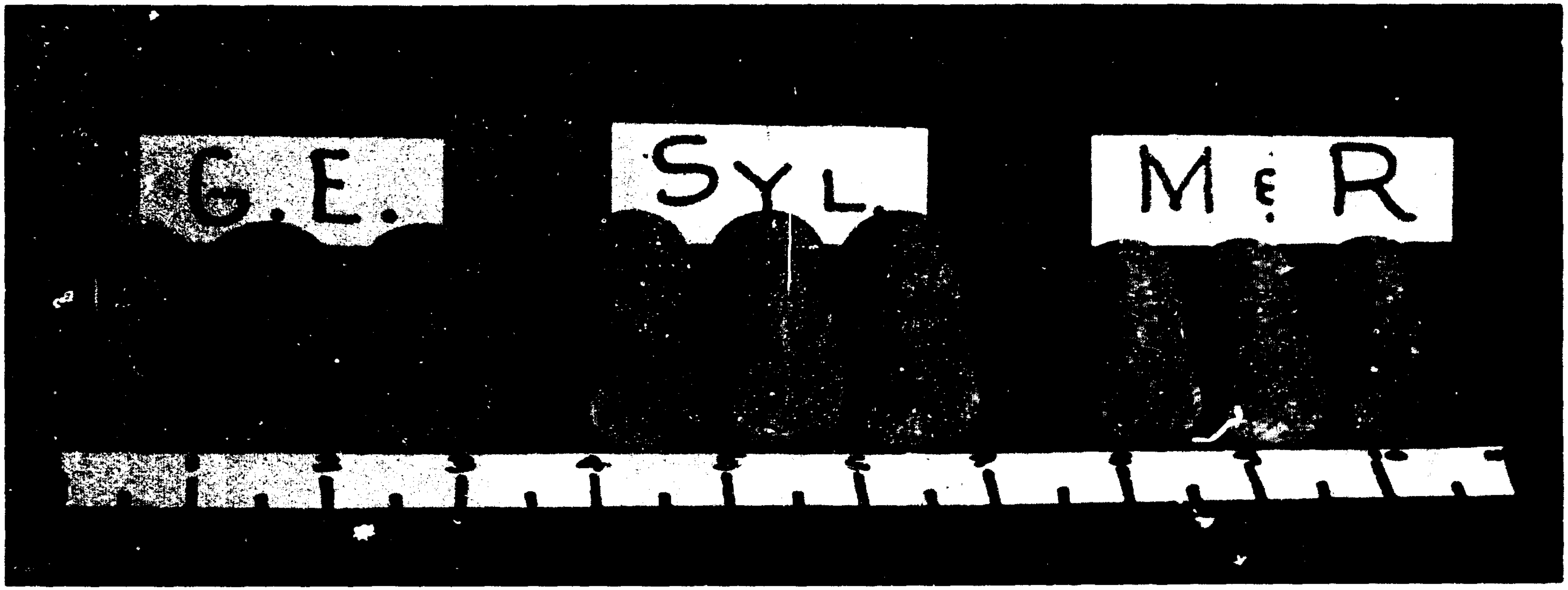
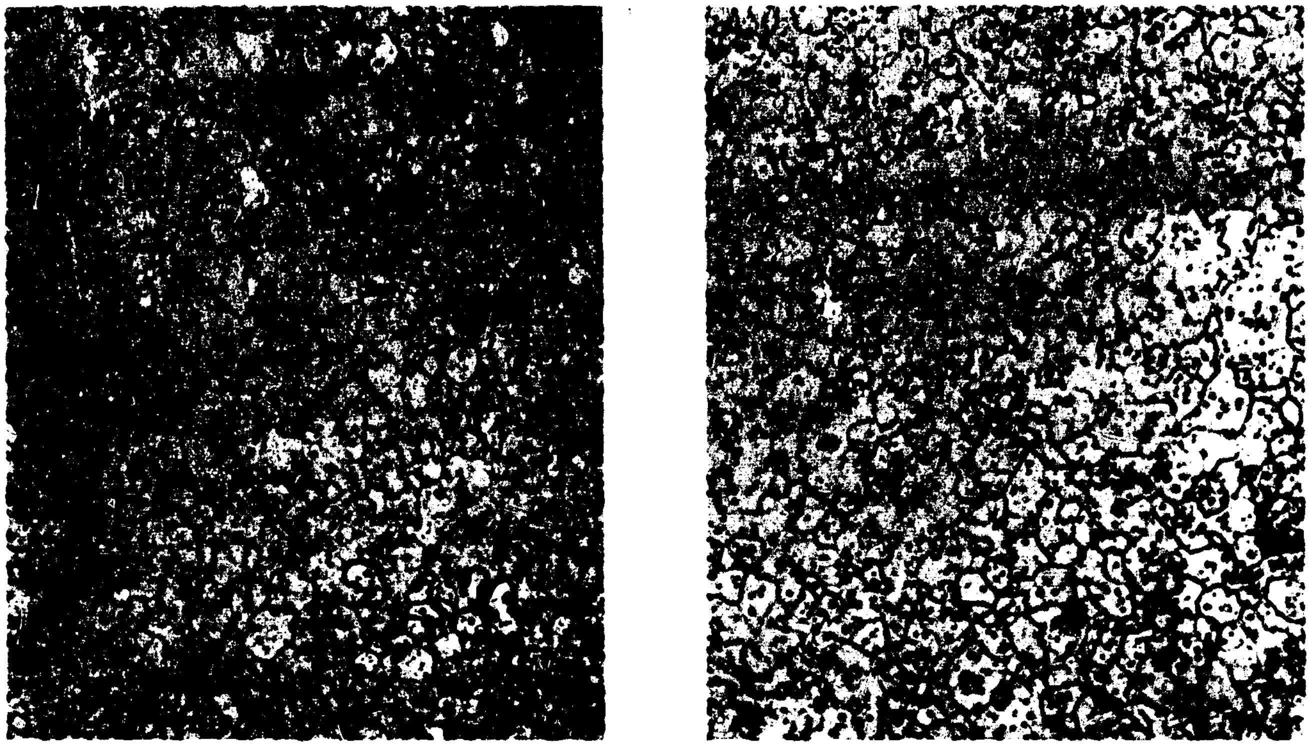


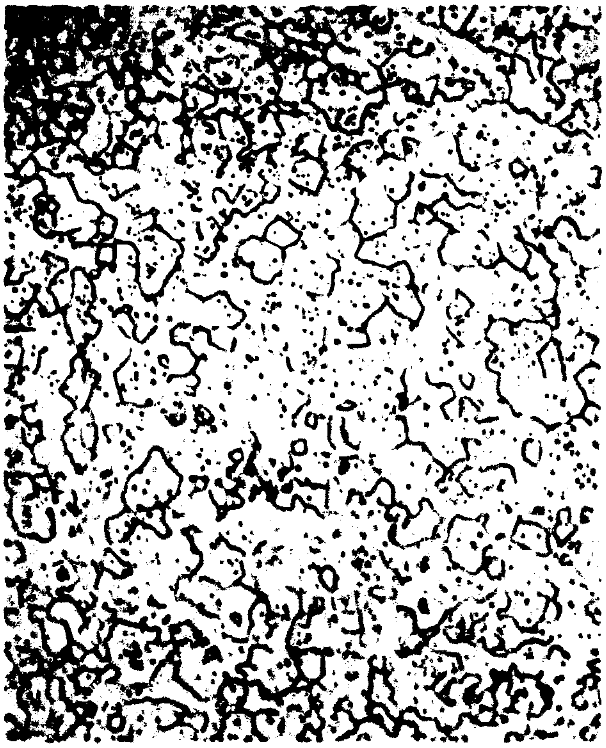
FIGURE 10

Typical Machined Cylindrical Bars After Various Sintering Cycles, 1X



General Electric Cr2966

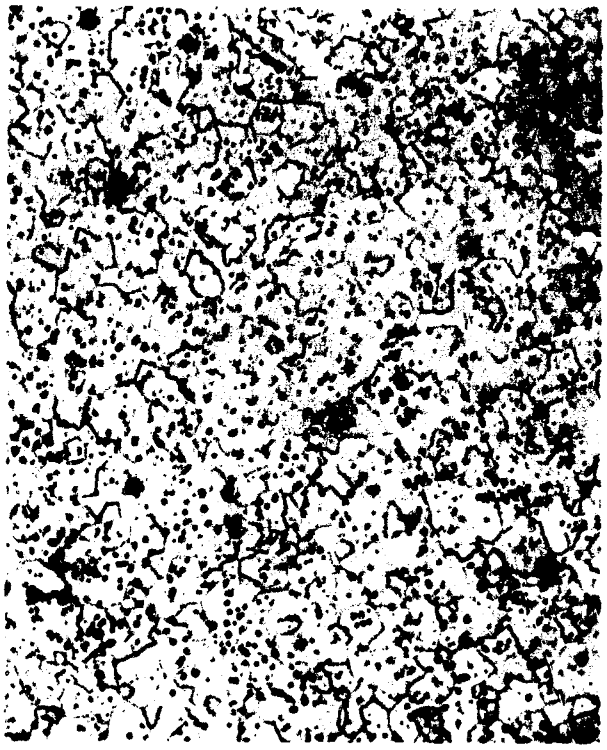
Sylvania Mo 530



Metals and Residues
Assay 5411

FIGURE 11

Microstructure After Sintering For
8 Hours at 2850°F, 200X



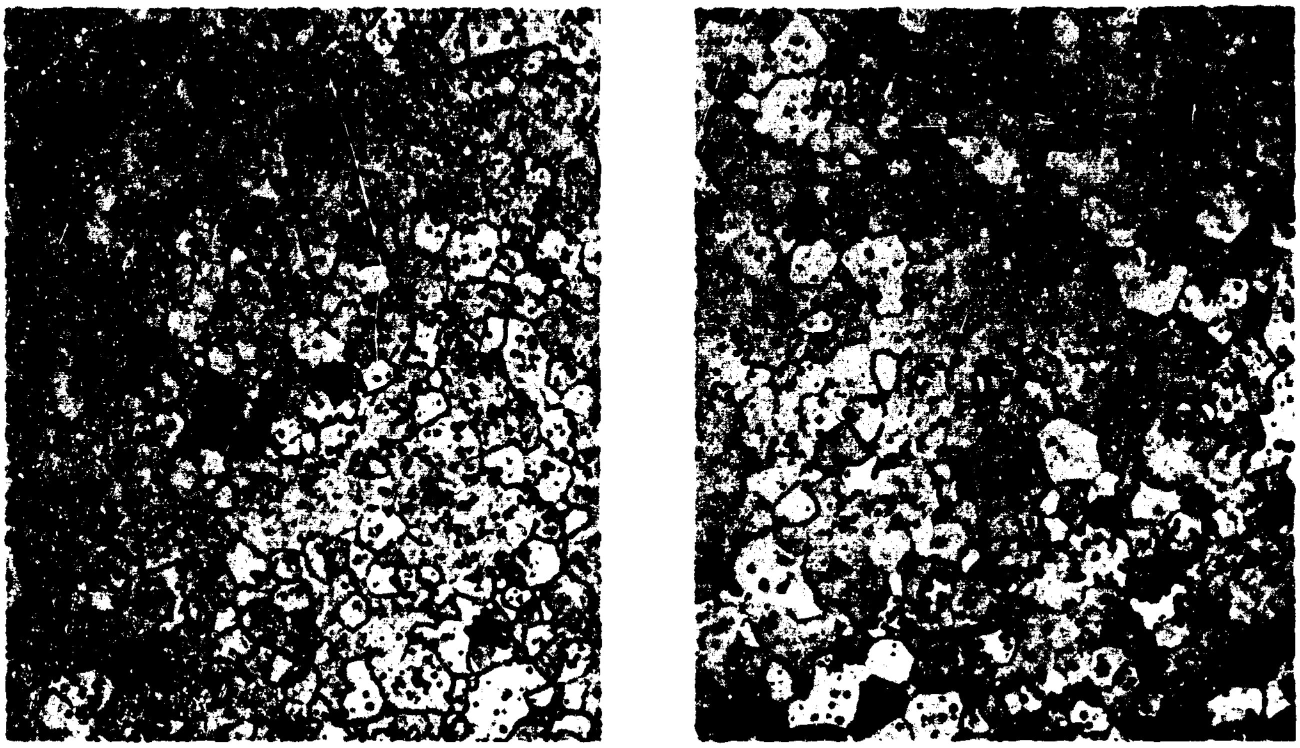
General Electric Cr 2966

Sylvania Mo 530

Metals and Residues
Assay 5411

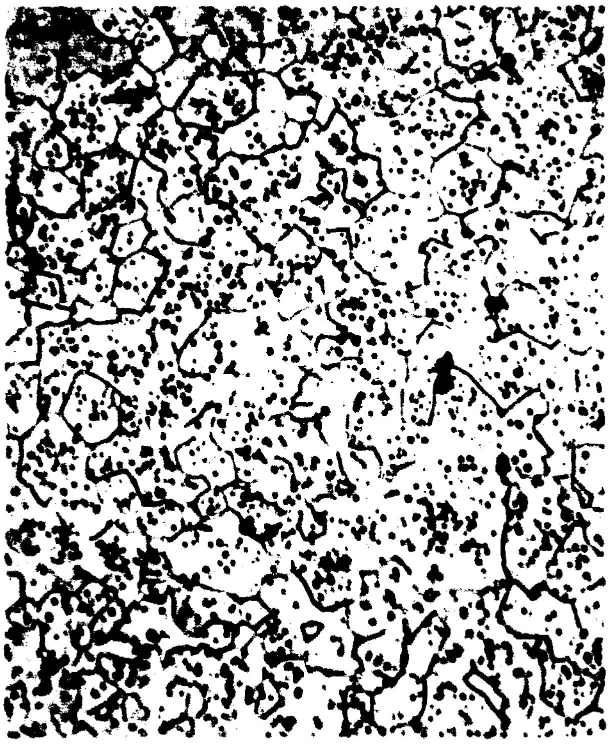
FIGURE 12

Microstructure After Sintering for 8 Hours at 2950°F, 200X



General Electric Cr2966

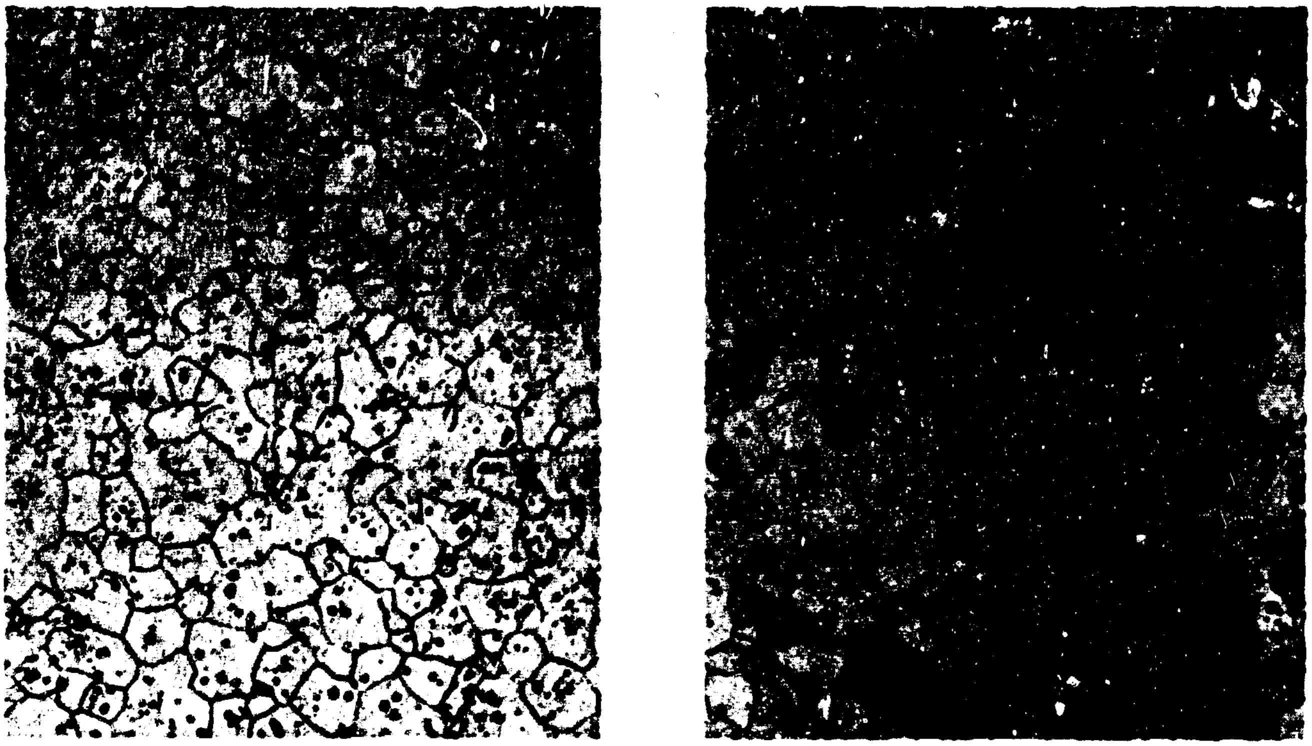
Sylvania Mo 530



Metals and Residues
Assay 5411

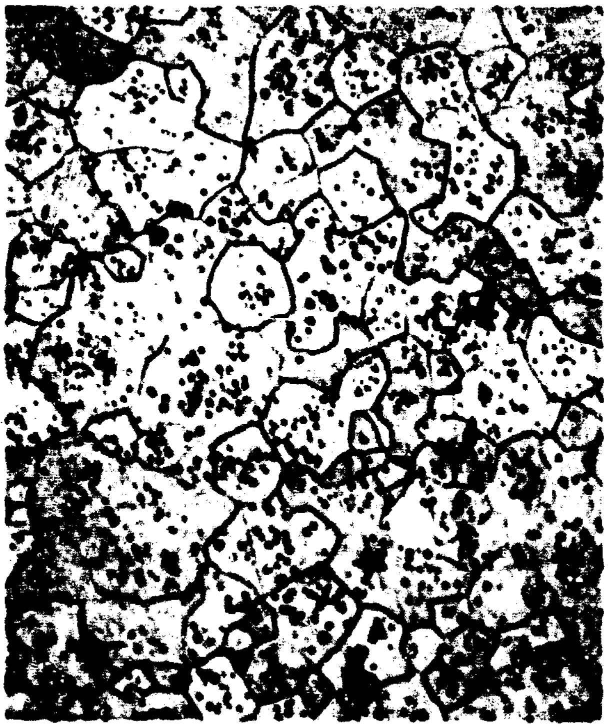
FIGURE 13

Microstructure After Sintering For 8 Hours
at 3100°F, 200X



General Electric Cr2966

Sylvania Mo 530



Metals and Residues
Assay 5411

FIGURE 14

Microstructure After Sintering For 24 Hours at 3100°F. 200X

After the full-sinter experiments were complete, several small bars were submitted for chemical analysis. The results of this analysis are shown in Table III. General Electric has stood for the best in residual chemistry after sintering. It can be seen from Table III that the M & R results compare favorably except for oxygen. This is undoubtedly due to the finer particle size and will have to be taken into consideration when working with the M & R powder. Methods of controlling the oxygen content at low values are under consideration and no difficulty is anticipated in controlling this element.

2. Scale-Up of Pressing and Sintering

In addition to the small experimental sintering specimens accomplished in the General Electric laboratory furnace, a number of larger pressings at 30, 000 psi were made and sintered in the Lindberg sintering furnace. The pertinent data on the scale-up of pressing and sintering studies is shown in Table IV. The long times at 1800°F for some of the sintering cycles were due to a one shift operation. These would not be used in production. The effect of these long times should have no effect on sintered density, but they would have a tendency to produce lower oxygen contents than may be expected in production.

The as-sintered bars resulting from pressings IP70, IP71, IP72, and IP73 are shown in Figure 15. These bars were sintered both in contact with the high alumina hearth plates on the furnace cars and in sheet molybdenum trays filled with fine alumina powder. In both cases, sound sintered shapes were obtained. Based on this preliminary work, no conclusions can be made as to the most suitable medium of sintering. Since the shapes used on the furnace car hearth have cracked and fallen down, their replacement is a necessity. If the present plans are followed and they are replaced with molybdenum plate, the use of fine alumina powder between the base and piece to be sintered seems the most practical solution to preventing any possible bonding between the bar and work piece. The fine alumina will also act as a "lubricant" and facilitate the movement during the required shrinkage associated with sintering.

The linear shrinkage from the initial can size to the final sintered size (final density between 94% and 95% of theoretical) is as follows:

TABLE III

CHEMICAL ANALYSIS AFTER SINTERING

Element	General Electric (Cr 2966)	Sylvania (Mo 530)	Metals & Residues (5411)
Si	26	55	22
Cr	3	33	3
W	< 100	< 100	< 100
Ni	< 1	< 1	< 1
Al	6	30	6
Cu	< 1	< 1	< 1
Fe	25	22	28
C	88	77	77
O_2	12-20	22-27	20-24
N_2	< 3	< 3	< 3
H_2	2-7	2-3	2-5

FIGURE 11 - As-Sintered 4" Diameter Bars

TABLE IV

PERTINENT DATA ON SCALE UP OF PRESSING AND SINTERING STUDIES

Pressing No.	Size		Density	Sintering Time (hrs.)	Cycle
	Green	Full-Sinter	Full Sinter		
IP64 (1)	2-1/8x9"	1-27/32" rd. x 7-3/4 (2)	96.1%	4, 4	1800°F, 2800°F
IP65 (3)	2-1/8x9"	1-29/32" rd. x 7-5/8 (2)	96.3	4, 4	1800°F, 2800°F
IP66 (3)(6)	-	2-1/32" rd. x 6-3/4 (2)	-	4, 5	1800°F, 3200°F
IP67 (3)(6)	-	2-1/32" rd. x 6-13/16 (2)	-	4, 5	1800°F, 3200°F
IP68 (3)(6)	-	2-1/16" rd. x 6-11/16 (2)	-	4, 5	1800°F, 3000°F
IP69A(1)	-	1-7/8" rd. x 6-3/4 (2)	93.3	4, 6	1800°F, 3200°F
B(3)(7)	-	1-31/32" rd. x 7-7/8 (2)	94.5		
IP70	4-7/8" x 19-1/2"	- (4)	-	4, 16	1800°F, 3200°F
IP71 (3)	4-7/8" x 18	4-1/8" rd. x 14.5 (4)	94.2	4, 4	1800°F, 2850°F
IP72 (3)	4-7/8" x 19	- (4)	-	16, 4	1800°F, 2850°F
IP73 (3)	4-7/8" x 20-1/16"	- (4)	-	16, 4	1800°F, 2950°F
IP74 (3)	1-3/8x4-1/8x5-3/16	1-3/16x3½x4½ (5)	94.3	16, 4	1800°F, 2950°F
IP75 (3)	1-5/16x3-7/8x5	1-1/8x3-5/16x4-3/8 (5)	94.8	64, 4	1800°F, 2950°F
IP76 (3)	1-3/8x4-1/8x5-1/4	1-3/16x3-1/2x4-1/2 (5)	94.6	"	"
IP77 (3)	1-3/8x4-1/8x5-5/16	1-3/16x3-1/2x4-9/16	95.0	"	"

(1) M & R Powder Lot Assay 5411
(2) Can Inside Diameter - 3"
(3) M & R Powder Lot Assay 5524
(4) Can Inside Diameter - 7"
(5) Sheet Bar Can Dimensions - 2" x 5-13/16" x L
(6) Intentional Carbon Additions Prior to Pressing
(7) Sintered billet was cracked.

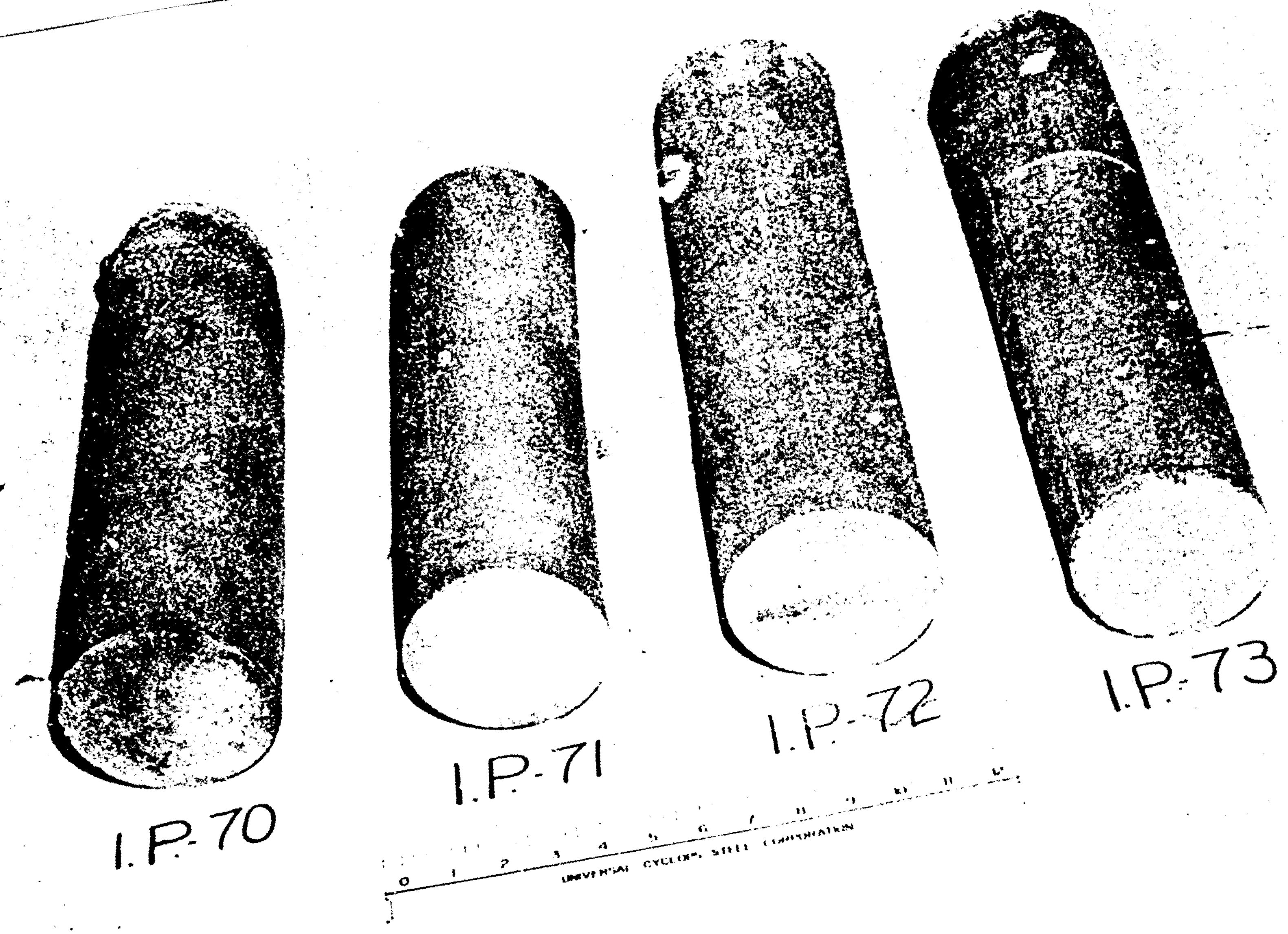


FIGURE 11 - As-Sintered 4" Diameter Bars

3" can to 1-7/8" diameter	37.8%
7" can to 4-1/2" diameter	41.1%
2" x 5-15/16" rectangular can	40.6%
1-3/16" x 3-1/2" sheet bar	41.0%

The average linear shrinkage is approximately 40%. The resulting volume shrinkage based on this value is approximately 78%. These shrinkage values are rather high in comparison to those for other powders. Sylvania, for example, uses a value of approximately 31% linear shrinkage for the powder being used in the pressed and sintered cylindricals and rectangular shapes being supplied to Universal-Cyclops for rolling.

This excessive shrinkage encountered with M & R powder from initial can size to final sintered shape results in two problems, the first being the limitation on the maximum billet cross section dimension that can be made using existing equipment. The practical inside diameter of the longest can that will fit the 12" gun bore is 11". This establishes the final maximum dimension to be approximately 6.5".

The second problem is one associated with maintaining the straightness of long pressings. Since the maximum length pressed and sintered to date is about 17 inches, a true evaluation of the straightness problem has not been accomplished. The straightness problem will be minimized since the isostatic pressing is being done in a horizontal position; however, the problem does exist and must be evaluated if an electrode bar length of 50" is to be made.

Analysis of the isostatically pressed bars sintered in the Lindberg furnace for interstitials is given in Table V. Several interesting observations can be made with regard to the carbon recovery in pressings IP66 through IP68. The carbon recovery (lathe chip analysis) in the 2" diameter bars is almost a linear function of the intentional carbon additions as shown in Figure 16. These bars were cut in half (3" lengths) and samples taken from the cut faces. The variation of carbon obtained from the solid samples taken at the center, mid-radius, and edge illustrates the problem encountered in using solid samples from a limited area to obtain carbon content of the entire bar. Lathe chips taken across the entire

TABLE V

CARBON, OXYGEN, AND NITROGEN ANALYSIS OF SELECTED PRESSED AND SINTERED BARS

Pressing Number and Location	Intentional Carbon Addition	Carbon Recovery	Oxygen
IP66	.030		
Center - Solid		.019	.0061
Mid-Radius - Solid		.022	.0070
Mid-Radius - Solid		.020	.0040
Edge		.010, .011	.0058
Lathe Chips		.013	
			.0057 Average
IP67	.050		
Center - Solid		.032	.0056
Mid-Radius - Solid		.032	.0015, .0020
Mid-Radius - Solid		.027	.0017, .0020
Edge - Solid		.010, .012	.0029, .0034
Lathe Chips		.028, .023	-
			.0031 Average
IP68	.070		
Center - Solid		.045	.0035
Mid-Radius - Solid		.052, .050	.0046
Mid-Radius - Solid		.048, .047	.0037
Edge - Solid		.019	.0064
Lathe Chips		.033, .037	-
			.0045 Average

		Oxygen	Nitrogen
IP74	Sheet Bar	.0140	.0007
IP76	Sheet Bar	.0105	.0003

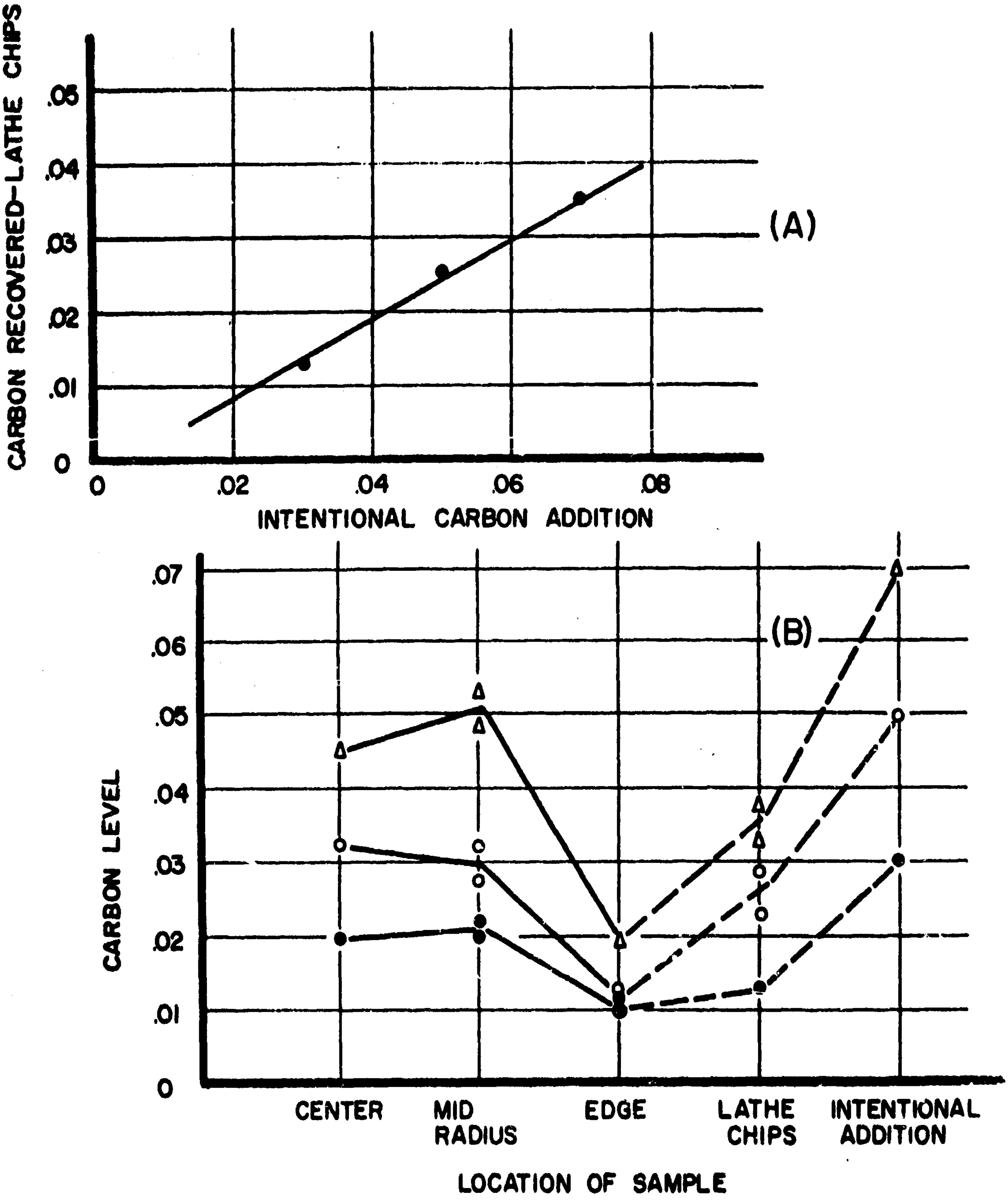


FIGURE 18

CARBON RECOVERY AND DISTRIBUTION VS
INTENTIONAL CARBON ADDITION

bar face, crushed, and blended prior to analysis, gives a much more reliable carbon value for the bar. If an average carbon value, based on the solid sample values weighted according to the areas (assuming unit length), is computed, the values of .015, .022, and .037 are obtained. These values are in fair agreement with the lathe chip values.

The oxygen values of the 2" diameter bars show no definite relation to the carbon content, either intentionally added or retained. The average oxygen value of the 2" diameter bars is 45 ppm as compared with about 22 ppm in the case of the 1" diameter bars. This difference could be explained due to differences in furnaces used for the sinter. However, it would be expected, based on size effect alone and should be carefully considered when attempting to produce low oxygen large diameter billets. If the oxygen level of the larger sections is to be reduced, the presinter time will have to be increased. The only approach to determining the times required at present is one based on trial and error.

3. Equipment Operation for Powder Facility

a. Bag Filling Operating Instructions

(1) Place powder charge in hopper.

(2) Place perforated metal container in pit. Remove cap.

(3) Heat seal bottom of Visten bag.

(4) Place rubber end plug in bottom of bag and clamp or strip bag to plug.

(5) Place top end of Visten bag in bag expander and expand bag until metal ring (same diameter as perforated metal container) can fit inside.

(6) Release pressure on bag expander and withdraw bag and ring.

(7) Place this assembly in perforated metal container.

(8) Start mechanical pump to expand bag to dimensions of container.

(9) Place spout from powder hopper over pit and clamp in place.

(10) Start metal container vibrating vertically by means of four way air valve to air cylinder below container.

(11) Start Syntron vibrators on powder hopper and spout.

(12) Open valve on powder hopper.

(13) When powder has all been transferred into the container, close valve and stop syntron vibrators and container vibrator.

(14) Unclamp hopper spout and remove from pit.

(15) Raise metal container partially out of pit by pressurizing air cylinder.

(16) Place rubber end plug inside bag on top of metal powder.

(17) Clamp or strap bag to plug.

(18) Insert hose through small hole in end plug.

(19) Evacuate bag through the hose.

(20) Remove hose and replace with small rubber plug.

(21) Heat seal top of Visten bag.

(22) Place cap on metal container, remove container from pit, and place in isostatic compactor.

b. Isostatic Compactor Operating Instructions

(1) Place charge in compacting vessel, close, and lock breech.

(2) Open air vent.

(3) Start prefill pump and commence filling compactor vessel.

(4) When flow from vent indicates all air has been purged from vessel, close vent and stop pump.

(5) Start supercharge pump to feed water soluble oil to inlet side of intensifier. Intensifier cannot operate due to action of low pressure interlock until a supercharge pressure of at least 80 psi is present at the inlet.

(6) Set Foxboro recording controller to the desired compaction pressure.

(7) Set the timer for the required time of compaction at pressure.

(8) Energize control panel by depressing button labeled "panel".

(9) Depress start button labeled "motor" to start Harwood pump motor. This also closes dump valve.

(10) The system is now ready for pressurizing. Depress start button labeled "intensifier" to start the pressurizing cycle.

(11) The vessel will now be pressurized. At the end of the preset timed sequence at pressure, the intensifier and pump motor are stopped automatically. The dump valve opens also. With draining of the fluid through the dump valve, the pressure will drop to near atmospheric.

(12) When the Foxboro recorder indicates that the pressure in the vessel is essentially atmospheric, stop super-charge pump.

(13) Open the air vent in the breech plug to bleed down the remaining pressure.

(14) After flow through this valve has stopped, open breech and remove charge.

c. Hydrogen Sintering Furnace Start-Up Operating Instructions

(1) Load furnace with all four cars. Front and rear loading chambers are to be rolled away from furnace.

(2) Open main water valve to the furnace. As the volume of water has been set at individual valves, opening the main water valve to the furnace is sufficient.

(3) At the transformers for Zone 3 and 4, set back the taps to No. 1 position.

(4) Open disconnect switches of elements to all four zones.

(5) At panel, turn selector switch 1-SS to "on" position. "Red" signal light "control circuit" will come on. In addition, the four "red" safety signal lights will come on:

(a) Process Nitrogen Pressure Low
(b) Hydrogen Pressure Low
(c) Chamber Safety Nitrogen Pressure Low
(d) Furnace Safety Nitrogen Pressure Low

(6) Open nitrogen process supply valve. Red signal light "Process Nitrogen Pressure Low" will go out.

(7) Turn selector switch 5-SS "Sample Furnace Purge" to "on". This starts the Ranarex motor. Set flow to Ranarex instrument at about 0.25" w.c. negative pressure on the manometer.

(8) Open valve (5) in "Nitrogen Process Purge Supply Line". Purge furnace with approximately 600 CFH of nitrogen as indicated on flow meter. When furnace is purged, Ranarex "green" signal light "Nitrogen Purge Completed" will come on. As a safety measure, continue to purge furnace for another half hour.

(9) Open the hydrogen supply line valve. "Red" signal light "Hydrogen Pressure Low" goes out. Open hydrogen safety valve (1SSV). Open nitrogen safety supply valve. "Red" signal light "Chamber Safety Nitrogen Pressure Low" and "red" signal light "Furnace Safety Pressure Low" will go out.

(10) Open manual valve (6) in hydrogen line and pass approximately 1000 CFH of hydrogen to furnace. Furnace should have a positive pressure of about 0.25" w.c. as indicated on manometer located at side of furnace. After furnace has purged for about one hour, close disconnect switches to element transformers. Be sure that transformers for Zone 3 and 4 are set bakc to Tap No. 1.

(11) For the drying out period, it is best that furnace be heated with only the first and second zones at the lower temperatures. Transfer cars every two hours in order to dry the cars out at the same timethe furnace is being dried out.

Drying Out Schedule

400°F	-	8 hours
800	-	8 hours
1200	-	8 hours
1600	-	8 hours

At this time, the elements for Zone 3 and 4 can be turned on. Caution: Raise temperature no faster than 100°F per hour.

2000°F	-	8 hours
2600	-	8 hours
3000	-	8 hours

(12) Furnace can now be set for any sintering cycle desired.

d. Hydrogen Sintering Furnace - Unloading Instructions

(1) Open valve (8) full, to pass 500 CFH of nitrogen through rear door housing.

(2) Spark ignite hydrogen at rear door. Atmosphere must burn before clamping unload chamber.

(3) Roll unload chamber into position and clamp. Open vent valve and connect union. Connect sample line to Ranarex. Open sample cock at lower rear of chamber.

(4) At panel, turn Ranarex selector switch 5SS (Sample Furnace Purge) to "off" position. Turn selector switch 2SS to "Unload Chamber". Ranarex will go up to .99+ indicating that sample is being pulled from unload chamber. This is a mixture of air, nitrogen, and water vapor.

(5) After approximately 30-60 minutes (30 minutes minimum) Ranarex should drop to .97 and "green" signal light "Purge Completed" will come on. This indicates unload chamber is purged and car can be unloaded. It may be necessary to increase flow of nitrogen to purge more rapidly.

(6) Open rear door by turning selector switch at rear of furnace to "open". When door is fully open, "green" light "Door Open" will come on.

(7) Hand crank car into unload chamber. Check travel of car from peep sight at unload chamber. When car is all the way into the unload chamber, close rear door.

(8) Continue to purge unload chamber. As hydrogen gas enters chamber when door is opened, Ranarex will drop to below .90. As the hydrogen is purged, it will recover to .94 and "green" signal light "Purge Completed" will come on. Time - 20-30 minutes (20 minutes minimum).

(9) Disconnect vent line and sample line; unclamp car and roll away from furnace. Close vent valve.

(10) Close nitrogen purge valve (8). There should be approximately 50 CFH flowing through by-pass to constantly purge discharge door chamber and prevent accumulation of hydrogen.

e. Hydrogen Sintering Furnace - Loading Instructions

(1) Open valve (7) to pass 500 CFH of nitrogen through front door housing.

(2) Spark ignite hydrogen at front door. If atmosphere does not ignite by spark, light by hand. Keep clear of front of furnace.

(3) Roll car into position and clamp. Open vent valve and connect union. Connect sample line to Ranarex. Open sample cock. Usually the sample to Ranarex is drawn from lower rear of chamber.

(4) At panel, turn Ranarex selector switch 5SS (Sample Furnace Purge) to "off" position. Turn selector switch 2SS to "Load Chamber". Ranarex will go up to .99+ indicating that sample is being taken from load chamber.

(5) After approximately 30-60 minutes, (30 minutes minimum), Ranarex should drop to .97 and "green" light "Purge Completed" will come on. This indicates load chamber is purged and car can be loaded into furnace. It may be necessary to increase flow of nitrogen to purge more rapidly.

(6) Open front door("green" light will come on when door is fully open) and hand crank car until it butts against car already in furnace. While cranking, you can observe car through peep sight on chamber. Be sure cars are butted before stopping crank. Press start button on roll drive. Rolls will carry all cars one position and stop.

(7) Close door.

(8) Continue to purge chamber. Ranarex will drop below .90 and as the nitrogen purge displaced the hydrogen fromthe furnace, it will slowly rise to .94 and "green" signal "Purge Completed" will come on. (Time - 20-30 minutes, 20 minutes minimum.)

(9) Disconnect vent line and sample line, unclamp car, and roll away from furnace. Close vent valve.

(10) Close hydrogen purge valve (7).

(11) After cars have been in the furnace for the required length of time, refer to "Unload Instructions" and continue process.

The operation of the pressing equipment has become routine. The Lindberg Sintering Furnace has continued to prove inoperable over any lengthof time and it is questionable what the final outcome of this problem will be. We are continuing to workwith the Navy and Lindberg Engineering Company to obtain a resolution of the present difficulties.

B. Forging Activities

1. Initial Forging Work

The initial development of forging techniques utilizing the impacter required a considerable amount of time indoctrinating in-room operators with basic forging techniques and principles. To demonstrate the basic techniques and principles, six inch diameter steel billets were forged to a rectangular cross-section. These finished forgings were of various sizes from 1.5 to 2" thick by 5 to 6" wide. Significant results and conclusions from these initial forging attempts were as follows:

a. The atmosphere of the facility and the mechanical principle of the impacter afforded a high degree of control during forging operations. Increased metal surface exposure and surrounding visibility, by the exclusion of metal oxides and volatile gases, were mainly attributed to the latter conclusion.

b. The tolerances of the forgings produced were within ± 1/16" in the width and thickness direction.

c. A device to indicate material deformation increments and sizes during forging operations is necessary to maintain better tolerances.

d. Preliminary billet "hook up" was accomplished by welding eyes or hooks to the ends of the billets. This method sufficed for preliminary indoctrination, but it was necessary to devise a new method when forging reactive and refractory metals.

e. The induction heating furnaces are of such a length that even heating zones exist only to the extent of approximately 30 inches. With the uneven heating zone it is difficult to finish forgings of lengths greater than 50 inches. Longer forgings will require longer furnaces which will eliminate excessive "one end at a time" forging practices.

f. Continued forging is necessary to provide the personnel with sufficient proficiency to produce high quality forgings.

g. Examination of the surfaces of the forgings produced, indicates that very little or no conditioning was necessary after forging. The surfaces of the finished forging evidenced a pronounced extenuation of local defects which usually prevail after ordinary forging operations.

These forging attempts on mild steel afforded the operators with their first experience with this type of equipment and indicated that much additional work had to be accomplished.

2. Development of Forging Practice

Several attempts were made to forge an arc-cast and machined 7.3" diameter Mo+0.5%Ti ingot. The ingot was heated to 3100°F and the first forging blow struck at approximately 2950°F using flat dies. The initial forging was started at a point 20" above the end of the ingot. The second blow was struck between 2700-2900°F and directly below the initial blow. The increments were continued to 8" from the end of the ingot. Cracking was noticed and the billet was allowed to cool to 1900°F, at which temperature the final 8" of the starting length was forged.

Final examination of the ingot after cooling revealed very slight intergranular cracking in the area where forging was done at 2950°F. The cracking was all in a direction which held very true to the periphery of the ingot. The cracking became pronouncedly worse as the temperature dropped, but was all intergranular and in a peripheral pattern. At the extreme low temperature (1900°F) severe center bursting was produced. All of the forging was done in one straight pass with no attempt to turn the billet since cracking was evident after each and every blow. Ultrasonic examination of the ingot revealed it to be sound beyond the forging areas. The ingot was cropped and the round material put aside for subsequent work.

The sound part of the ingot was again heated at 4000°F and forged at approximately 3950°F. Workability of the ingot at the latter temperature was amazingly high. Approximately 2" of reduction was experienced from one blow. Cracking of the same type as initially experienced was encountered and the forging was stopped. During this latter forging attempt, the Al_2O_3 ceramics in the induction furnace broke down because of the extreme heat, and a high percentage of oxygen was present in the furnace atmosphere. The oxygen was recognized in the form of various combustion products which produced a heavy flame emanating from the induction furnace.

It was believed that molybdenum ingots could be directly forged under the proper conditions and additional ingots were prepared for another more closely controlled attempt when the furnace was repaired. Procedures that will be used are as follows:

a. A high purity atmosphere will have to prevail to the extent of 5 ppm oxygen. The high purity will preclude any oxygen from diffusing into the grain boundaries by reason of concentration gradients.

b. The temperature will be more closely controlled and compensations for black body reflections will be made.

c. The ingot will be heated to 4100°F and forged between 3900-4100°F using slight reductions and forging around the periphery.

d. If the ingot does not respond to flat die forging, we will have to revert to a semi-closed or swage die to confine the ingot during forging.

From the preliminary forging experience with Mo+0.5%Ti there does seem to be a critical temperature range where the ingot is more susceptible to cracking. Obviously, it is not actually the temperature range but more so the strength of the large grain boundaries at the various temperatures which effects forgeability. At the high temperatures we are using and with the structures we are attempting to forge, such things as carbon could significantly affect forgeability since heavy grain boundary carbides are present in most arc-cast ingots. Results of upset testing under contract NOas 59-6142-c and high temperature forging by other investigators support these views.

To enable recording the temperature while working and control the forging in relation to minimum working temperatures, it was necessary to find the relationship between optical temperature and true temperature. This was accomplished by the use of a thermocouple in the billet and reading the temperature from the thermocouple simultaneously with the optical pyrometer.

The resulting curve from this work is shown in Figure 17.

3. Lawrence Radiation Laboratory Forging

This work for the University of California was covered under the same purchase order as the previously described rolling.

Sample #1 - EB Melted Tungsten - 3" Diameter x 4" Long

Heating No	KW	Heating Time	Starting Temp.	Finish Temp.	Size
1	140	15 min.	3210°F	3125°F	3-1/2"x3"long

This material appeared sound after two blows with the hammer; however, after removing a slice from the upset forging, it was found to be cracked.

Sample #2 - Westinghouse Arc Melted Ingot - 4-1/2" Diameter x 5"

Heating No	KW	Heating Time	Temperature Attained
1	150	20 min.	3800°F

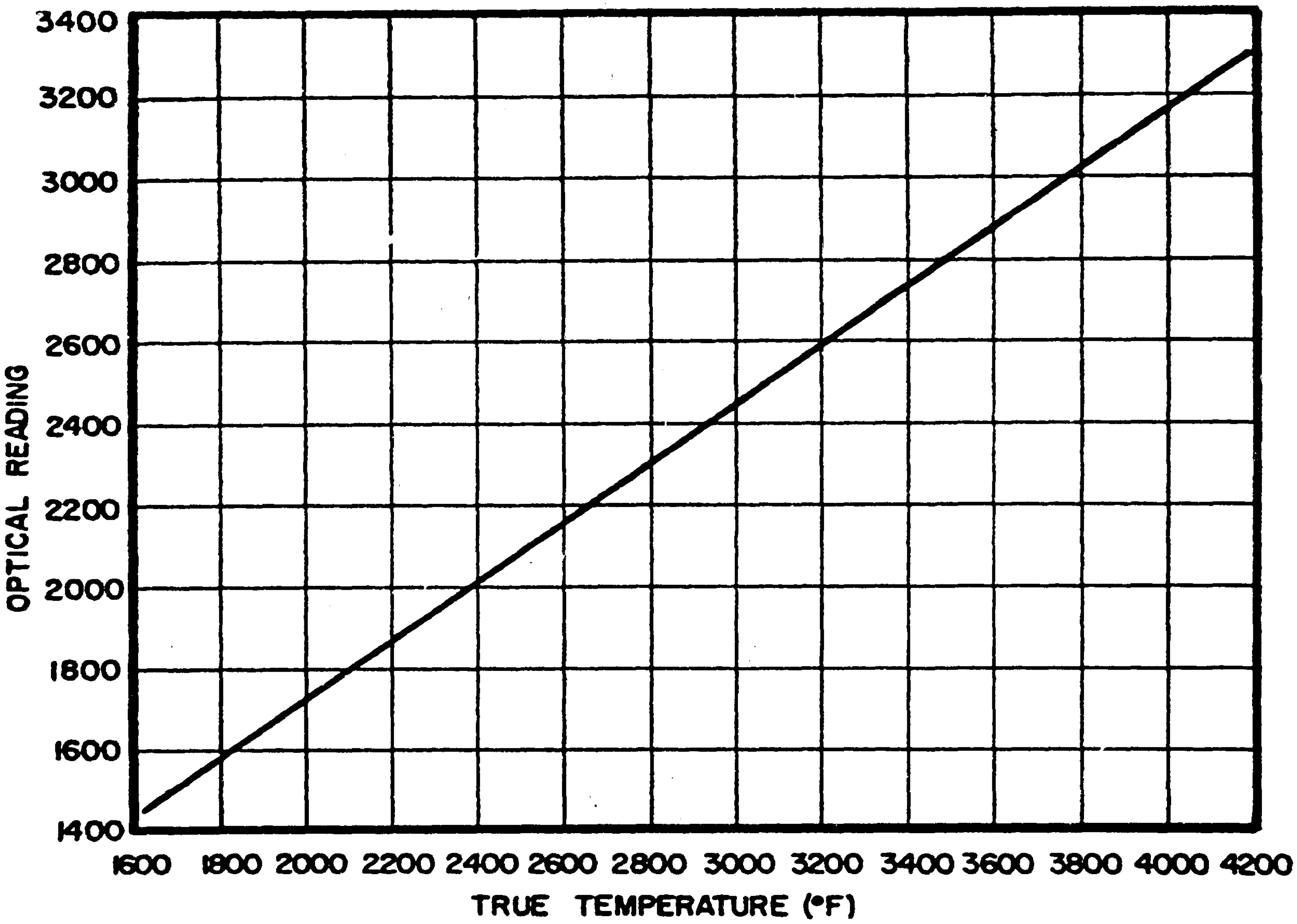


FIGURE 17

OPTICAL READING VS TRUE TEMPERATURE FOR INFAB IMPACTOR

This ingot cracked upon heating it to forging temperature. No forging was accomplished on this ingot.

Sample #3 - Universal-Cyclops Arc Melted Tungsten - 3" Diameter x 2" Long

Heating No.	KW	Heating Time	Forging Temp.	Size
1	140	13 min.	3870°F	3-5/16" Dia. x 1-1/2"

This material was struck with one blow of the impacter at the above temperature. The material showed small peripheral cracks but was completely sound otherwise.

Sample #4 - Bureau of Mines Arc Melted Tungsten - 3-1/4" Diameter x 2-3/4" Long

Heating No	KW	Heating Time	Forging Temp.	Size
1	140	5 min.	4000°F	1" thick

This material was struck twice with a starting temperature of 4000°F and finishing temperature of 3810°F. The final size of the material was 1" thick with the material showing severe edge cracking.

Sample #5 - Bureau of Mines Arc Melted Tungsten 3-1/2" Diameter x 4-1/4" Long

Heating No.	KW	Heating Time	Forging Temp.	Size
1	120	12 min.	4100°F	3-1/2" long

This material was upset forged to 3-1/4" long at a starting temperature of 4100°F and finished at 3850°F with two blows of the hammers. There was no apparent cracking of the ingot after working but the hot topped end did not upset. After working, a slice was cut off for micros and it was found that the ingot was cracked.

Sample #6 - TZC with .02% Carbon 3-1/8" Diameter x 8" Long

Heating No.	KW	Heating Time	Forging Temp. Start	Forging Temp. Finish	Size
1	180	20 min.	3500°F	3150°F	-
2	180	5	3900	3550	-

The ingot was radially forged with light hammer blows but began to crack after the first two blows. The ingot was flattened anyway but at 2" thick was so badly cracked that work was stopped.

Sample #7 - TZC with .30% Carbon - 3-1/8" Dia. x 8" Long

This sample was started the same as Sample #6 but was initially forged at 4000°F. However, the ingot began to crack immediately and forging was discontinued to recondition the ingot to permit upset forging.

Powder Metallurgy Molybdenum Upset Forging

Two pieces of as-sintered powder metallurgy molybdenum 2" diameter x 3" long were upset using one blow of the hammer on each piece.

Sample #8

This material was struck at 2300°F and this upset the material to 2-5/16" thick. The surface of the bar showed slight discoloration but exhibited no cracks or flaws.

Sample #9

This material was struck at 2700°F and had a final upset size of 2-1/8" thick. This surface was similar to the aforementioned sample.

All of this failed material has been returned to the University of California. Several experiments remain to be completed, and this is discribed below.

Sample #10 - TZC with .30% Carbon

Initial Size - 2-3/8" Dia. x 4" long to be upset forged.

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Starting	Temperature Finish
1	100	10 Min.	3500°F	3400°F	3150°F
2	100	4 Min.	3580	3460	
3	100	6 Min	3640	3560	

This sample was 1.850" thick after upset forging.

Sample #11 - Universal-Cyclops Arc-Melted Tungsten

Heat No. KC1037, Starting Size - 3" Dia. x 3" Long

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Starting	Temperature Finish
1	50 to 100	30 min.	4020°F	3700°F	Not recorded. One blow per heating.
2	150	4 min.	3960	3600	
3	150	3 min.	3960	3850	
4	150	3 min.	3970	3700	

Final Size - 1-1/2" Thick

Sample #12 - Universal-Cyclops Arc-Melted Tungsten

Heat No. KC1037, Starting Size - 3" Dia. x 3" Long

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Starting	Temperature Finish
1	20 to 150	41 min	4040°F	3780°F	Not recorded. One blow per heating.
2	150	4 min.	4040	3820	
3	150	3 min.	4020	3780	

Final Size - 1-1/2" Thick

4. Forging Cb-132

This piece of material - 2.8" dia. x 6-1/2" long - was forged to 1-1/2" thick sheet bar with a starting temperature of 3800°F and a finish temperature of 3280°F with six blows of the impacter. This material was subsequently rolled to sheet in InFab. InFab forging of this material is desirable due to the alloy's high affinity for contamination elements. No contamination occurred during high temperature forging.

5. Forging Cb+30%Zr

This material was forged to 2-3/4" thick at 2100°F and rolling was attempted as outlined in the portion of this report under "Rolling Activities". As in the case of the previous alloy, this material is also very susceptible to contamination, which makes InFab forging very desirable unless canning of the alloy is employed.

6. Forging Zirconium Ingots

This work was undertaken to determine the feasibility of forging zirconium in InFab and eliminating costly conditioning and material loss that is attendant with performing this operation in air. Six ingots were forged and the results are listed below.

Ingot #1

Heat No. SEI-1, Heating Coil Size - 12", Initial Size - 5-3/4" dia. x 3-27/32" x 23 pounds

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Starting	Temperature Finish
1	100	10 min.	1770°F	1700°F	1500°F
2	100	10 min.	1750	1700	1550

Six blows were taken on the first heating and five blows on the second. Sheet bar size - 1-3/4" thick.

Ingot #2

Heat No. SEI-2, Heating Coil Size - 12", Initial Size - 5-5/8" dia. x 3-1/2" x 20 pounds

Heating No	KW	Heating Time	Temperature Furnace	Temperature Starting	Temperature Finish
1	100	10 min.	1780°F	1700°F	1560°F

Five blows reduced this ingot to 1-3/4" thick.

Ingot #3

Heat No. SEI-3, Heating Coil Size - 12", Initial Size - 5-3/4" x 3-1/2" x 23 pounds

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish
1	100	10 min.	1760°F	1700°F	1640°F
2	100	10 min.	1770	1700	

Four blows on the first heating and five on the second heating reduced this ingot to 2" thick.

Ingot #4

Heat No. SEI-4, Heating Coil Size - 12", Initial Size - 5-15/16" x 3-7/16" x 23 pounds

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish
1	150	10 min.	1790°F	1740°F	1505°F

Seven blows reduced this ingot to 1-3/8" thick.

Ingot #5

Heat No. SEI-5, Initial Size - 5-13/16" x 6-3/16" x 39 pounds

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish
1	100	10 min.	1780°F	1720°F	1565°F
2	100	10 min.	1750	1700	

This ingot was reduced to 2" thick by seven blows on the first heating and twelve on the second one.

Ingot #6

Heat No. SEI-6, Heating Coil Size - 12", Initial Size - 5-5/8" x 5-3/16" x 36 pounds

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish
1	50	10 min.	1770°F	-	1640°F
2	50	10 min.	1700°F	-	-

Four blows on the first heating and seven on the second heating reduced this ingot to 1-3/4" thick.

impacter. This material was subsequently rolled to sheet in InFab. InFab forging of this material is desirable due to the alloy's high affinity for contamination elements. No contamination occurred during high temperature forging.

5. Forging Cb+30%Zr

This material was forged to 2-3/4" thick at 2100°F and rolling was attempted as outlined in the portion of this report under "Rolling Activities". As in the case of the previous alloy, this material is also very susceptible to contamination, which makes InFab forging very desirable unless canning of the alloy is employed.

6. Forging Zirconium Ingots

This work was undertaken to determine the feasibility of forging zirconium in InFab and eliminating costly conditioning and material loss that is attendant with performing this operation in air. Six ingots were forged and the results are listed below.

Ingot #1

Heat No. SEI-1, Heating Coil Size - 12", Initial Size - 5-3/4" dia. x 3-27/32" x 23 pounds

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Starting	Temperature Finish
1	100	10 min.	1770°F	1700°F	1500°F
2	100	10 min.	1750	1700	1550

Six blows were taken on the first heating and five blows on the second. Sheet bar size - 1-3/4" thick.

Ingot #2

Heat No. SEI-2, Heating Coil Size - 12", Initial Size - 5-5/8" dia. x 3-1/2" x 20 pounds

Heating No	KW	Heating Time	Temperature Furnace	Temperature Starting	Temperature Finish
1	100	10 min.	1780°F	1700°F	1560°F

Five blows reduced this ingot to 1-3/4" thick

Ingot #3

Heat No. SEI-3, Heating Coil Size - 12", Initial Size - 5-3/4" x 3-1/2" x 23 pounds

Heating No.	KW	Heating Time	Temperature		
			Furnace	Starting	Finish
1	100	10 min.	1760°F	1700°F	1640°F
2	100	10 min.	1770	1700	

Four blows on the first heating and five on the second heating reduced this ingot to 2" thick.

Ingot #4

Heat No. SEI-4, Heating Coil Size - 12", Initial Size - 5-15/16" x 3-7/16" x 23 pounds

Heating No.	KW	Heating Time	Temperature		
			Furnace	Starting	Finish
1	150	10 min.	1790°F	1740°F	1505°F

Seven blows reduced this ingot to 1-3/8" thick.

Ingot #5

Heat No. SEI-5, Initial Size - 5-13/16" x 6-3/16" x 39 pounds

Heating No.	KW	Heating Time	Temperature		
			Furnace	Starting	Finish
1	100	10 min.	1780°F	1720°F	1565°F
2	100	10 min.	1750	1700	

This ingot was reduced to 2" thick by seven blows on the first heating and twelve on the second one.

Ingot #6

Heat No. SEI-6, Heating Coil Size - 12", Initial Size - 5-5/8" x 5-3/16" x 36 pounds

Heating No.	KW	Heating Time	Temperature		
			Furnace	Starting	Finish
1	50	10 min.	1770°F	-	1640°F
2	50	10 min.	1700°F	-	-

Four blows on the first heating and seven on the second heating reduced this ingot to 1-3/4" thick.

7. Tantalum Alloys for National Research Corporation

During this period, forging of tantalum alloys was accomplished under contract with the National Research Corporation.

The following is the forging record for the alloy processed.

Billet #1

Starting Size - 3-1/4" dia. x 3", Heating Coil - 8", 6",
Grade - Ta+30W

Heating		Heating	Temperature			
No.	KW	Time	Furnace	Starting	Finish	Size
1	100	13 min.	3750°F	3700°F	No Hit	
2	140	7 min.	3900	3850	3735°F	2 blows(ring)
3	160	15 min.	3550	3500	3300	4 blows(ring)
4	180	7 min	3650	3600	3200	8 blows(sprue)

Finish Size - 7/8" Thick

Billet #2

Starting Size 3-1/4" dia. x 3", Heating Coil - 8", 6",
Grade Ta+30W

Heating		Heating	Temperature			
No.	KW	Time	Furnace	Starting	Finish	Size
1	110	13 Min	3320°F	3300°F	3150°F	1 blow
2	110	8 Min.	3640°F	3620°F	3100°F	2 blows
3	160	13 Min.	3530°F	3500°F	3300°F	2 blows(sprue)

Finish Size - 3/8" Thick

Billet #3

Starting Size - 3-1/4" dia. x 3", Heating Coil - 8", 6",
Grade - Ta+30W

Heating		Heating	Temperature			
No	KW	Time	Furnace	Starting	Finish	Size
1	130	12 min.	4040°F	4000°F	3870°F	1 blow (ring)
2	160	14 min.	3600	3500	3310	2 blows(sprue)
3	120	12 min.	3630	3550	3460	1 blow(sprue)
4	120	7 min	3690	3600	3200	6 blows(sprue)

Finish Size - 3/4" Thick

Billet #4

Heating No	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	100	8 min.	4040°F	4000°F		
2	145	8 min.	4035	4000		
3	200	10 min.	3650	3600		7/8" thick

Billet #5

Starting Size - 3-1/4" dia. x 3", Heating Coil - 6", 8",
Grade - Ta+15W

Heating No	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	80	19 min.	3480°F	3440°F	3200°F	
2	110	4 min.	3600	3580		
3	160	7 min.	3620	3450		3/4" thick

Billet #6

Starting Size - 3-1/4" dia. x 3-7/8", Heating Coil - 6",
Grade Ta+8W+2HF

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	70	10 min	3100°F	3020°F	2700°F	
2	100	16	3090	3000	2850	
3	100	15	3100	3000		1-1/8" thick

Billet #7

Starting Size - 3-1/4" dia. x 3-5/8", Heating Coil - 6",
Grade - Ta+8W+2Hf

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	120	6 min.	3500°F	3400°F	3070°F	1-1/4" thick

Billet #8

Starting Size - 3-1/4" dia. x 3-5/8", Heating Coil - 6",
Grade - Ta+30Cb+7-1/2V

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	50	15 min.	2850°F	2710°F	2400°F	Approx. 2" thick very badly cracked.

Billet #9

Starting Size - 3 1/4' dia. x 4", Heating Coil 6", Grade - Ta+30Cb+7-1/2V

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	60	15 min.	3090°F	3070°F		
2	80	9 min.	3050	3020		
3	70	14 min.	3080	3040	2700°F	1-1/4" thick Badly cracked.

Billet #10

Starting Size - 3-1/4" dia. x 4-15/16", Heating Coil - 6", Grade Ta+30Cb+7-1/2V

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	100	7 min.	3570°F	3450°F		
2	100	8 min.	3690°F	3550°F		Approx 1-1/2" thick - badly cracked.

The results of this work have been very conflicting. We are not sure of the absolute success of this work.

8. Under the above contract, there were two samples of vacuum arc melted and extruded tungsten forged to 1/2" thick sheet bar. This material was rolled to .040" thick sheet via InFab. The material forged well and exhibited no tendencies to crack.

Listed are the processing records for the two samples that were forged.

Sample #1

Heat No. KC1082A, Starting Size -7-1/2" dia. x 10", Heating Coil - 6", Grade - Tungsten

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	200	10 min.	3800°F	3700°F	3100°F	Edged
2	160	8	3850	3600	3000	1/2" flat

Sample #2

Heat No. KC1082C, Starting Size - 7-1/2" dia. x 14", Heating Coil - 6", Grade - Tungsten

Heating No.	KW	Heating Time	Temperature Furnace	Starting	Finish	Size
1	100	11 min.	3550°F	3200°F	2850°F	Edge Flattened
2	170	4	3450	3210	2830	
3	170	5	3525	3390	3000	
4	170	5	3050	2700	2250	
5	170	5	2790	2680	2375	1/2" Flat

9. Powder Metallurgy Tungsten Forging

During this period forging of a powder metallurgy tungsten billet was accomplished. This powder metallurgy billet was forged from 4" round x 14" long to 2-3/4" round diameter x length. The resultant forging gave approximately 75% yield of good material. The conditions for forging were as follows.

Billet #1

Heat No - Powder Metallurgy, Initial Size - 4" rd. x 14", Heating Coil - 6", Grade - Tungsten, Condition - Powder Met

Heating No.	KW	Heating Time	Furnace	Start	Finish	Size
1	250	20 min.	4100°F	4000°F	3330°F	
2	200	5 min.	4110	3975	3390	
3	240	4	4120	4005	3400	
4	240	5	3790	3670	2500	2-3/4" dia.

10. Forging W+5%Mo Alloy

During the period two pieces of 1-1/2" round extruded arc cast tungsten were forged to 1/2" thick sheet bar so they could be rolled to sheet. The material was rolled to sheet successfully outside of InFab and was taken to gages ranging from .008" to .015".

The processing records are listed below for the two pieces forged.

Billet #1

Heat No. 1127B, Initial Size - 1-1/2" rd. x 15", Heating Coil - 6", Grade - W+5%Mo, Condition - As Extruded

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Start	Temperature Finish	Size
1	160	14 min.	3300°F	3200°F	3050°F	
2	170	4	3300	3100	2800	
3	160	5		2670		
4	170	5		2600		1/2" thick

Billet #2

Heat No. 1127A, Initial Size - 1-1/2" rd. x 12", Heating Coil - 6", Grade - W+5%Mo, Condition - As Extruded

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Start	Temperature Finish	Size
1	160	13min.	3400°F	3300°F	3100°F	
2	180	3	3600	3540	2700	1/2" thick

11. Additional Forging of Cb-132 Sheet Bar

The forging of several additional pieces of Cb132 are reported below.

Sample #1

Grade - Cb-132, Initial Size - 2-7/8" rd. x 8-3/8" long, Heating Coil - 6", Condition - As Cast, Finish Size - 1-3/8" thick x length

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Start	Temperature Finish
1	200	5 min.	3600°F	3410°F	2900°F
2	200	3	No Reading	3400	-
3	200	5	"	3390	-
4	180	6	3600°F	3420	3350
5	180	4	3600	3240	No Reading

Sample #2

Section of above material - same characteristics, Starting Size - 2-7/8" round. x 3" long.

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Start	Temperature Finish
1	150	6 min.	3800°F	3420°F	3100°F
2	150	4	3600	3410	2850

Finish Size - 7/8" thick

Sample #3

Grade - Cb-132, Size - 2-7/8" rd. x 7-3/4" long

This sample was hot forged with a maximum temperature of 4000°F and a minimum forging temperature of 3450°F. The following is the data for this forging.

Heating No.	KW	Heating Time	Temperature Furnace	Temperature Starting	Temperature Finish	Remarks
1	170	7 min.	4000°F	3950°F	3500°F	2 blows edging, 1 flattening blow.
2	180	6 min.	4070°F	4000°F	3450°F	Flattened to 1-1/8" thick.

All of this material was converted to sheet and is reported in a separate section.

12. Forging Under Contract NOas 59-6142-c

The forging of both arc-cast ingots and extruded billets was accomplished under this contract. Over 75 extruded billets and 20 as-cast ingots have been forged. Comprehensive results have been reported and no attempt will be made to summarize this work under this contract as it will be covered fully under the final report of Contract NOas 59-6142-c.

There have, however, been limitations on the impacter that have been learned from this contract. Ingots no larger than 5" in diameter and extrusions no larger than 6" in diameter of molybdenum alloys can be handled in the impacter. Material of any larger size can be forged, but only to the detriment of the forging equipment.

13. Problems Still Remaining in Forging

There are some problems that continue to cause difficulty in the operation of this equipment. Some of these have been outlined previously but a review is now in order.

a. A device is still required to determine the material deformation increment and sizes during forging operations. Work with any material over a long period of time will give this information and, for molybdenum alloys, it is pretty well known. However, this information cannot be translated to other metals or alloys.

b. Billet hook-up on refractory metals was accomplished by threading and nippling for molybdenum and tungsten, and welding for tantalum and columbium. There is still room for improvement in this area in an effort to improve product yields.

c. Heating devices capable of handling longer lengths would be a definite advantage and cut down on operating costs.

d. Additional work is required in die design to allow for improvement in forged shapes whether they be sheet bar, rounds, or any other configuration.

C. Rolling Activities

1. Initial Rolling Experience

Three pieces of mild steel sheet bar, manufactured on the impacter, were cross rolled to approximately .175" x 12" x length. Normal working temperatures for mild steel (1550°F - 2250°F) were used in rolling the sheets. No attempts were made to closely control the reductions and finishing temperatures since the work was more of personnel indoctrination. Mill stalling was experienced several times and was caused by material admission at mill speeds less than full. It is definitely apparent that reductions will have to be controlled because of the mill horsepower.

The above experience provided us with the following conclusions.

a. Added material support, close to the rolls, was necessary to facilitate material entry. The latter support was provided by affording two heavy plates which set on the run out tables and can be moved to or from the mill. The plates also facilitate furnace entry.

b. The surfaces of the finished mill steel sheets were exceptionally good especially since no conditioning was performed after forging. The surfaces did possess an oxide film, but not to the extent of scaling.

c. Tolerance capabilities cannot yet be projected.

In addition to rolling the mild steel, two attempts were made to roll molybdenum. During the first attempt, a recrystallized section, 3" x 9" x 8", was rolled to .5" thick. The temperature range was between 1800°F and 2600°F and reductions were kept below 15% per pass. The finished plate evidenced a considerable amount of transverse surface cracking which is believed to be from one or all of the following reasons:

a. Failure to remove all of the fine surface cracks originally present in the piece.

b. High O_2 content in the furnace causing excessive grain boundary diffusion.

c. A reaction between molybdenum and carbon from the graphite susceptor.

d. A reaction between Mo and Fe from the rolls.

Metallographic examination of the section subsequently confirmed that the rolls, after rolling the Mo, exhibited a roughness consisting of fine cracks, pitting, and flaking caused by the reduction of a formulated alloy. The extent of the latter was not severe and another section of Mo - 1" x 4" x 8" - was rolled. The temperatures used in rolling the latter section were between 1700°F - 2300°F. After the fourth pass, the top roll cracked. The crack initiated near the neck of the roll and progressed on an angle of approximately 45°. Temperature estimation of the piece during the fourth pass was approximately 1700°F.

Examination of the piece after the roll cracked showed that only two inches (length) of approximately six inches were reduced and that the actual reduction was approximately 20%. Although the roll failure is attributed to excessive reduction of the piece, other possibilities cannot be overlooked. The other possibilities are as follows:

a. A defective roll.

b. An unbalanced roll setting. This could have been caused by a turning of the block beneath one of the screws which would not allow the roll to screw down evenly. The latter had been experienced before while running the screw down very fast.

Whatever the real cause was, definite precautionary measures were established to prevent the recurrence. The precautions consisted of the setting with the screw down counter and a visual record of how much screw is to be added after each pass. The final result was that the rolls were not suitable for the operation and this has been covered in an earlier section of this report.

A consolidation of the mill experience on the initial trials was as follows:

a. A definite problem with the alloying of rolls and rolling material exists at high temperatures.

b. A refractory metal hearth should be provided for the mill furnace to prevent intimate contact between the metal and the graphite susceptor.

c. A means of reducing the high CO_2 atmosphere in the furnace will be vital in heating reactive metals.

d. A means of expanding the cold rolls will be necessary before actual rolling.

e. More accurate settings of the screw down counter and the heating eye will be necessary to closely control temperatures and reductions.

f. A considerable amount of time will be necessary to indoctrinate the personnel with even the important basic aspects of rolling procedures.

The conclusions reached after this first rolling experience of over eighteen months ago are still pertinent today. Many of them have been resolved and a more thorough understanding and control of others is being accomplished. However, efforts must be continually expended to completely resolve all the problems as will be seen in subsequent sections of this report.

After the first rolling experience, continued control of all variables finally resolved the problem to poor roll design. The manufacturer was requested to change the roll design and switch from cast iron to forged steel rolls. Credit was issued for the old rolls and new rolls were procured and installed.

2. Development of Rolling Practice

While the mill rolls could not be used for rolling sheet, development work progressed on the rolling of round bars. Mild steel - 3" x 3" - was rolled to 1" round bar stock to obtain satisfactory product. Initial attempts required as much as four reheats to achieve the desired product, but after experience had been gained, these bars could be produced in one reheat.

As has been reviewed previously, many difficulties were encountered in the operation of the InFab rolling mill. Most serious of these was the continual breakage of flat rolls used for sheet rolling. The rolls used for bar rolling have worked quite effectively and no problems have been encountered. Additional problems were encountered with the mill tables, and these have subsequently been removed from the room. There has been some successful rolling accomplished and a description of some of the results will be covered below.

a. High Strength, Solid Solution Strengthened Columbium Alloy

The alloy used for this rolling experiment was vacuum arc melted and then quadruple electron beam melted. The resultant ingot was conditioned and then extruded to a 1.5" diameter. Conditioning of the extrusion reduced the diameter to 1.3". The extrusion was cut into two pieces and one piece was rolled bare in air and the other rolled bare in InFab.

From previous experience with canned rolling of the alloy, it was known that an extrusion could be rolled at temperatures from 1600°F to 1800°F. To minimize contamination in air, the lower temperature (1600°F) was selected for rolling. The piece was heated for twenty minutes at 1600°F in a slightly reducing atmosphere. On the second pass, a crack initiated in the nose and propagated the full length of the bar.

The second piece was rolled to 1" diameter (45% reduction) bar in the argon atmosphere of InFab. Initial rolling temperature was 1800°F with a finishing temperature of 1200°F. No difficulty was encountered, although the bar exhibited laps and seams due to inexperience of the in-room operators in bar rolling. Figure 18 shows the results of these rolling experiments.

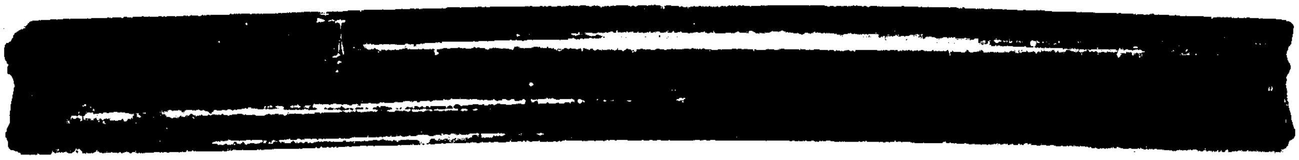
A black contaminated layer formed on the bar during heating in the graphite susceptor. The layer is attributed to fine graphite particles suspended in the furnace atmosphere by the high frequency induction field. Furthermore, if the contamination were due to oxygen impurities in the argon, the bar probably would have cracked during rolling since the use of two reheats gave sufficient time for diffusion.

b. Molybdenum + 0.5%Titanium Bar

In attempts to roll 2" diameter rounds to 1" diameter bar stock in InFab, many problems resulted. It was found that the operators could not work for sustained periods of time in the InFab suits while rolling heavy pieces by hand and, as a consequence, had to work in Scott Air-paks. It was noted that surface contamination of the bar did occur during rolling in this manner.

The InFab rolled bar had a starting temperature of 2600°F and with one reheat was taken down to 1" round

ROLLED IN INFAB—1800°F



ROLLED IN AIR — 1600°F

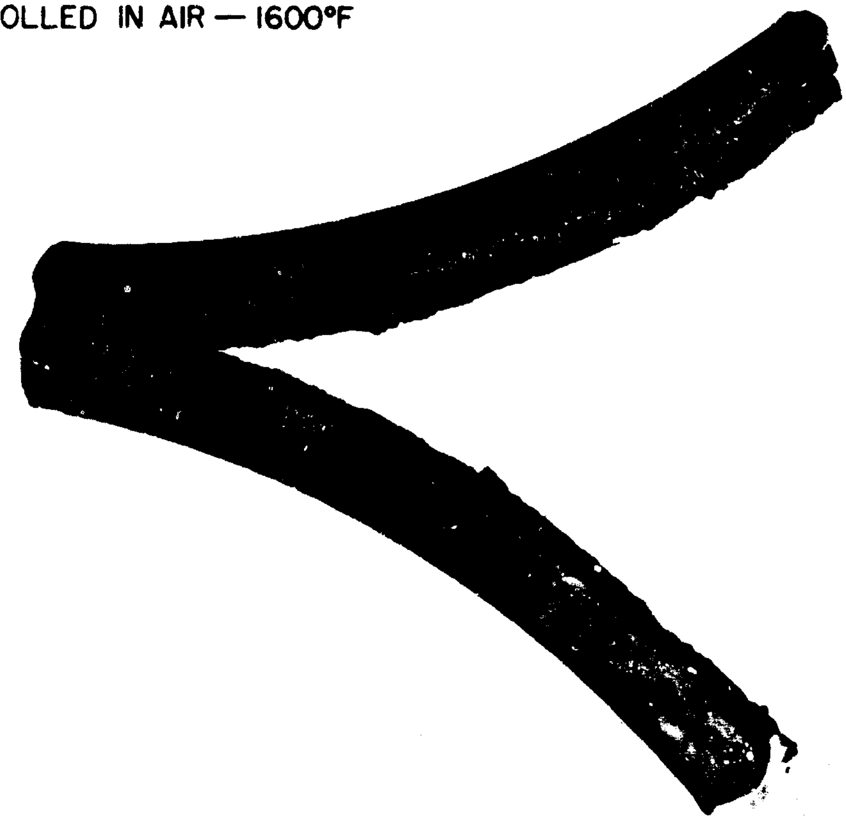


FIGURE 18

RESULTS OF ROLLING SECTIONS OF ALLOY 132 BAR IN AIR AND IN INFAB WITHOUT CANS with a finishing temperature of 1700°F. The degree of contamination was minimal and could be removed by centerless grinding .002 mils from the surface.

c. Molybdenum + 0.5% Titanium Sheet Bar

Three sections of Mo+0.5%Ti were rolled between 1800-2000°F. During the last two attempts roll failure occurred. Both times the top roll cracked on a 45° angle from the neck toward the center. During this rolling experience, we noticed on occasion that rapid use of the automatic screw down revealed a lag in the rolls to follow the screw. The latter problem is associated with the hydraulic system. The rolls are supposed to be kept against the screw and react to any change through two valves on lifters. The hydraulic system was checked and corrections were made that would allow more positive action of the roll against the screw. Subsequent analysis revealed that this problem was not related to roll failures.

The mill has a separating force of 850,000 psi and supposedly a force greater than the separating force plus a safety factor would cause roll failure in the weakest section or the middle of the roll. When the second failure occurred, we were rolling a section of Mo+0.5%Ti which was 10" wide and we were rolling from 2.350 to 2.00" in thickness. The temperature at the time was approximately 2000°F. Using a compressive yield strength of 60,500 psi for Mo+0.5%Ti at 2000°F, the calculated force on the mill was 925,000 psi. The latter figure is calculated using a 16-3/16" diameter roll. Since the force is not greatly in excess of the separating force, it is unlikely that the failure occurred because of the reduction. Subsequent evaluation by Universal-Cyclops and the roll manufacturer revealed that roll design was inadequate.

A small (4-1/2" wide) piece of sheet bar of molybdenum and one of Mo+0.5%Ti was rolled at 2000°F in the InFab enclosure. The Mo+0.5%Ti was rolled from 1.25 to .74 inches and the pure molybdenum from 1.62 to 1.0 inches. During the rolling of the pure molybdenum, the work piece curled around the mill breaking a portion of the shoe plate.

In an effort to determine the capabilities of the InFab rolling mill and gather information relative to the new roll design, the power requirements for the above operation were recorded. The power requirements for this reduction on 4-1/2" wide material are as follows:

Mo+0. 5%Ti

Starting Gauge	Final Gauge	Horsepower
1. 250	1. 125	331
1. 125	. 995	408
. 995	. 87	348
. 87	. 74	310

Molybdenum

Starting Gauge	Final Gauge	Horsepower
1. 62	1. 50	120
1. 50	1. 375	306
1. 375	1. 250	298
1. 250	1. 125	331
1. 125	1. 00	382

It is quite evident that even on narrow material with reductions of approximately 10%, an overload of as much as 100% is experienced on the mill. Power requirements were taken under no-load conditions and found to be 12. 1 horsepower. Since rolling times are quite short, the overload figures should not be taken with great alarm. The mill motors have an overload capacity of 150% for short time operation. Further, it must be remembered that under ordinary rolling conditions, several passes may be used to effect the stated reduction and this would obviously lower power requirements.

d. Molybdenum + 0. 5%Titanium Mold-Out

An attempt was made to roll several pieces of . 250 inch thick material to . 060 inch sheet at 1425°F. The pieces were rolled to intermediate gauges with the mill roll breaking during the operation. One piece reached . 190 inches and the other . 165 inch gauge before the roll broke.

3. Microstructural Examination

In an effort to ascertain if any contamination was present on molybdenum and its alloys when rolled in InFab, microstructural studies were conducted on several pieces rolled at different times.

a. 1" Thick Molybdenum Rolled In InFab

Figure 19 shows material that was rolled from 1.62" to 1.00" at a temperature of 2000°F with the reduction being effected in five passes. The figure illustrates a very low level of contamination present, even though the material was rolled using air packs. Rolling with air packs was a necessity because of difficulties encountered in attempting to roll while wearing full in-room suits.

b. 3/4" Thick Mo+0.5%Ti Rolled in InFab

This material was rolled from 1.250" to .74" at a temperature of 2000°F with the reduction being accomplished in four passes. Figure 20 does not show a normal contaminated layer and only a slight layer is discernible. The resultant structure shown in Figure 20 was apparently caused by the chilling of the surface by the cold rolls and is not considered to be contamination.

c. 1" Diameter Bar of Mo+0.5%Ti Rolled in Air

This material was rolled from 2" diameter round bar to 1" diameter round bar in air with a maximum temperature of 2200°F and a minimum temperature of 1700°F. Figure 21 shows the high degree of contamination prevalent in air rolling. The contamination is shown by the thick surface layer of unrecrystallized material.

d. 1" Diameter Bar of Mo+0.5%Ti Rolled In InFab

This material was rolled from a section of the same material rolled in air with the maximum temperature on this bar being 2600°F and the minimum 1700°F. Figure 22 shows a very shallow layer of retarded recrystallization denoting a very low level of contamination. The surface, however, does show a layer resulting from a possible chilling by the rolling mill.

Murakami's 200X

FIGURE 19

Surface of 1" Thick Molybdenum Plate Rolled in InFab
Then Flash Annealed 15 Minutes at 2300°F in Vacuum

Murakami's 200X

FIGURE 20

Surface of 3/4" Thick Mo+0.5%Ti Plate Rolled in InFab
Then Flash Annealed 15 Minutes at 2400°F in Vacuum

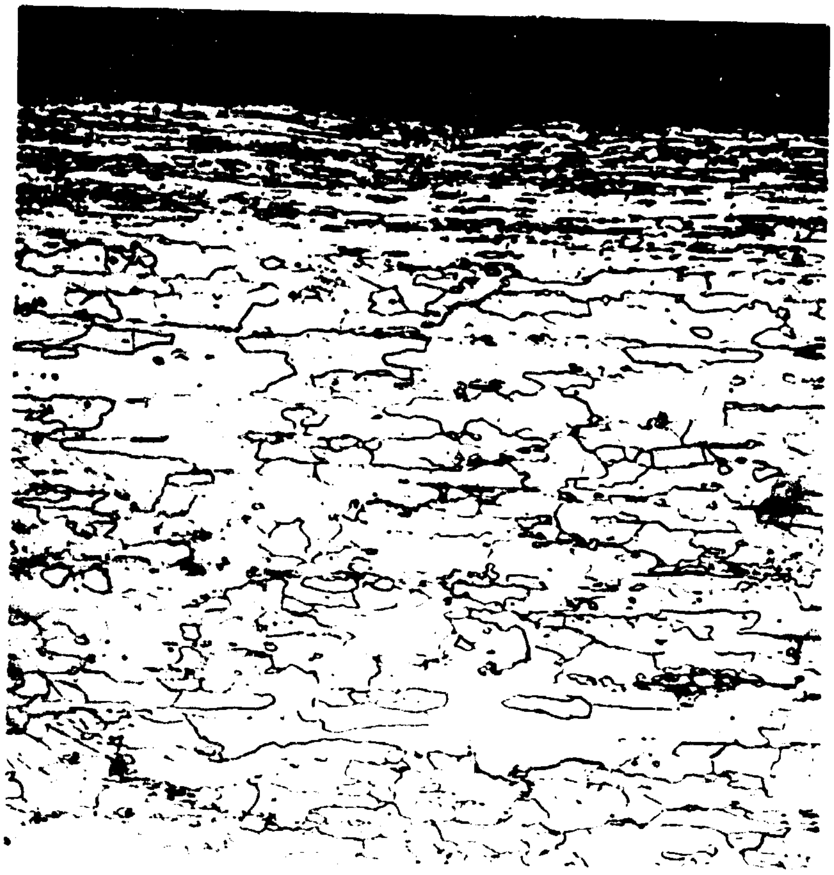


FIGURE 21

Surface of 1" Diameter Mo+0.5%Ti Bar Rolled in Air
Then Flash Annealed 15 Minutes at 2300°F in Vacuum

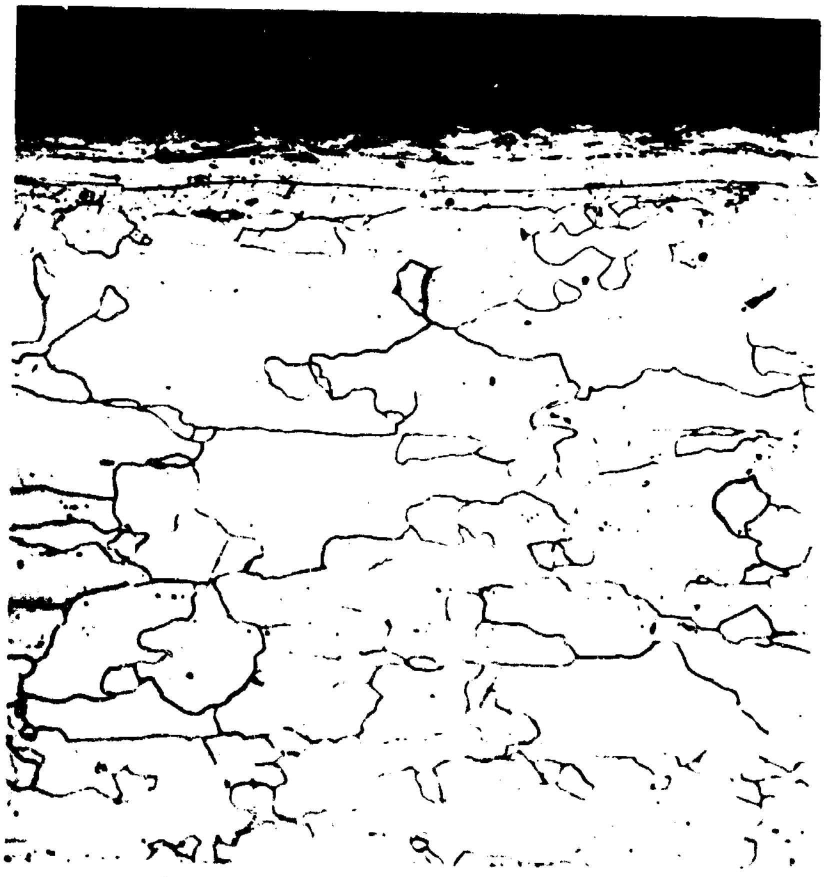


FIGURE 22

Surface of 1" Diameter Mo+0.5%Ti Bar Rolled in InFab
Then Flash Annealed 15 Minutes at 2500°F in Vacuum

This work indicates that the contamination of molybdenum and molybdenum alloys is at a minimum when rolled in InFab and, if the furnace is changed, should be completely eliminated.

4. Lawrence Radiation Laboratory Rolling

A purchase order was received from the University of California Radiation Laboratory at Livermore to convert tungsten ingots produced by various techniques. Both arc-cast and pressed and sintered material was incorporated into the program to be converted by both forging and rolling. The results of the initial rolling experiments are as follows.

Sample #1 - General Electric Hydrogen Sintered Tungsten
5" x 5-3/4" x .955"

Pass	Furnace Temp.	Total Heating Time	Start	Finish	Percent Reduction
1	2800°F	18 min.	.955	.855	10.5
2	2800	26	.855	.755	11.7
3	2800	34	.755	.655	13.2

On the third pass this material was cross rolled and came out of the mill "alligatored". At this point, rolling was discontinued.

Sample #2 - Hydrogen Sintered Tungsten
4-1/2" x 3-5/8" x 1.125"

Furnace Temperature - 2800°F

The first pass was attempted from 1.125" to 1.00" and the sheet bar cracked badly in the center. The total heating time before rolling was eighteen minutes.

Sample #3 - EB Melted Tungsten - 3" Dia. x 2" Thick

Pass	Furnace Temp.	Total Heating Time	Start	Finish	% Reduction
1	3200°F	20 min.	2.00	1.80	10.0
2	3200	26	1.80	1.60	11.1
3	3200	32	1.60	1.40	12.5

During the second pass, visible cracks began to open up perpendicular to the rolling direction and on the third pass the cracks widened to the point of making further rolling unwise.

The material listed here is a continuation of the work reported on this contract in the nineteenth monthly report. The results of this group of rolling experiments are as follows:

Sample #4 - General Electric Hydrogen Sintered Tungsten

5" x 5-3/4" x .991"

Pass No.	Temperature	Heating Time	Mill Settings	Remarks
1	3500°F	Preheat 30 Min. Heat 12 Min.	.850	
2	3500		.750	
3	3500	10 Min.	.700	Sample
			.600	Alligatored

Sample #5 - Hydrogen Sintered Tungsten

4-1/4" x 3-1/2" x 1.00"

Pass	Temp.	Heating Time	Mill Settings	Remarks
1	3500°F	Preheat 30 Min. Heat 14 Min.	.850	
2	3500		.750	Sample broke up

Sample #6 - TZC with .30% Carbon

2-3/8" dia. x 4" long

This sample was conditioned after partial forging as outlined in the report for the nineteenth month and was then upset forged as outlined in the section on forging.

The sample was then rolled at 2450°F with the starting size being 1.850" thick. However, after three passes taking .100" reductions, the material began to alligator, and after one more pass taking .150" reduction, the effect was so bad that rolling was discontinued.

Samples #7 and #8 - Universal-Cyclops Arc Melted Tungsten

Heat KC1037, Starting Size - 3" diameter x 3" Long,
Grade - Tungsten

These samples were upset to 1-1/2" by the process given in the forging section before rolling. The two samples were then rolled to 1/2" thick by identical processes and then given a one hour recrystallization treatment at 3000°F. The rolling schedule for both samples is given below.

Pass No.	Temp.	Heating Time	Mill Settings	Actual Gage
1	2818°F	Preheat 20 Min. Heat 10 Min	1. 300	
2	2820	2 min.	1. 125	
3	2835	4 min.	1 080	
4	2835	4 min.	. 90	
5	2850	4 min.	. 80	
6	2850	4 min.	. 70	
7	2820	4 min.	. 60	. 75
8	2800	4 min.	. 50	
9	2800	4 min.	. 40	
10	2800	4 min.	. 35	. 50

The above two samples will next be cross rolled at 2400°F to . 250" and finally finish rolled to . 150" at 2100°F.

Samples #9 and #10

The rolling of the . 50 inch thick piece to . 150 inch is described below.

Heat KC1037, Starting Sizes - . 500" x 4" x 6", . 500' x 4" x 3", Finish Size - . 150" x R/L x R/W

Series	Temp.	Heating Time	Mill Setting	Actual Size
1	2350°F	25 Min.	. 450	
2	2350	3	. 405	
3	2350	3	. 360	
4	2350	3	. 324	
5	2350	2	. 291	
6	2350	2	. 260	. 275
7	2350	2	. 235	

Series	Temp.	Heating Time	Mill Setting	Actual Size
8	2150°F	5 Min.	.210	
9	2150	3	.190	
10	2150	3	.170	
11	2150	3	.150	.165
12	2150	3	.130	.150

5. D-31 Alloy - Contract AF33(616)-8212

Pass	Temperature	Heating Time	Size Start	 Finish
1	1800°F	5 Min.	.490	.432
2	1815	2	.432	.388
3	1815	2	.388	.306
4	1815	2	.306	.260
5	1835	2	.260	.230
6	1835	2	.230	.195
7	1835	2	.195	.170
8	1835	2	.170	.140
9	1835	2	.140	.105
10	1835	2	.105	.090
11	1835	2	.090	.060
12	1835	2	.060	Closed
13	1835	2	Closed	Closed
14	1835	2	Closed	Closed
15	1835	2	Closed	Closed

The above is the pass schedule for two pieces of material. One piece was stopped at .070 after pass No. 12 while the other was given the additional passes and reduced to .053. The material was discolored with a thin film but did not have any contamination and was used for starting sizes on the aforementioned contract.

6. Powder Metallurgy Tungsten

In an attempt to find out if InFab could impart good surface to powder metallurgy tungsten sheet, two pieces were rolled at 2200°F to .070" gauge. The material was rolled from .095" to .070" gauge in five passes and possessed a very good surface finish.

7. Zircaloy-2 Half Ingot

This piece of material was a half ingot weighing 23 pounds. The material was to be worked at 1750°F ± 25°F down to approximately .300" thick. This work was accomplished completely in protective suits. The following is the working information for this material.

Pass Series	Temp.	Mill Setting In	Mill Setting Out	No. of Passes	Remarks
1	1730°F		2"	3	15 min. heating
2	1750	2"	1.02"	4	All reheats
3	1750	1.02"	.500	4	8 min.
4	1750	.500	.350	2	Actual Gauge-.318
5	1730	.350	.250	2	Actual Gauge-.280
6	1740	.250	.220	1	

8. Beryliium Interstitial Compounds

This material was heated slowly to 3050°F with the heating taking 17 hours to accomplish. This material was double wrapped for rolling at this temperature. It was covered with hot pressed tungsten powder and then wrapped in tantalum.

The rolling was unsuccessful on both pieces taking a 15% reduction. The one sample alligatored and the second sample separated and broke in the center.

9. Rolling Cb-132 - Columbium Base Alloy

A piece of Cb-132 was rolled from .337" x 4" x 3" to .070" thick. The following is the rolling schedule that was followed.

Pass	Temperature	Heating Time	Mill Setting	Actual Gauge
1	2410°F	6 Min	.287	
2	2410	5	.257	
3	2420	5	.227	
4	2410	5	.190	
5	2410	3	.170	
6	2400	3	.150	
7	2400	3	.120	
8	2400	2	.095	
9	2410	2	.082	
10	2410	2	.060	
11	2410	2	.042	.089
12	2410	2	.032	.078
13	2410	2	.020	.070

The results of this rolling were excellent and have shown that this alloy can be rolled in the InFab enclosure without canning.

10. Rolling D-41 Alloy

Two pieces of D-41 columbium alloy - .650" x 5-3/8" x 2-5/8" - were rolled to .225" thick and then one piece was further rolled .110". The following is the rolling schedule that was followed.

Pass	Temperature	Heating Time	Mill Setting	Actual Gauge
1-2	2200°F		.300-.240	
3	± 25°F	3 Min.	.200	.285
4		3 Min.	.160	
5		3 Min.	.120	.205
6		3 Min.	.100	
7		3 Min.	.080	
8		3 Min.	.060	.150
9		3 Min.	.040	
10		3 Min.	.020	.120
11		3 Min.	.010	.110

The results of this rolling were also successful when rolled within the InFab enclosure without canning.

11. Rolling F-48

Two pieces of F-48 were rolled to .250" thick from (1) 4-3/8" x 3-1/2" x .650" and (2) 5-5/16 x 2-5/8" x .656". The rolling schedule is listed below.

Pass	Temperature	Heating Time	Mill Setting	Rolling Direction
1	2200°F	6 Min.	.580	Longitudinal
2	2200	6	.510	Cross
3	2200	6	.450	Cross
4	2200	6	.390	Cross
5	2200	6	.330	Cross
6	2200	4	.275	Cross
7	2200	7	.230	Cross

Mill Speed - 70 RPM, Actual Finish Gauge - .250"

No difficulties were encountered and on the basis of these results, it appears that any columbium alloy can be rolled in InFab without the necessity of canning. From the results to date, it appears that considerable saving of material could be realized if columbium alloys were processed in InFab.

12. Rolling Cb+30%Zr

Two pieces of this material as-forged in InFab were rolled at 2100°F. After one pass on each sample, severe cracking occurred and rolling had to be stopped. The cracking appeared to be due to improper hot topping of the ingots during melting and a resultant crack being present and propagating. It is anticipated that further work along these lines will be performed in the future.

13. Additional Rolling Cb-132 - Columbium Base Alloy

This material as-forged at 1-1/2" thick is shown in Figure 23, before any conditioning was effected on the sheet bar. The sheet bar was then rolled as follows:

Pass Series	Temperature	Heating Time	Mill Settings		Actual Gauge
1	2700°F	15 Min.	1.40	1.20	
			1.30		
2	2700	4	1.10	.80	
			.90		
3	2700	4	.70		
			.60		
4	2705	3	.50	.40	
5	2710	3	.30		.527
6	2700	3	.20		
7	2700	3	.10		.362
8	2700	2	.000		.337

The material at this point was very sound and showed only minor edge checking. The material had been wrapped in molybdenum sheet to protect it from the carbon in the furnace during high temperature rolling, and rolling was effected with this molybdenum sheet lying on the piece. The plate showing the minor edge checking and the outline of the cover plate are shown in Figure 24. A sample of the hot rolled plate was polished and etched and Figure 25 shows the as-rolled structure at .337" gauge.

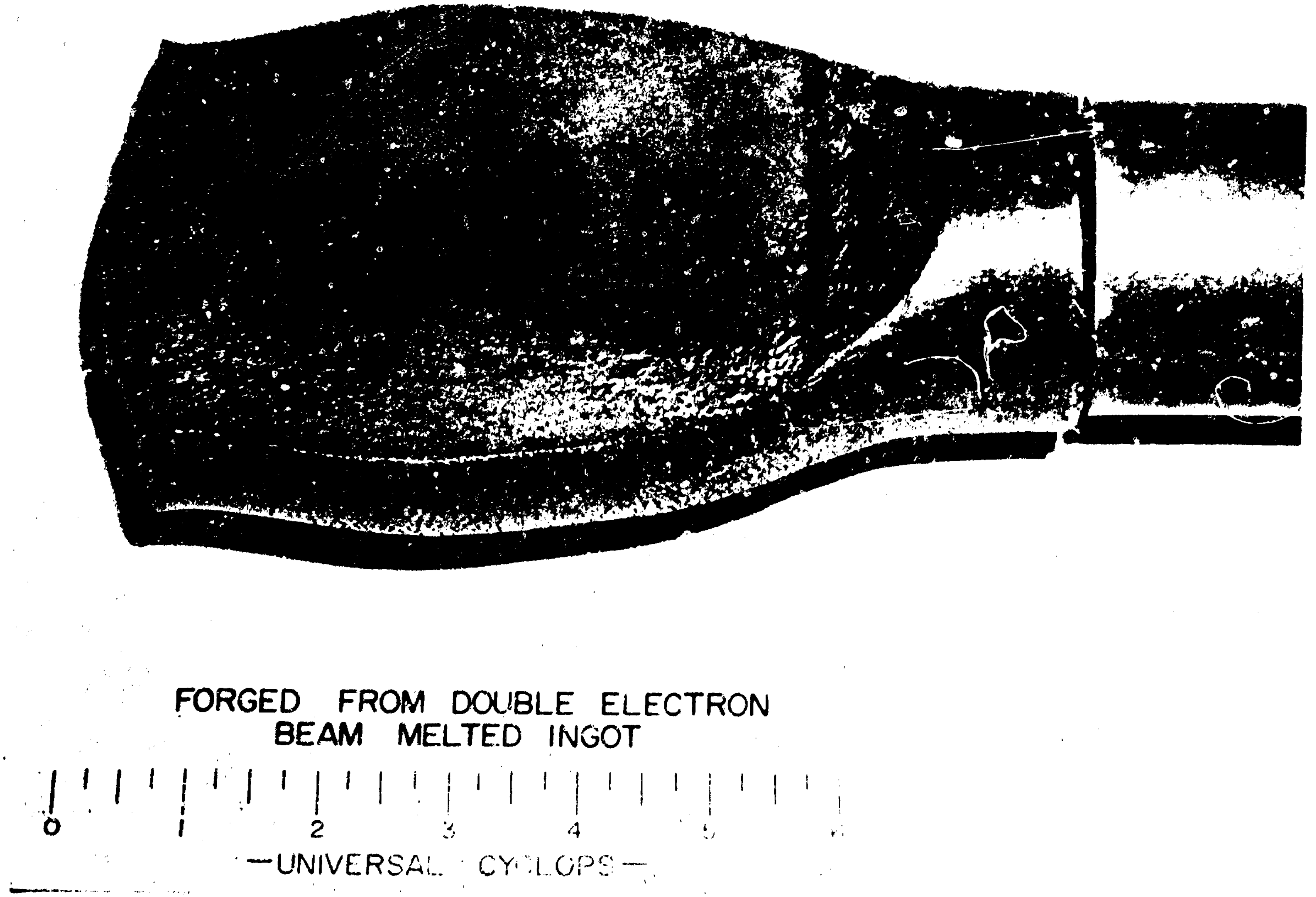


FIGURE 23
Impact Forged Sheet Bar of Cb-132 -

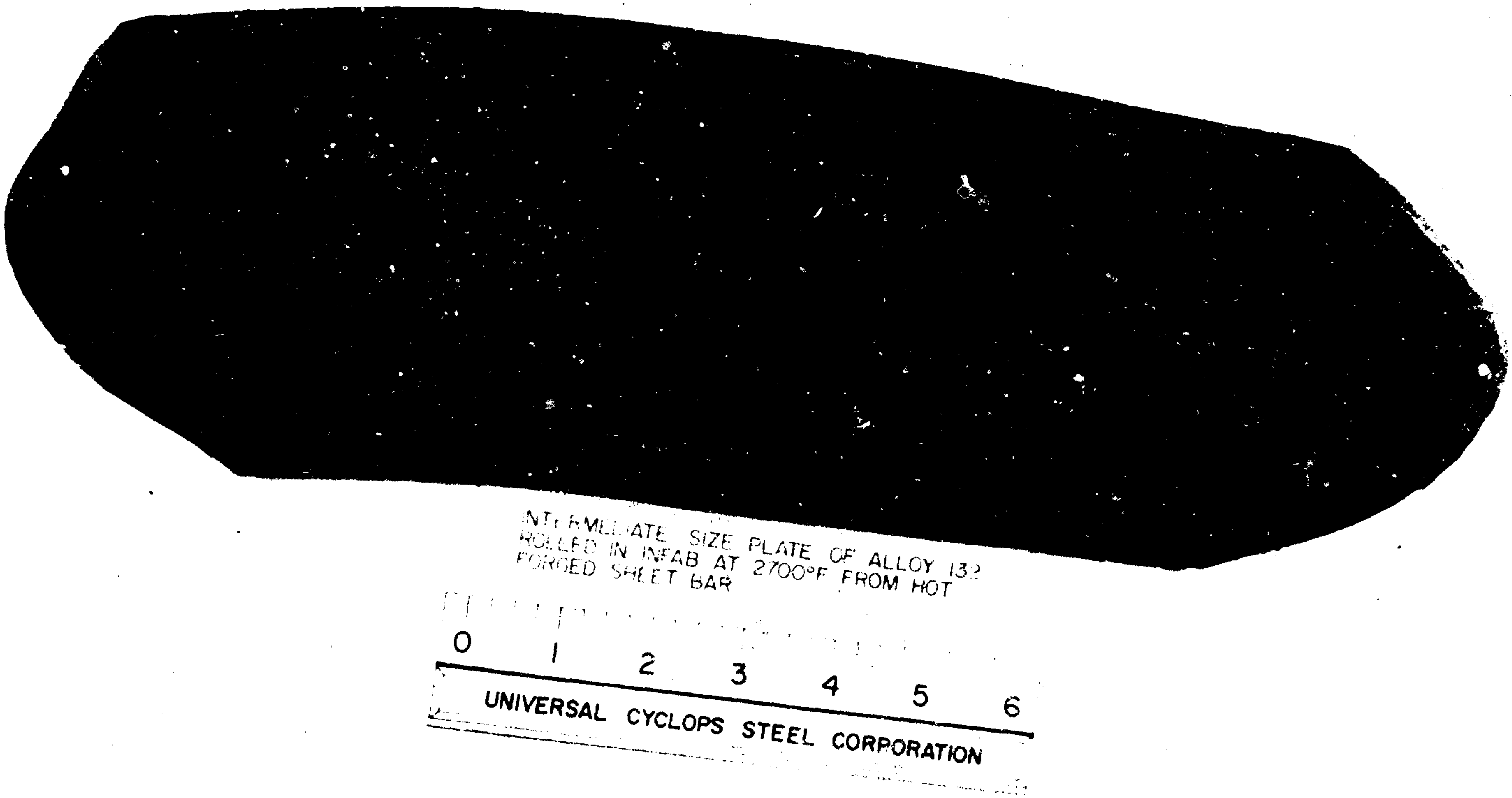


FIGURE 24

Rolled Plate of Cb-132 -
.337" x 15" x 5" Showing Minor Edge Checking

R10386 Cb-132 100X

FIGURE 25

As-Rolled .337" Gauge Sheet Microstructure

In addition to the rolling to .070" sheet at 2400°F as reported above, another piece from the same forged sheet bar was rolled to .070" sheet at 1800°F. The rolling schedule for this piece is given below.

Pass	Temperature.	Heating Time	Mill Setting	Actual Gauge
1	1800°F	6 Min.	.287	
2	1800	5	.257	
3	1815	5	.227	
4	1810	5	.190	
5	1800	3	.170	
6	1800	3	.150	
7	1805	3	.120	
8	1800	3	.095	
9	1800	2	.082	
10	1800	2	.060	
11	1800	2	.042	
12	1800	2	.032	
13	1800	2	.020	.085
14	1800	2	.010	.076
15	1800	2	000	.070

The results of this rolling were excellent and show that this alloy can be rolled in InFab without canning.

14. Rolling F-48 Alloy

Four samples of F-48 were rolled at 2000°F with no covering in InFab. The following is the data collected from these four samples.

Sample #1

Starting Size - .240" x 5-3/4" x 5", Finish Size - .074"

Pass	Temperature	Heating Time	Mill Setting	Actual Gauge
1	2000°F	7 Min.	.165	
2	2000		.150	
3	2000	3	.130	
4	2000		.110	
5	2000	2	.090	
6	2000	2	.070	
7	2000	2	.050	
8	2000	2	.035	.097
9	2000	2	.035	.082
10	2000	2	.020	.081
11	2000	2	.010	.076
12	2000	2	.000	.074

Sample #2

Starting Size - .245" x 7-1/4" x 4-1/2", Finish Size - .054" Gauge

Pass	Temperature	Heating Time	Mill Setting	Actual Gauge
1	2000°F	4 Min.	.160	
2	2000		.140	
3	2000	3	.115	
4	2000	3	.095	
5	2000	2	.075	
6	2000	2	.055	
7	2000	2	.035	
8	2000	2	.020	
9	2000	2	.000	.086
10	2000	2	.000	
11	2000	2	.000	.071
12	2000	2	.000	
13	2000	2	.000	.062
14	2000	2	.000	
15	2000	2	.000	.054

Samples #3 & #4

Starting Size - .250" x 6" x 3", Finish Size - .105"

Pass	Temperature	Heating Time	Mill Setting	Actual Gauge
1	2000°F	5 Min.	.160	
2	2000		.140	
3	2000	2	.115	
4	2000	2	.095	
5	2000	2	.075	
6	2000	2	.055	.105

Both samples were processed identically to .105" gauge. This alloy rolled excellently without any canning and demonstrates the usefulness of InFab rolling of columbium alloys.

15. Rolling As-Forged Zirconium Sheet Bar

The material for this rolling was direct forged from as-cast and conditioned ingots. The forging of this material is reported in another section of this report. The as-rolled material exhibited surface discoloration but this is believed to be only a very thin surface layer and the material is not believed to be contaminated as air rolled material would be. The following is the rolling schedule for the six samples processed.

Sample #1

Heat SEI-1, Starting Size - 1-3/4" thick, Finish Size - .282" thick, Grade - Zirconium

Pass Series	Temperature	Heating Time	Mill Settings			Actual Gauge
1	1750°F	15 min.	1.60	1.20	.800	
			1.40	1.00		
2	1750	3	.650	.400	.278	
			.500	.350	.250	.282

Sample #2

Heat SEI-2, Starting Size - 1-3/4" Thick, Finish Size - .261" Thick, Grade - Zirconium

Pass Series	Temperature	Heating Time	Mill Settings			Actual Gauge
1	1750°F	15 Min.	1.500	1.100	.700	
			1.300	.900	.500	
					.350	
2	1750	4	.300	.250		
			.250			.261

Sample #3

Heat SEI-3, Starting Size - 2" Thick, Finish Size - .290" Thick, Grade - Zirconium

Pass Series	Temperature	Heating Time	Mill Settings			Actual Gauge
1	1750°F	15 Min.	1.80	1.40	1.00	
			1.60	1.20	0.80	
2	1750	4	.650	.400	.278	
			.500	.350	.245	.290

Sample #4

Heat No. SEI-4, Starting Size - 1-3/8" Thick, Finish Size - .289" Thick, Grade - Zirconium

Pass Series	Temperature	Heating Time	Mill Settings	Actual Gauge
1	1750°F	15 Min	1.15 .800 .500	
			1.00 .650	
2	1750	3 Min	.350 .270	
			.270	.289

Sample #5

Heat SEI-5, Starting Size - 2" Thick, Finish Size - .281" Thick, Grade - Zirconium

Pass Series	Temperature	Heating Time	Mill Settings	Actual Gauge
1	1750°F	20 Min.	1.80 1.40 1.00	
			1.60 1.20	
2	1750	4	.800 .500 .350	
			.650 .400	
3	1750	5	.278 .265	.281

Sample #6

Heat SEI-6, Starting Size - 1-3/4" Thick, Finish Size - .289" Thick, Grade - Zirconium

Pass Series	Temperature	Heating Time	Mill Settings	Actual Gauge
1	1750°F	15 Min.	1.60 1.20	
			1.40 1.00	
2	1750	3 Min.	.800 .500	
			.600 .400	
3	1750	3 Min.	.350 .268	
			.280	.289

16. Rolling for Climax Molybdenum Company

During this period, rolling was accomplished for the Climax Molybdenum Company. These samples were of various compositions and rolling was accomplished according to Climax specifications. The following is all processing data for the material.

Sample #1

Heat 4052-N, Starting Size - .872" x 7" x 2",
Grade - Mo+25W+1Zr

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2600°F	34 Min	.640		
2	2600	6	.556		
3	2600	5	.393		
4	2600	5	.276		
5	2600	5	.194		
6	2600	4	.137		
7	2600	6	.096	8-5/8" wide	
8	2200	12	.064		
9	2200	4	.030		
10	2200	2	.010		
11	2200	2	.185	.129	Rolled in pack. Trimmed material caused breakage.

Sample #2

Heat 4052, Starting Size - .875" x 7" x 2", Grade-Mo+25W+1Zr

Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2600°F	33 Min.	.640		
2	2600	5	.558		
3	2600	5	.393		
4	2600	5	.276		
5	2600	5	.194		
6	2600	4	.137		
7	2600	5	.096		
8	2200	10	.064		
9	2200	4	.030		
10	2200		185	.129	Trimming sample caused breakage.

Sample #3

Heat 4051-2P, Starting Size .883" x 7" x 2", Present Size - .053 x RW x RL

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2388°F	13 Min.	.614	.883	
2	2385	5	.430		
3	2400	45	.301		
4	2400	5	.210		
5	2400	4	.147		
6	2400	3	.103	8-1/2"	Rolled to width
7	1500	11	.072		
8	1500	5	.040		
9	1500	6	.015		
10	1500	3	.000	.104	
11	1500	7	.140		Packed
12	1500	3	.100		
13	1512	7	.072		
14	1510	4	.040		
15	1510	4	.015	.062	
16	1510	5	.000	.053	

Sample #4

Heat No. 4051-2N, Starting Size - .876" x 7" x 2", Present Size - .052 x RL x RW

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2388°F	14 Min.	.614	.876	
2	2385	6	.430		
3	2400	44	.301		
4	2400	5	.210		
5	2400	4	.147		
6	2400	4	.103	8"	Rolled to width.
7	1500	9	.072		
8	1500	5	.040		
9	1500	7	.015		
10	1500	3	.000	.100	
11	1500	7	.140		Packed
12	1500	3	.100		
13	1512	7	.072		
14	1510	4	.040		
15	1510	4	.015	.057	
16	1510	5	.000	.052	

Sample #5

Heat No. 4053-4N, Starting Size - .890" x 7" x 2", Present Size - .052" x RL x RW, Grade - TZC

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2800°F	14 Min.	.646	.890	
2	2800	7	.558		
3	2810	5	.393		
4	2820	10	.276		
5	2820	7	.194		
6	2806	5	.137		
7	2500	13	.096		
8	2617	28	.064	8" wide	Rolled to width
9	2520	4	.030		
10	2525	4	.010		
11	2470	4	.000		
12	2396	11	.136		Packed
13	2400	4	.096		
14	2400	4	.068	.077	
15	2400	5	.040	.066	
16	2400	5	.015	.057	
17	2400	4	.000	.052	

Sample #6

Heat No. 4056-5, Starting Size - .576" x 7" x 2", Present Size - .050" x RL x RW

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2400°F	44 Min.	.430	.576	
2	2400	5	.301		
3	2400	4	.210		
4	2400	4	.147		
5	2405	4	.103		
6	2405	4	.072		
7	2406	5	.040		Cracked
8	2196	10	.194		Pack Size - .280"
9	2201	3	.137		
10	2200	3	.096		
11	2400	4	.103		
12	2400	3	.072		
13	2400	4	.040	.050	

Sample #7

Heat No. 4053-4P, Starting Size - .895" x 7" x 2", Present Size - .052" x RL x RW, Grade - TZC

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2800°F	15 Min.	.640	.895	
2	2800	5	.558		
3	2810	5	.393		
4	2820	10	.276		
5	2806	4	.194		
6	2800	5	.137	8" wide	Rolled to width.
7	2500	14	.096		
8	2517	28	.064		
9	2520	4	.030		
10	2525	5	.010		
11	2470	4	.000		
12	2396	11	.136		Pack
13	2400	4	.096		
14	2400	4	.068	.077	
15	2400	5	.040	.066	
16	2400	5	.015	.058	
17	2400	4	.000	.052	

Piece #8

Heat No. 4054-5P, Starting Size - .890" x 7" x 2",
Grade - 97W+3Mo

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2800°F	13 Min	.640	.890	
2	2800	5	.558		
3	2810	5	.393		
4	2820	10	.276		
5	2820	6	.194	6-3/4" wide	
6	2806	5	.137	7" wide	Rolled to width
7	2800	8	.096		
8	2500	10	.054		Cracked-stopped rolling.

Piece #9

Heat No. 4056-4N, Starting Size - .935" x 7" x 2",
Grade - 50Mo+50W

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2800°F	15 Min.	.750		Split Up

Piece #10

Heat No. 4056-4P, Starting Size - .930" x 7" x 2", Grade - 50Mo+50W

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2800°F	17 Min.	.750		Split Up

Piece #11

Heat No. 4052-1, Starting Size - .930" x 7" x 2", Grade -

Pass Series	Temp.	Heating Time	Mill Settings	Actual Size	Remarks
1	2600°F	19 Min.	.393		
2	2605	4	.276		
3	2610	3	.194		
4	2609	3	.137	4" wide	
5	2605	3	.096	5-1/2" wide	
6	2600	4	.070	7x6x.173"	Rolled to width
7	2200	7	.210		Packed
8	2200	5	.147		
9	2200	3	.103	.110"	
10	2205	3	.065		
11	2207	4	.040		
12	2210	4	.010		
13	2210	4	.000	.051"	

17. Tungsten Rolling Contract AF33(600)-41917

Sample #1

Heat - KC1083, Starting Size - .556" x 9" x 3"

Pass	Temp.	Heating Time	Mill Settings	Actual Gauge	
1	2400°F	8 Min.	.475	--	
2	2400	4	.400, .325	--	
3	2400	5	.250, .190	.275	Stress Relieved at 2800°F
4	2100	5	.165, .115	--	
5	2100	4	.060	.155	
6	2100	3	.140		
7	2100	3	.125, .110	132	
8	2100	3	.090 .070	.119	

Pass	Temp.	Heating Time	Mill Settings	Actual Gauge
9	2100°F	3 Min.	.050	.107
10	2100	3	.048	.100
11	1800	10	.040	
12	1800	3	.015, .000	
			.000	.085
13	1800	10	.080, .025	
			.050, .000	Pack Rolling
14	1800	5	.000, .000	.038

Final Gauge - .038"

Material cracked after the last pass due to partial bonding to stainless cover plates and the subsequent stresses imposed by the differential shrinkage.

Sample #2

Heat No. KC1082, Starting Size .475" x 6-3/4" x 3-1/4"

Pass	Temp.	Heating Time	Mill Settings	Actual Gauge
1	2400°F	6 Min.	.400, .325	
2	2400	3	.250, .190	.281 Stress Relieved
3	2100	10	.250	1 Hour at 2800°F
4	2100	4	.225	
5	2100	3	.200	
6	2100	3	.180	
7	2100	3	.160	
8	2100	5	.140	
9	2100	3	.120	.157
10	2100	3	.100	
11	2100	2	.080	.130
12	2100	3	.060	.118
13	2100	3	.045	.106
14	2100	3	.038	.096
15	1800	7	.040, .015	Pack Rolling .072
16	1800	3	.000, .000	
17	1800	9	.080, .025	
			.050, .000	Repack
18	1800	4	.000	.049

Final Gauge - .049

It was necessary to repack the material at .072 in. thick due to bonding problems with the stainless steel pack material.

Sample #3

Heat No. KC1082, Starting Size - .430" x 6-3/4" x 3-1/4"

Pass	Temp.	Heating Time	Mill Settings	Actual Gauge
1	3000°F	9 Min.	.300, .225	
2	3000	4	.150, .090	.189 Bonding to roll.
3	2100	10	.100	
4	2100	3	.080	
5	2100	3	.060	.107
6	2100	3	.058	.097
7	1800	7	.040, .015	
8	1800	3	.000, .000	.075
9	1700	6	.080, .050	Pack Rolling
			.025	
10	1700	3	.000, .000	.041

Final Gauge - .041

Severe bonding took place when packing in stainless and rolling at 1800°F.

Sample #4

Heat No. KC1082, Starting Size - .385" x 6-3/4" x 3-1/4"

Pass	Temp.	Heating Time	Mill Settings	Actual Gauge
1	3000°F	8 Min.	.275, .200	
2	3000	2	.150, .190	.183 Bonding to roll.
3	2100	10	.100	
4	2100	3	.080	
5	2100	3	.060	.123
6	2100	3	.040	
7	2100	3	.038	.100
8	1800	7	.040, .015	Cross Rolled
9	1700	3	.000, .000	.075
10	1700	6	.080, .050	
			.025, .000	Pack Rolled
11	1700	3	.000	.036

Finish Gauge - .036"

Material was packed ween stainless steel at 1800°F and severe bonding occurred; therefore, the temperature was subsequently lowered to 1700°F but again some bonding occurred. This problem is undoubtedly associated with the clean surfaces of the material being pack rolled. It will be necessary in future rolling of this material to incorporate the use of a barrier layer such as chrome oxide to minimize roll bonding.

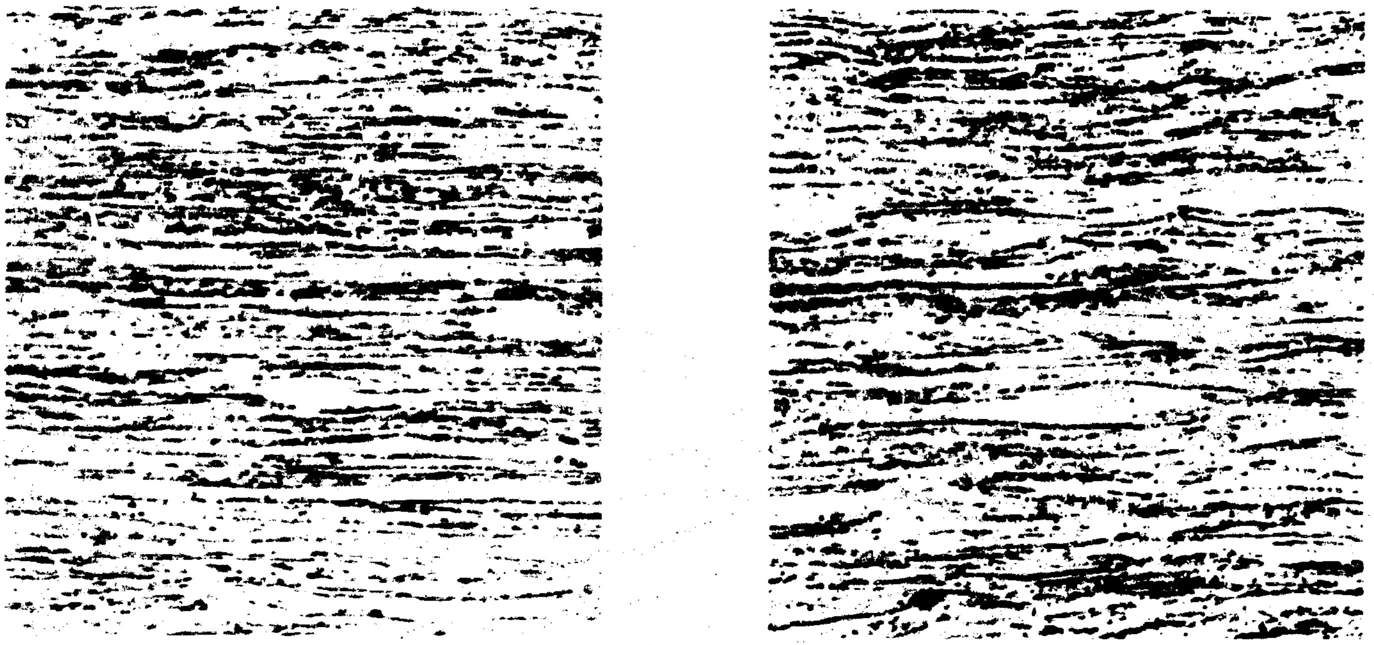
The surface of the tungsten in the areas where no bonding occurred was considerably better than that produced in air. Again, this improvement can be related to the absence of oxides, in the InFab enclosure. There was a discoloration of the tungsten sheets, but metallographic examination revealed no contamination.

The as-rolled material was evaluated and the evaluation consisted of metallographic observation of the as-rolled structures, a determination of the response to heat treatment as determined by metallographic studies and hardness data on the initiation and acceleration of recrystallization, and bend transition determinations.

Hot-cold rolling versus hot rolling and straight versus cross rolling result in significant differences in grain structure as shown in Figures 26 and 27. In Figure 26 the hot-cold rolled structures of straight and cross-rolled material are compared. Note that the grain structure of the cross-rolled material is wavy and coarser than the straight rolled. Also, the grain boundaries of the longitudinal hot-rolled material are not continuous, which is characteristic of a transverse structure in straight rolled material. The transverse and longitudinal structures of the cross-rolled material are consequently shown to be quite similar. The longitudinal hot-cold rolled structure shown is similar to that obtained on conventional rolling utilizing the same general rolling temperatures and reductions.

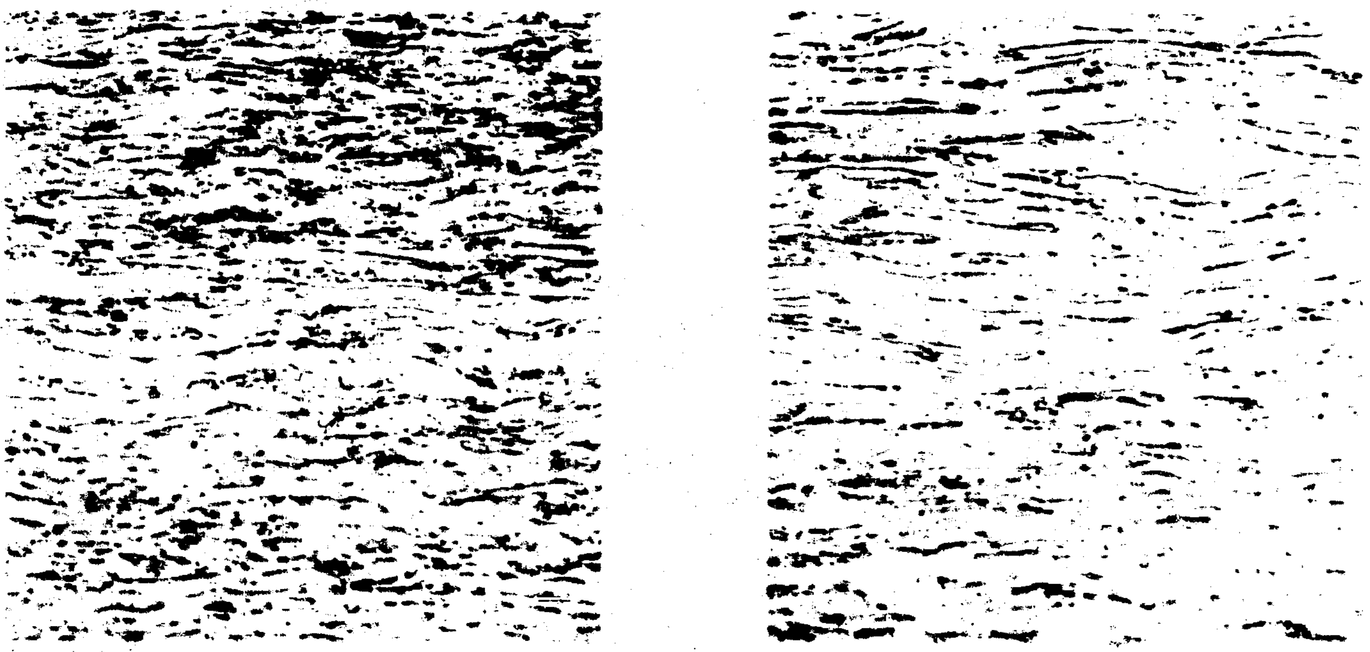
Figure 27 shows the as-rolled structures obtained with initial hot rolling and subsequent hot-cold rolling. It is immediately evident that the hot rolling has resulted in a very coarse grain structure. Except for the coarser structure, the effects of cross rolling are shown to be the same as that previously discussed for hot-cold rolled material.

Samples from all sheets were annealed for one hour at temperatures from 1800°F to 2400°F at 100°F increments. The



Longitudinal Straight Rolled R11310

Longitudinal Cross Rolled R11313



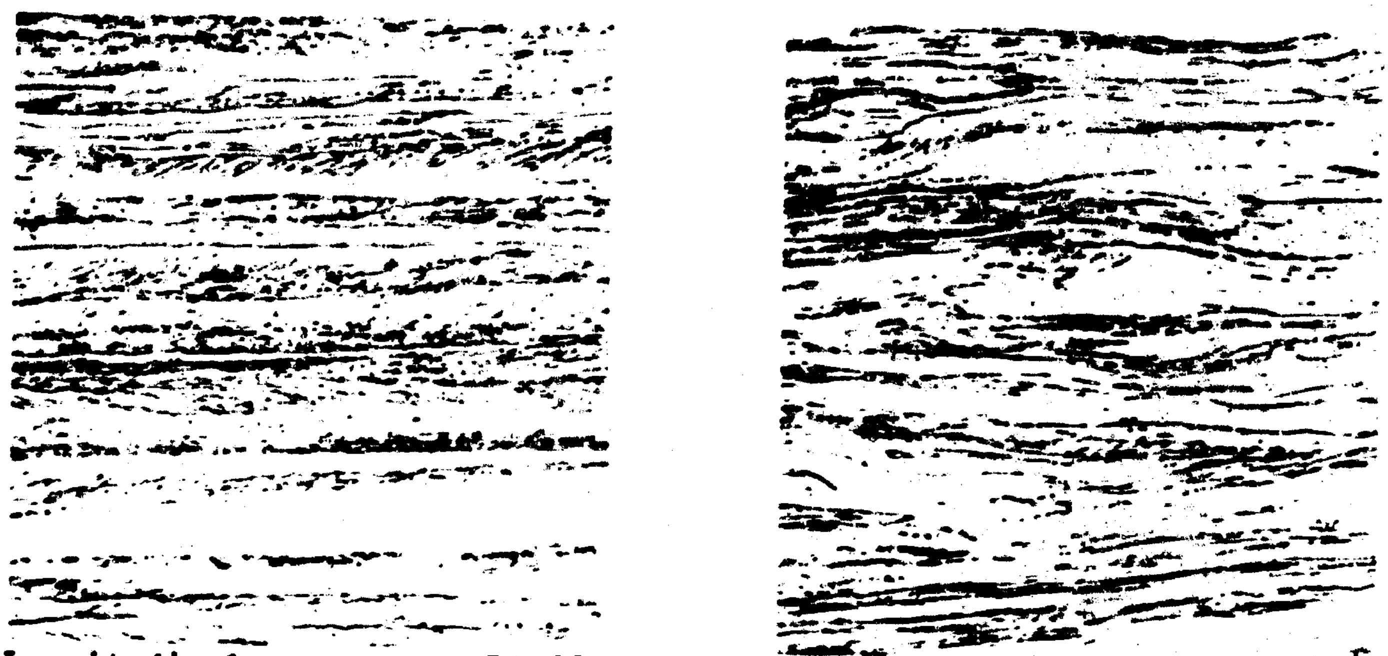
Transverse Straight Rolled R11311

Transverse Cross Rolled R11312

Initial Rolling Temperature - 2400°F
Final Rolling Temperature - 1700-1800°F
Constant Gage - 0.040"
Magnification - 200X

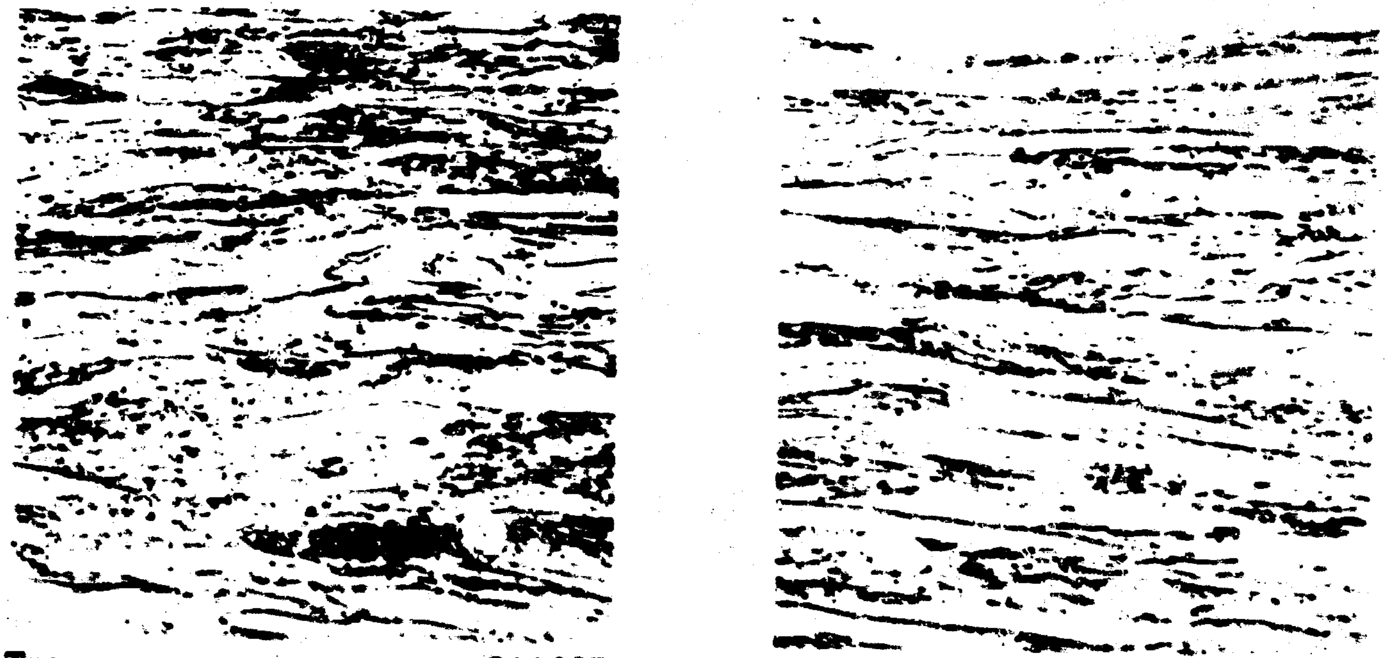
FIGURE 26

AS-ROLLED INFAB HOT-COLD ROLLED MICROSTRUCTURE
TUNGSTEN ROLLED FROM ARC CAST, EXTRUDED, AND HOT FORGED
KC1032



Longitudinal Straight Rolled R11306

Longitudinal Cross Rolled R11308



Transverse Straight Rolled R11307

Transverse Cross Rolled R11309

Initial Rolling Temperature - 3000°F
Final Rolling Temperature - 1700°F
Constant Gage - 0.040"
Magnification - 200X

FIGURE 27

AS ROLLED INFAB HOT ROLLED MICROSTRUCTURES
TUNGSTEN ROLLED FROM ARC CAST, EXTRUDED, AND HOT FORGED
KC1082

response to these heat treatments as measured by hardness is shown in Figure 28. As shown, there is little effect on the hardness drop due to the different rolling techniques. The two hot-cold rolled sheets do show a sudden drop above 2200°F. However, they converge again at 2400°F. This early drop off would indicate a slightly lower recrystallization temperature for the hot-cold rolled material as would be expected. A plot of conventionally rolled material is also shown. No explanation can be given as to why the hardness is lower for this material.

The recrystallization behavior, as determined by microstructural observation, is similar to that previously determined for material conventionally rolled. Figure 29 shows the structure obtained on hot and hot-cold straight rolled material after a one hour 2400°F stress relief as compared to conventionally rolled material after the same heat treatment. The grain size and degree of recrystallization are relatively equivalent for the InFab versus conventional rolling. The grain size of the hot worked material is slightly larger in both cases although the difference is not as significant as that shown for the as-rolled structures in Figures 26 and 27.

Bend transition was determined for all sheets after 1800°F and 1900°F stress relief anneals. This data is contained in Table VI. The data again points out that hot rolling is detrimental to bend properties. Cross rolling is shown to have no effect on reducing the transverse bend transition temperature. More extensive rolling and evaluation should disprove this statement based on extensive work with other materials. The lowest transition temperature compares favorably with that obtained on material rolled an equivalent amount (85%) in air. It must be pointed out that this material did not have as much reduction from the last recrystallization anneal as that shown to be optimum (92%) for air rolled material. Increased reductions on material rolled in InFab would, therefore, be expected to show improved bend transition temperatures.

18. Sheet Bar Rolled at 3400°F

A sheet bar rolled at 3400°F exhibited large surface cracks and was considered unusable. Figure 30 shows the as-rolled material with the large surface cracks. This is a continuing

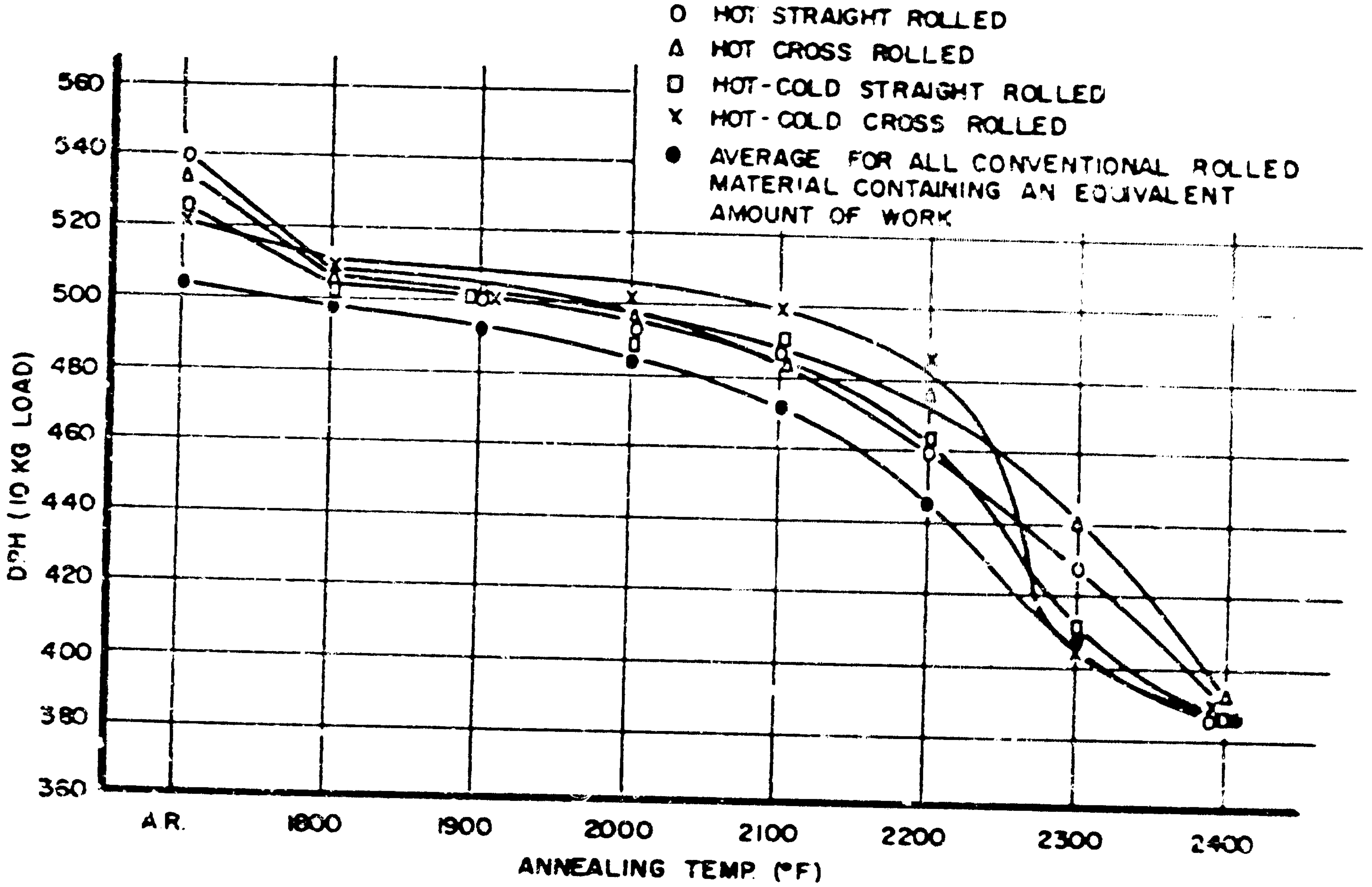
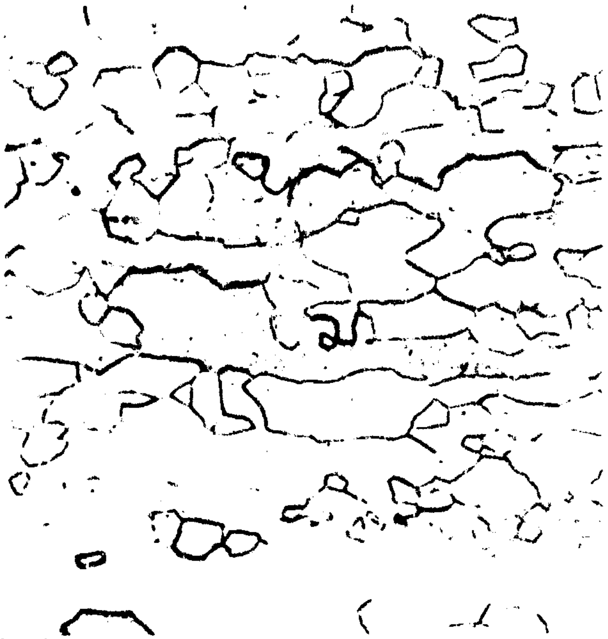


FIGURE 28

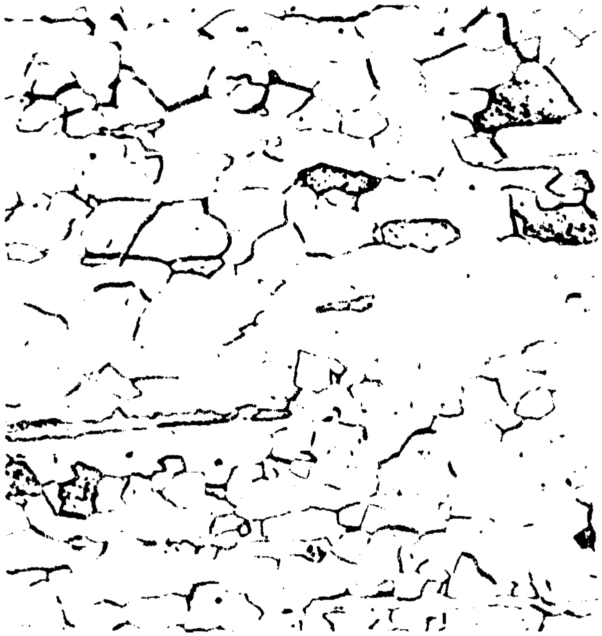
EFFECT OF VARIOUS INFAB ROLLING METHODS ON THE RESPONSE TO HEAT TREATMENT

RTA-0251

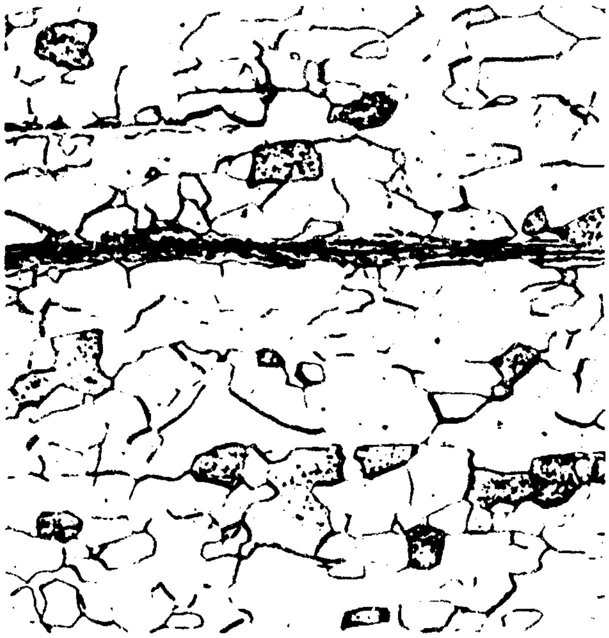
InFab Hot-Cold Rolled R11315



InFab Hot Rolled R11314



R11031
Conventionally Hot-Cold Rolled



R11033
Conventionally Hot Rolled

InFab Rolling Temperatures	
Hot-Cold	2400-2100-1800°F
Hot	3000-2100-1700°F

Conventional Rolling Temperatures	
Hot-Cold	2300-1900°F
Hot	2700-1900°F

All Samples Annealed One Hour - 2400°F
Magnification - 200X

FIGURE 29

COMPARISON OF INFAB AND CONVENTIONALLY ROLLED RECRYSTALLIZED MICROSTRUCTURES OF ARC CAST TUNGSTEN MATERIAL

TABLE VI

BEND TRANSITION TEMPERATURES - INFAB PROCESSED TUNGSTEN

Identification	Bend Direction	Rolling Temperature Initial	Rolling Temperature Inter.	Rolling Temperature Final	Bend Transition After Indicated Annealing 1800°F	Bend Transition After Indicated Annealing 1900°F
Hot-Cold Rolled	Long.	2400°F	2100°F	1800°F	375°F	400°F
Straight Rolled	Trans.				475°F	500°F
Hot-Cold Rolled	Long.	2400°F	2100°F	1800°F	400°F	400°F
Cross Rolled	Trans.				575°F	500°F
Hot Rolled	Long.	3000°F	2100°F	1700°F	450°F	425°F
Straight Rolled	Trans.				575°F	575°F
Hot Rolled	Long.	3000°F	2100°F	1700°F	450°F	400°F
Cross Rolled	Trans.				600°F	600°F

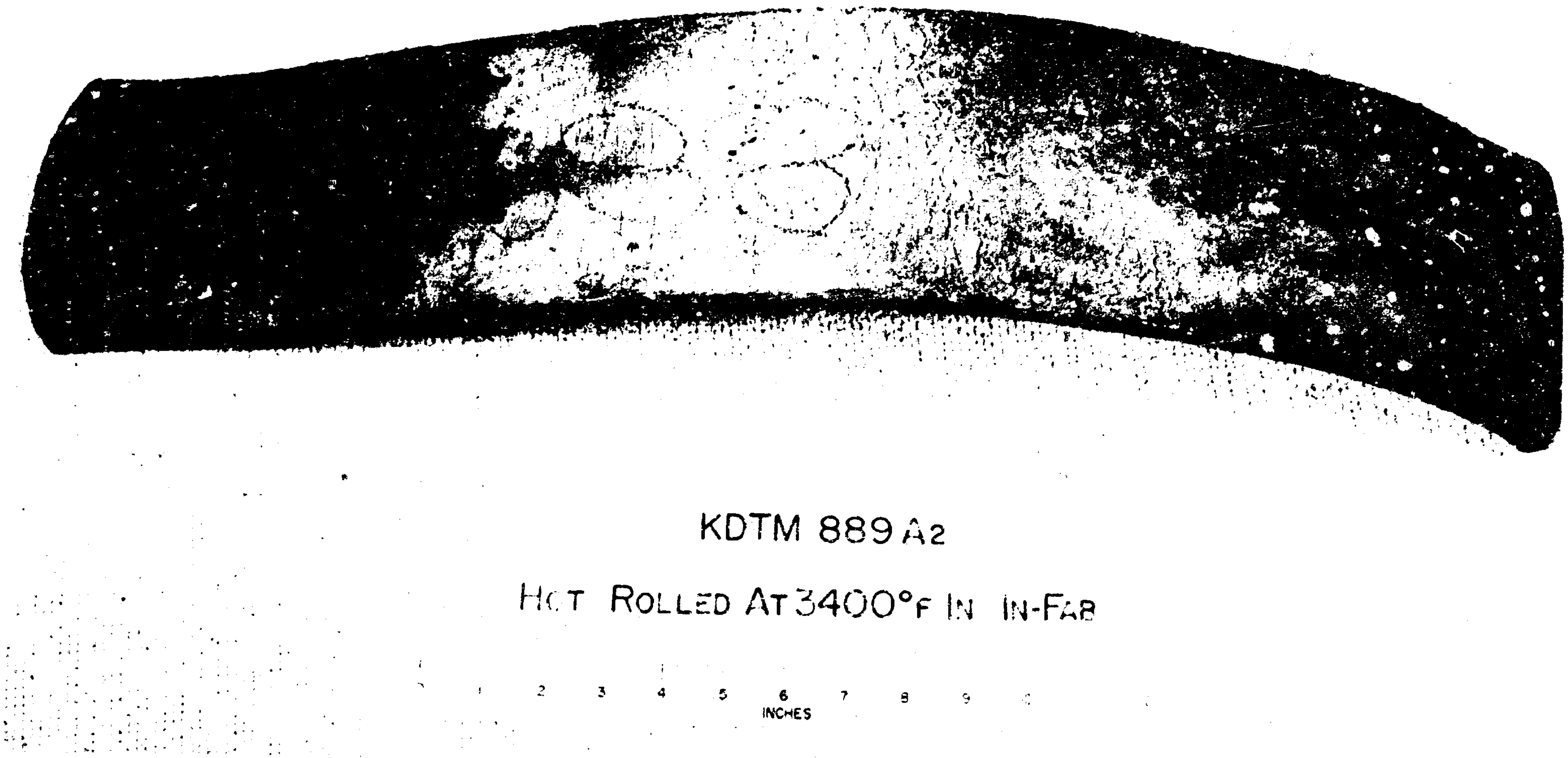


FIGURE 30

MO+0.5%TI HOT FORGED SHEET BAR ROLLED AT 3400°F problem when attempting to roll at very high temperatures. Superficial melting of the roll surface occurs, contaminating the material being rolled and causing it to stick to the rolls. Molybdenum spray coating may possibly eliminate this difficulty.

19. Rolling Under Contract NOas 59-6142-c

More than fifty samples of molybdenum alloys have been rolled under Contract NOas 59-6142-c. No attempt will be made in this report to give the details of these findings. A complete review of this work can be found in the interim reports and the final report to be published under Contract NOas 59-6142-c which will give a full account of the work accomplished.

20. Problems Still Remaining in Rolling

There are many improvements that could be made in the rolling set-up presently installed within the InFab enclosure. An attempt will be made to list all of the known ones although there are undoubtedly others that will be skipped.

a) Tolerance capabilities versus mill setting will have to be improved for each grade of material rolled.

b) The problem of carbon contamination should be eliminated with the installation of the new rolling furnace. This should improve operations considerably.

c) The problem of high temperature rolling and the associated contamination with iron from the rolls will be solved. This will require additional work on roll surfaces such as molybdenum or tungsten spray coating, refractory coating, cooling of rolls, use of special lubricants, or other modifications to roll surfaces.

d) A larger mill is required if sheet larger than 10" to 12" width is desired on a production basis.

e) Bar rolling must still be developed to the point where completely round bars are produced on a continuing and reproducible basis. This sounds easy, but requires considerable development effort.