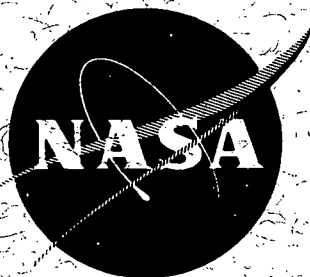


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NASA CR-120925



DEVELOPMENT OF WIRE DRAWING PROCESSES FOR  
REFRACTORY METAL FIBERS

by

G. W. King

CASE FILE  
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WESTINGHOUSE ELECTRIC CORPORATION

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## FOREWORD

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Stress-Rupture Testing of B-88 and ASTAR-811C	- Mr. R. Ammons (WANL)
Vacuum Encapsulation of B-88	- Mr. L. Stemann (WANL)
Electron Transmission Microscopy of B-88 and ASTAR-811C	- Dr. J. Cornie (R & D)
Continuous Annealing of B-88 Wire	- Dr. G. Comenetz (R & D)

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## Abstract

Fabrication schedules were developed for producing wire, 0.25 mm to 0.51 mm diameter, from the refractory metal alloys ASTAR-811C, B-88 and W-Hf-C. Tensile properties were evaluated at room temperature and up to 1204°C (2200°F). Also, the stress rupture properties of the alloys at 1093°C (2000°F) were determined. W-Hf-C and B-88 were found to have the best mechanical properties on a strength to density basis. The fabrication schedules for producing wire from these two alloys were further improved with regards to the wire quality and material yield under the optimization of fabrication schedule of the contract effort. An additional quantity of wire was produced for evaluation by NASA - Lewis.

## I. Summary

A fabrication schedule was developed (Task I) for producing approximately 31 meters (100 ft.) of 0.25 mm (0.01"), 0.38 mm (0.015") and 0.51 mm (0.02") diameter wire from the alloys ASTAR-811C, B-88 and W-Hf-C. The alloy ASTAR-811C proved to be the least difficult to fabricate, whereas W-Hf-C was the most difficult to fabricate.

The mechanical properties of the wire produced from these alloys were evaluated by tensile testing (room temperature, 1093°C and 1204°C) and also by 100 hour stress-rupture tests at 1093°C. The results showed that the W-Hf-C alloy is vastly superior in stress-rupture to B-88 or ASTAR-811C having a strength advantage (on a strength to density basis) which is 3 to 4 times greater than that of B-88 or ASTAR-811C. The alloy ASTAR-811C has the next highest stress-rupture strength, but on a strength to density basis is only slightly stronger than B-88. On the same basis, B-88 has the best room temperature tensile strength, followed by W-Hf-C and ASTAR-811C in that order. However, the elevated temperature tensile strength of W-Hf-C is best, followed by B-88 and ASTAR-811C in that order. The ductility of W-Hf-C is inferior to that of B-88 and ASTAR-811C, however, its room temperature ductility is comparable to that of commercial tungsten.

The alloys W-Hf-C and B-88 were selected for further development work in Task II. The fabrication schedules employed for producing wire from both alloys were optimized, with the major emphasis placed on improving the material yield and/or the quality of wire at finished sizes. Finally, ~2,994 meters of 0.38 mm diameter W-Hf-C wire and ~2,389 meters of 0.51 mm diameter B-88 wire were produced by an improved fabrication schedule. This wire was delivered to NASA - Lewis in a straightened and cleaned condition. Thus, all of the objectives of this contract effort were successfully met.

## 11. Introduction

Refractory-metal alloy wires are of interest for fiber-reinforcement of superalloy type matrix materials for use between 1093°C and 1204°C because of their high strength at these temperatures. In previous work at the NASA - Lewis Research Center, composites of refractory metal fiber reinforced-nickel base alloys were produced that had stress-rupture properties superior to conventional superalloys at use temperatures of 1093°C and 1204°C (1, 2). Stress for 1000-hour rupture values as great as six times that for the strongest conventional superalloys were obtained at 1093°C. Even stronger composites are possible if higher strength fibers were available since the high strength properties of the composite is largely based on the properties of the fiber. The need for stronger refractory metal alloy wire was recognized and efforts were sponsored by the Lewis Research Center to fabricate stronger alloys into wire form.

The purpose of the present contract was to produce wire from two of three high strength refractory metal alloys, for use in fiber reinforcing studies. The candidate materials (nominal compositions in weight percent) were: (1) W-Hf-C (W-0.35 Hf-0.026 C), (2) B-88 (Cb-28 W-2 Hf-0.07 C) and (3) ASTAR-811C (Ta-8 W-1 Re-1 Hf-0.25 C). These alloys were furnished by NASA - Lewis in the form of centerless ground swaged rods or extrusion bars. The work was performed in two tasks. In Task I, the objectives were to develop a fabrication schedule for each of the three alloys and to produce ~31 meters of 0.25 mm, 0.38 mm and 0.51 mm diameter wire for an evaluation of their mechanical properties at both room temperature and at 1093°C. Based on the outcome of these evaluations, two alloys were to be selected for further development work under Task II.

In Task II, the objectives were to optimize the fabrication schedules developed under Task I (with regard to material yield and/or improving mechanical properties) and to produce from each of the two selected alloys a maximum of 3,040 meters of wire at a size between 0.25 mm and 0.51 mm diameter.

### III. Detailed Description of Program

#### A. Task I

##### 1. Starting Materials

The alloys ASTAR-811C and B-88 were supplied by NASA - Lewis in the form of 6.35 mm diameter centerless ground rods, and the alloy W-Hf-C in the form of  $\sim 25.4$  mm diameter Mo-clad extrusion bars. The vendor for all three alloys was Westinghouse Astronuclear Laboratories, Pittsburgh. The vendor's report on the production and primary fabrication of the three alloys is summarized in Appendix A.

##### 2. Experimental Procedures

In the course of this development program for fabricating wire from high strength refractory metal alloys, the techniques employed were mainly those common to the art. Therefore, only a brief description of the experimental procedures employed and a definition of terms is given in the following:

Hand Swaging - Rods were heated to furnace temperature and then removed to swage one-half of the rod. The procedure was repeated for the second half of the rod. Temperatures reported are furnace temperatures.

Continuous Swaging - Rods were dynamically heated during swaging. Temperatures reported are approximated from the color temperature of the rod during swaging.

Continuous Annealing - Same as for continuous swaging except that the temperatures reported are furnace temperatures.

Chemical Removal of Cladding (Molybdenum) - Molybdenum cladding was removed from W-Hf-C extrusion bars or swaged B-88 rods in a solution of 4 parts  $\text{HNO}_3$  (40%): 2 parts  $\text{H}_2\text{SO}_4$  (66%): 1 part  $\text{H}_2\text{O}$ .

W-Hf-C extrusion bars ( $\sim 2.54$  cm O.D. x  $\sim 38$  cm long) were immersed in an open fiber glass tank under a hood. Swaged B-88 rods were placed in a closed system consisting of 2.54 cm O.D. PVC tubing vented on both ends to a hood. The solution was gradually admitted to the system through a valve connected to one end.

Chemical Cleaning of W-Hf-C Extrusion Bars - The operation was performed in a fused salt bath of sodium nitrate ( $\text{NaNO}_3$ ). The extrusion bars were heated to a dark red and then immersed with steel hooks into the fused salt bath. The reaction is rapid and appropriate safety precautions must be exercised.

Continuous Chemical Cleaning of B-88 and ASTAR-811C Swaged Rods and Wire - A solution of 1 part HF (70%): 1 part  $\text{HNO}_3$  (40%): 1 part  $\text{H}_2\text{O}$  was employed to remove 0.025-0.125 mm from the diameter of B-88 wire or swaged rods. ASTAR-811C was electrolytically or chemically cleaned in 1 part HF (70%): 1 part  $\text{H}_2\text{O}$  containing 160 gms NaCl per 100 mls of solution. B-88 swaged rods were fed by hand at a rate of about 3 cm/sec through 2.54 cm O.D. PVC tubing (U-shaped) which contained about 150 mls of the solution. The reaction was extremely volatile with copious fumes of  $\text{NO}_2$  being exuded. This operation is potentially hazardous and further development work is needed to improve the safety of the operation.

In wire cleaning, the same solution ( $\sim 200$  mls) is placed in a U-shaped 2.54 cm O.D. tygon tube about 0.56 meters long. A wire speed of about 4.23 cm/sec removes about 0.127 mm from the diameter of black (graphite lubricated) wire.

Split Detection in Wire - A Forster Crack Detector (Model No. 2.156) was used to non-destructively detect splits in drawn B-88 wire (0.51 mm to  $\sim 1.21$  mm diameter). The instrument works on an induced eddy current principle in which cracks or splits are indicated by a sharp change in the output of the induced eddy current.

### 3. Preliminary Investigations

#### a. ASTAR-811C and B-88

The primary concern in considering fabrication schedules for these alloys was the possibility of catastrophic atmospheric contamination occurring should high temperatures be required for swaging or annealing during wire fabrication. Previous investigations (3, 4) on the response of these alloys to heat treatment involved high temperature anneals ( $\geq 1600^\circ\text{C}$ ) of one hour or more in vacuum. However, to satisfy practical wire production conditions, it would be necessary to continuously preheat or anneal long lengths of rod at high temperatures for short times in an inert atmosphere (a vacuum furnace of this type being too costly to consider).

Therefore, a preliminary investigation was made to determine the effect of high temperature anneals in argon on the mechanical properties and microstructure\* of B-88 and T-222 (an alloy very similar to ASTAR-811C). Disc samples (about 3.20 mm thick) were cut from  $\sim 7.60$  mm diameter rods and were annealed in a radiant furnace at  $1700^{\circ}\text{C}$  for 5 minutes in argon (99.6% pure). For comparison, samples were also annealed for 5 minutes in the same furnace at  $1700^{\circ}\text{C}$  in vacuum ( $\sim 10^{-5}$  torr). After annealing, the hardness of the samples was measured, and they were also examined metallographically. The results showed both samples (whether in argon or vacuum) to be recrystallized into an equiaxed structure, and there was no evidence whatsoever to indicate that local contamination had occurred. The hardness of the B-88 was reduced from  $\sim 360$  VHN to  $\sim 310$  VHN, and the hardness of T-222 was reduced from  $\sim 360$  VHN to  $\sim 295$  VHN. These are typical hardness values for the recrystallized alloys.

The above results established the feasibility of recrystallizing B-88 and ASTAR-811C for short times in an inert atmosphere without deleterious effects to their mechanical properties. Later on in the program, various in-process anneals were employed in fabricating these alloys to the required wire sizes. For this purpose, an existing induction furnace was employed for continuous annealing of alloy rods. The furnace (Fig. 1) consists of a six turn 3.2 cm I.D. induction coil powered by a 50 KW, 10 KC motor generator. The coil was placed around a 1.65 cm I.D. x 2.54 cm O.D. x 7.62 cm long molybdenum susceptor which provided a uniform heat zone of about 7.62 cm at temperatures up to  $\sim 1800^{\circ}\text{C}$ . The chamber surrounding the susceptor and the induction coil was sealed and flushed with argon to minimize oxidation of the molybdenum susceptor during operation. Also, argon was passed under pressure through an inlet tube in the molybdenum susceptor (Fig. 1) in order to prevent contamination of the alloy rods during the anneal. Interchangeable steel plugs were inserted in the ends of the susceptor (water-cooled nearest the hot zone) in which a hole just large enough was drilled to accommodate the particular size rod being annealed. This arrangement resulted in a high rate of flow of argon from the area between the plug and the rod and prevented air from leaking into the furnace during the anneal. It was possible to anneal rods with diameters as small as  $\sim 1.78$  mm diameter at temperatures of  $1500^{\circ}\text{C}$ - $1800^{\circ}\text{C}$  without contamination.

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\* See section on Evaluation of Wire Properties, page 29 for a detailed description of microstructures.

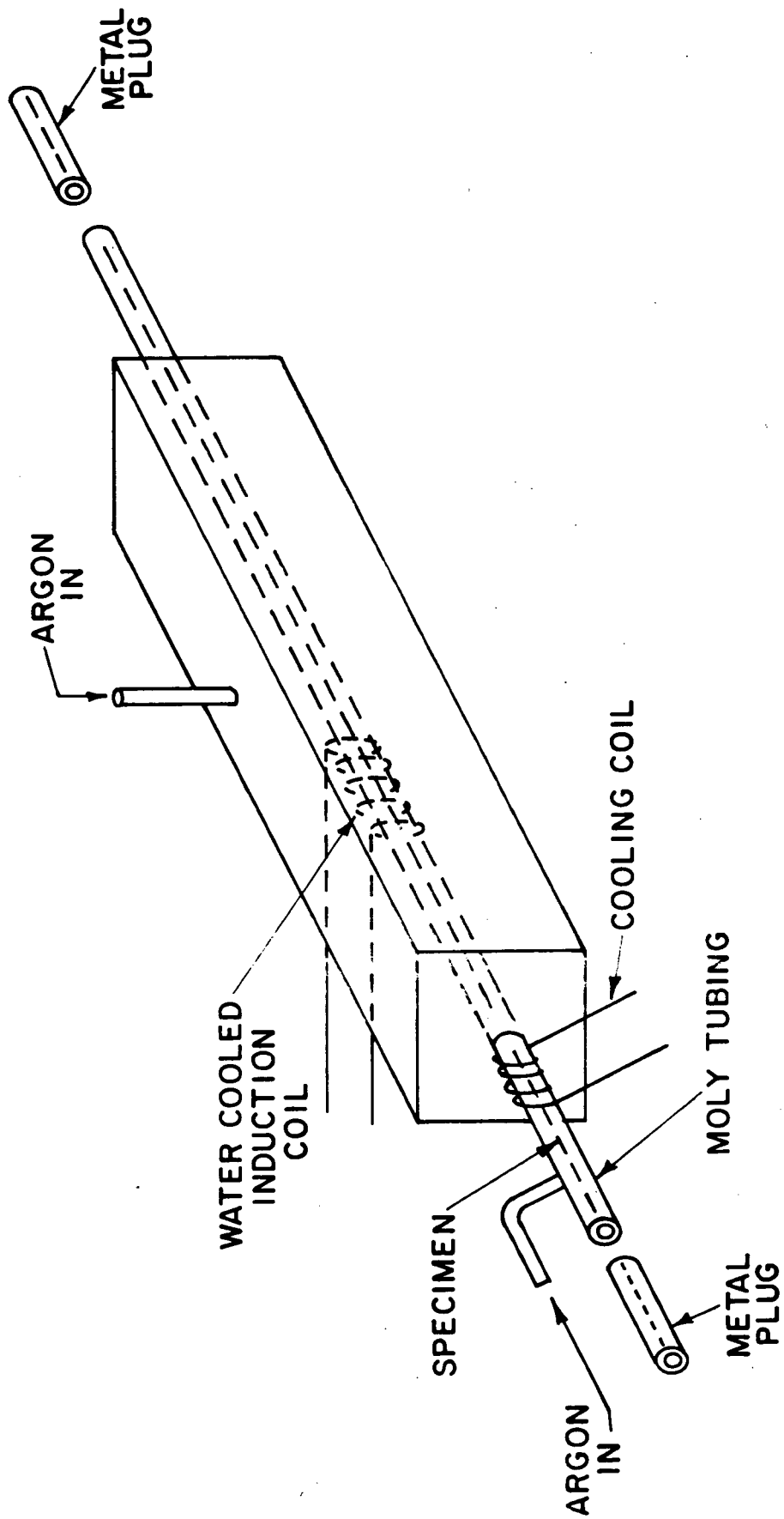


Fig. 1 Schematic Representation of an Induction Furnace Used for Annealing ASTAR-811C and B-88 Rods.

## b. W-Hf-C

Previous work (5) performed in the development of this alloy had indicated that carbon loss could occur during annealing at temperatures above 1600°C, particularly if oxygen was present. Swaging and annealing temperatures in excess of 1600°C were anticipated for fabricating W-Hf-C wire. Therefore, a test was made to determine whether a standard hydrogen atmosphere molybdenum wound swaging furnace could be employed for preheating during fabrication, without carbon losses occurring in the W-Hf-C. Disc samples of W-Hf-C alloy ( $\sim 6.35$  mm diameter after conditioning) were annealed for 1 hour at 1650°C in the H<sub>2</sub> atmosphere swaging furnace and also for 1 hour in vacuum ( $\sim 1 \times 10^{-5}$  torr) for comparative purposes. The anneals, whether in H<sub>2</sub> or vacuum, had no noticeable effect on the composition, microstructure or hardness of the alloy.

## 4. Development of Fabrication Schedules and Production of Wire

The main problems encountered in developing a fabrication schedule for each alloy are outlined below, and the approaches taken to remedy the problem are also stated.

### a. ASTAR-811C

The main difficulty encountered in producing wire from ASTAR-811C stemmed from wire breaks which occurred sporadically during drawing, and attempts to remedy the problem of local brittleness were only partially successful.

The variables investigated in developing a preliminary fabrication schedule for this alloy are summarized in Table I, with a brief discussion of the effect of each of the processing steps given below.

#### 1. Swaging Conditions

##### Preheat Temperatures

The alloy was swaged unclad to  $\sim 1.78$  mm diameter at room temperature and at 750°C. The hardness (Fig. 2) and microstructures of the rods swaged at room temperature and at 750°C were comparable. Also, the number of wire breaks which occurred during drawing were about the same regardless of swaging temperature. Therefore, all rods were subsequently swaged at room temperature so as to avoid the possibility of atmospheric contamination and/or the need for cladding.

Table 11

Summary of Variables Investigated in Developing a  
Fabrication Schedule for ASTAR-811C (Task 1)

<u>Operation</u>	<u>Extent of Investigation</u>
1. Swaging	
a. Preheat Temperature	Room Temperature and 750°C
b. Red. in Area Per Pass	10% to 31%
c. In-Process Anneals	1600°C to 1800°C
2. Wire Drawing	
a. Preheat Temperature	Room Temperature and 700°C
b. Die Temperature	Room Temperature and 300°C
c. Drawing Speed	6.1 cm/sec to 15.8 cm/sec
d. Red. in Area Per Pass	10% to 20%
e. Surface Preparation	Chem. Cleaning and Electropolishing
f. Lubricants	Chlorolube 40H, Cargyl and Vydax AR
g. Oxidation	500°C and 700°C

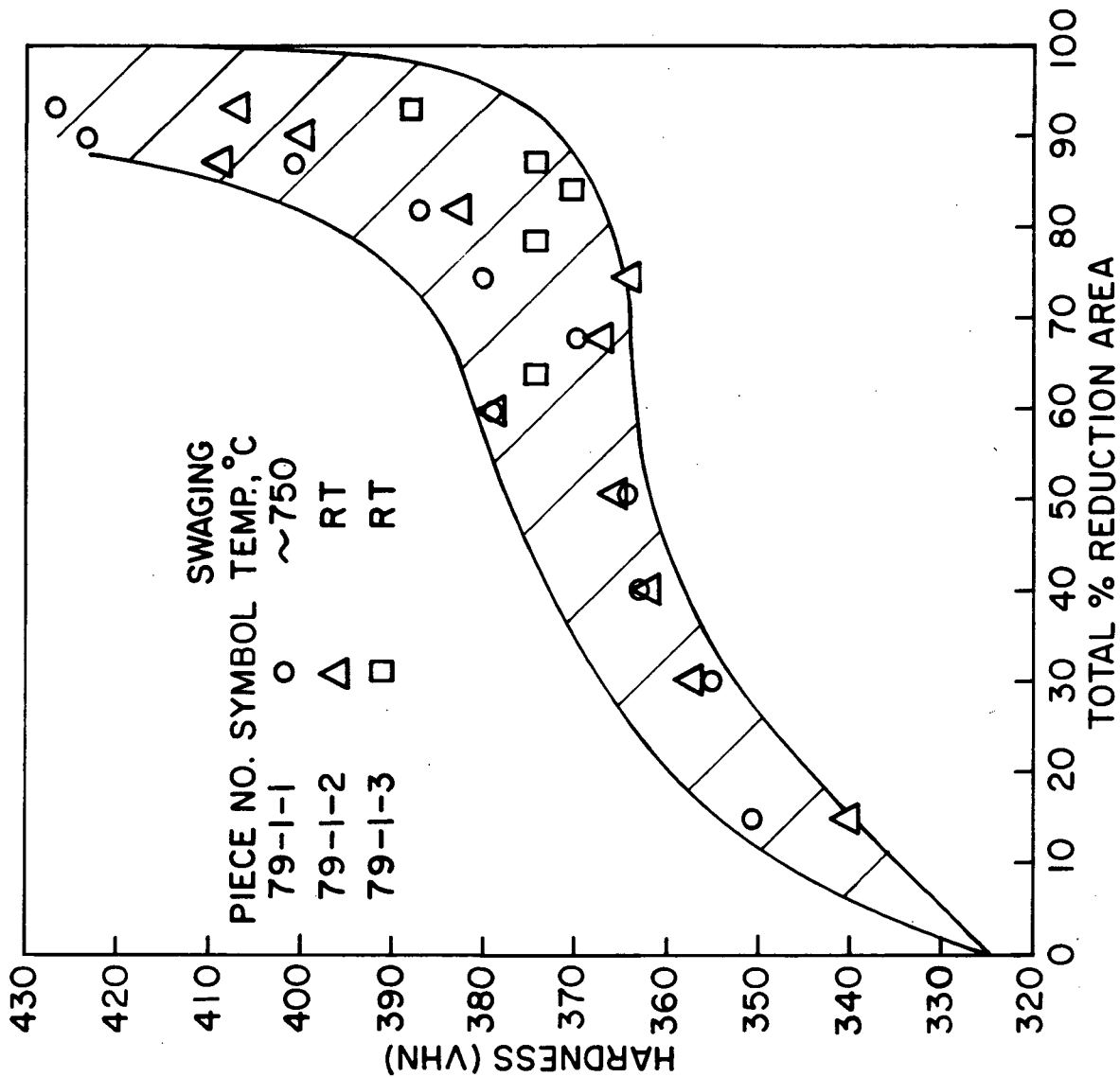


FIG.2 EFFECT OF SWAGING ON ROOM TEMPERATURE HARDNESS OF ASTAR-811C.

### Reduction in Area Per Pass

Reductions in area were varied from  $\sim 10\%$  R.A. to 31% R.A. Without deleterious effect.

### In-Process Anneals

Swaged rods were annealed at temperatures between 1600°C and 1800°C during, and/or, at the completion of swaging. Annealing at 1600°C for short times results in recovery, whereas, at 1700°C or higher the alloy recrystallizes with a loss in ductility.

## 2. Wire Drawing Conditions

### Preheat Temperatures

Wire was drawn at room temperature and at 700°C. Numerous breaks occurred when the wire was drawn at 700°C, and the wire was generally brittle after drawing. On the contrary, wire drawn at room temperature was generally ductile after drawing except for locally brittle areas which could not be associated with wire drawing conditions.

### Die Temperatures

Wire was drawn, with comparable results, using dies which had no preheat and dies which were preheated to 300°C.

### Wire Drawing Speeds

Wire drawing speeds were varied from about 6.1 cm/sec to about 15.8 cm/sec. A greater number of breaks occurred at the higher wire drawing speeds.

### Reductions in Area Per Pass

Reductions in area per pass up to 20% R.A. were employed. A greater number of breaks occurred at 20% R.A. and therefore, the reductions in area per pass were limited to 10% R.A.

### Surface Preparation

Removing 0.10 to 0.20 mm from the diameter by chemical cleaning or electropolishing prior to annealing did not eliminate local brittleness.

## Lubricants

Vydax AR or Chlorolube 50H were both suitable for lubrication. Cargyl was only used when drawing at elevated temperatures.

## Oxidation of Wire

Rods were oxidized at temperatures of 500°C and 700°C in order to obtain better lubrication for wire drawing. Rods oxidized at 700°C became brittle, while rods oxidized at 500°C were not embrittled.

### 3. Production of Wire for Task I Evaluation

The fabrication schedules employed for producing between 46 meters and 59 meters of ASTAR-811C wire at sizes of 0.25 mm, 0.38 mm and 0.51 mm diameter, respectively, are reported in Tables II and III\*. The fabrication schedules differ mainly by the fact that one rod (79-4) was annealed twice for  $\frac{1}{2}$  minute at 1600°C during fabrication, whereas the second rod (79-4A-1) was annealed only once for 1 minute at 1700°C.

#### b. B-88

A number of the variables investigated in developing a fabrication schedule for B-88 were important to the successful production of wire (see Table IV). The most critical requirement stemmed from the fact that surface cracks were formed during swaging, and these cracks caused the rods to be extremely brittle. However, after removing approximately 0.10 to 0.20 mm from the rod diameter by chemical cleaning, the swaged rods ( $\sim 1.78$  mm diameter) were completely ductile at room temperature.

A brief discussion of the effect of the variables listed in Table IV on fabricability of B-88 is given below.

#### 1. Swaging Conditions

##### Preheat Temperatures and Use of Cladding

Rods were swaged at temperatures of 750°C with and without the use of molybdenum cladding. The rods swaged

---

\* Of the wire produced, between 31 meters and 56 meters of wire at each size was delivered to NASA - Lewis in a straightened and cleaned condition.

Table II

Fabrication Schedules for Producing 0.51 mm and  
0.38 mm Diameter ASTAR-811C Wire Under Task I

<u>Rod No.</u>	<u>Step No.</u>	<u>Operation(a)</u>
79-4 and 79-4-1	I	Swaged from 6.60 mm to $\sim 3.66$ mm diameter ( $\sim 15\%$ R.A. per pass), then electropolished to 3.58 mm diameter and annealed $\frac{1}{2}$ minute at $1600^{\circ}\text{C}$ in argon. Swaged from 3.58 mm to 2.82 mm diameter.
79-4 and 79-4-1	II	Centerless ground at 2.82 mm diameter to 2.72 mm diameter and annealed $\frac{1}{2}$ minute at $1600^{\circ}\text{C}$ in argon. Swaged to 1.78 mm diameter ( $\sim 15\%$ R.A. per pass).
79-4	III	Drawn to 0.70 mm diameter ( $\sim 10\%$ R.A. per pass). Wire was oxidized at $500^{\circ}\text{C}$ at sizes of 1.78 mm, 1.65 mm, 1.33 mm and 1.07 mm diameter, after which it was oxidized each pass at $500^{\circ}\text{C}$ in line with drawing to 0.70 mm diameter.
79-4	IV	Acid cleaned at 0.70 mm diameter and drawn to 0.51 mm diameter (10% R.A. per pass) without further oxidation using Vydax AR as lubricant.
79-4-1	I	A second rod was fabricated to 1.07 mm diameter by the same schedule reported for 79-4, then acid cleaned and drawn to 0.38 mm diameter without oxidizing using Vydax AR as lubricant.

(a) All swaging was performed at room temperature, and all drawing was performed at room temperature but with the die heated to  $\sim 200^{\circ}\text{C}$ . Chlorolube 50H was used for lubrication prior to using Vydax AR. Wire speed varied from 1.22 to 3.69 meters/min.

Table III

Fabrication Schedule for Producing 0.25 mm Diameter  
ASTAR-811C Wire (Rod No. 79-4A-1) Under Task I

<u>Step No.</u>	<u>Operation(a)</u>
I	Swaged from 6.60 mm to 1.65 mm diameter (10-21% R.A. per pass). Rod acid cleaned at 4.17 mm diameter then annealed 1 minute at 1700°C.
II	Drawn to 0.38 mm diameter (10% R.A. per pass). Wire oxidized 1 minute at 500°C at sizes of 1.65 mm, 1.14 mm, 0.74 mm and 0.36 mm diameter. Chlorolube 50H used as lubricant.

(a) All Swaging was performed at room temperature, and all drawing was performed at room temperature but with the die heated to ~200°C. Wire speed varied from 1.22 to 4.57 meters/min.

Table IV

Summary of Variables Investigated in Developing  
A Fabrication Schedule for B-88 (Task I)

<u>Operation</u>	<u>Extent of Investigation</u>
1. Swaging	
a. Preheat Temperature	1100°C to 600°C
b. Use of Cladding	Unclad & Vacuum Clad in Molybdenum
c. In-Process Anneals	1100°C to 1800°C
d. Red. in Area Per Pass	10% to 33%
2. Wire Drawing	
a. Surface Preparation	Electropolishing & Chem. Cleaning, Oxidation at ~ 600°C
b. Preheat Temperatures	400°C
c. Die Temperature	400°C
d. Drawing Speed	1.22 meters/min. to 3.69 meters/min.
e. Red. in Area Per Pass	10%
f. Lubricants	Cargyl

without cladding were badly split and broke several times during swaging. On the other hand, the rods swaged with cladding (vacuum encapsulated) were inspected after the cladding was removed and were found to be free of macroscopic defects but were extremely brittle. Subsequently, all rods were vacuum encapsulated in 6.35 mm I.D. molybdenum tubing for swaging. In general, rods were initially preheated to  $\sim 1100^{\circ}\text{C}$  and continuously swaged to 1.78 mm diameter (core) with the swaging temperature being reduced to about  $700^{\circ}\text{C}$  as soon as the cladding ruptured locally. The lowest swaging preheat temperature employed in swaging was  $\sim 600^{\circ}\text{C}$ . The reason for reducing the swaging temperature was to avoid excessive local atmospheric contamination.

### In-Process Anneals

The clad B-88 alloy was annealed at temperatures between  $1100^{\circ}\text{C}$  and  $1800^{\circ}\text{C}$  at various points in the swaging schedule. Rods annealed at  $1800^{\circ}\text{C}$  were recrystallized and broke up badly in subsequent swaging. On the other hand, rods were also annealed at  $1100^{\circ}\text{C}$  and/or at  $1500^{\circ}\text{C}$  during swaging, and these rods were not recrystallized but were softened by the anneal and were swaged without fracturing.

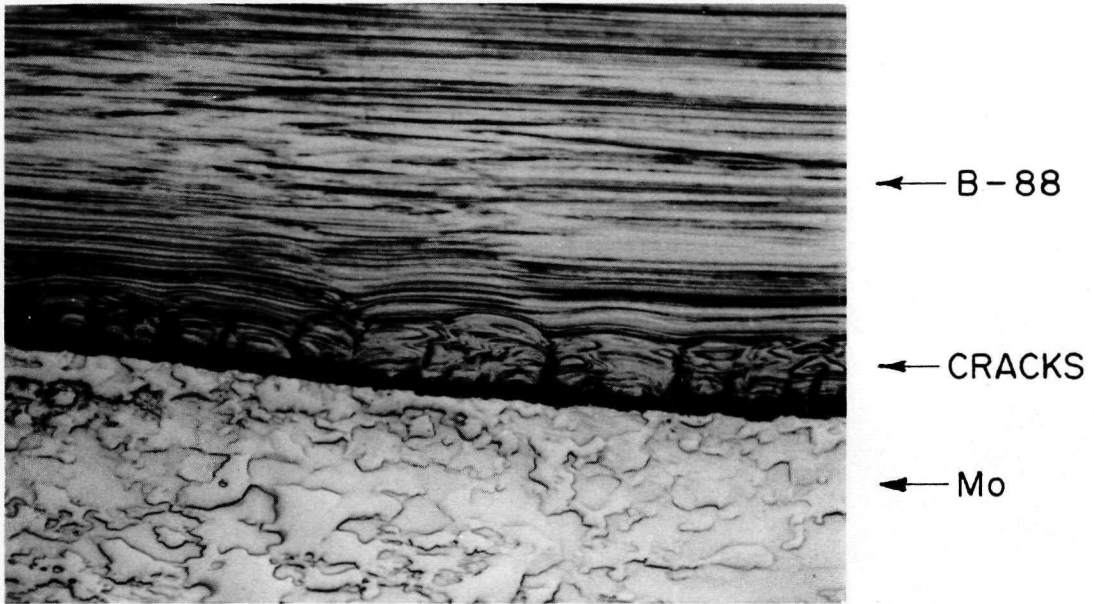
### Reductions in Area Per Pass

Reductions in area were varied from 10% R.A. to about 33% R.A. per pass. There appeared to be a greater tendency for the cladding to fracture at the higher ( $>20\%$ ) reductions in area; therefore, in wire production, swaging was limited to a maximum of about 20% R.A. per pass.

## 2. Wire Drawing Conditions

### Surface Cleaning and Oxidation for Wire Drawing

The most important steps in producing wire from B-88 are surface cleaning and oxidation of rods for subsequent wire drawing. All as-swaged rods of B-88 ( $\sim 1.78$  mm diameter) after the removal of the cladding were found to be extremely brittle at room temperature, regardless of the swaging conditions employed. The cause of the brittleness was a damaged surface layer containing cracks up to about 0.05 mm in depth (Fig. 3). However, by removing about 0.10 to 0.20 mm from the diameter by chemical cleaning or electropolishing, the swaged rods were completely ductile at room temperature. In order to provide an oxide surface for better lubrication in wire drawing, the cleaned rods



200X

Fig. 3 Photomicrograph Showing Surface Cracks in B-88 (Molybdenum Clad) Rod Swaged to 3.66 mm Diameter.

were swaged one to two passes at  $\sim 600^{\circ}\text{C}$  before drawing to wire.

### Preheat Temperatures, Die Temperatures and Drawing Speeds

In wire drawing, the rods were continuously heated in a 1.3 meter long furnace at  $\sim 400^{\circ}\text{C}$ , at speeds of 1.22 to 3.66 meters per minute and drawn thru dies heated to  $\sim 400^{\circ}\text{C}$ . The preheat temperatures for drawing were kept at a maximum of about  $400^{\circ}\text{C}$  in order to minimize surface contamination. The relatively low speeds employed for wire drawing were necessary to prevent "chatter" of the wire in the die (The chatter was probably caused by poor lubrication resulting from the relatively thin oxide film.). The effect of die temperature was not investigated; but, in general, decreasing the die temperature from  $400^{\circ}\text{C}$  would possibly have caused more wire breaks.

### Reductions in Area Per Pass

Because of the die chatter (mentioned above), all wire drawing of B-88 (in Task I) was at 10% reduction in area per pass.

### Lubricants

The only lubricant employed was Cargyl.

### 3. Production of Wire for Task I Evaluation

A total of about 46 to 61 meters of B-88 wire was produced at sizes of 0.25 mm, 0.38 mm and 0.51 mm diameter, respectively, and 31 meters of wire at each size was delivered to NASA - Lewis. Three different rods were used to produce the wire, and all rods were fabricated by the schedule reported in Table V.

#### c. W-Hf-C

This alloy was very difficult to fabricate because not only was it extremely brittle in the starting condition, but also the quality of the molybdenum clad extrusion bars was very poor. However, a fabrication schedule was developed in Task I for producing 0.25 mm to 0.51 mm diameter wire. The main features of the fabrication schedule are the use of high temperatures for initial breakdown swaging passes on molybdenum clad, as-received, extrusion bars, and re-cladding of swaged rods for further fabrication after surface conditioning in a fused salt bath.

Table V

Fabrication Schedule for Producing 0.51 mm, 0.38 mm  
and 0.25 mm Diameter B-88 Wire Under Task I

<u>Step No.</u>	<u>Operation</u>
I	Swaged* with molybdenum cladding from $\sim 8.61$ mm diameter to 3.66 mm diameter (clad dimensions) from a preheat temperature of $1100^{\circ}\text{C}$ (10% to 20% R.A. per pass). Rods annealed for 1 minute at $1100^{\circ}\text{C}$ at sizes of 4.78 mm, 4.45 mm and 3.94 mm diameter.
II	Swaged to $\sim 2.39$ mm diameter ( $\sim 10\%$ to 20% R.A. per pass) from a preheat temperature of $\sim 650^{\circ}\text{C}$ .
III	Annealed clad rods $\frac{1}{2}$ minute at $1500^{\circ}\text{C}$ in argon, then removed cladding (rods $\sim 1.78$ mm diameter).
IV	Removed 0.10 mm to 0.20 mm from rod diameters by acid cleaning or electropolishing, then swaged 1 to 2 passes to $\sim 1.52$ mm diameter from a preheat temperature of $\sim 600^{\circ}\text{C}$ (oxidation of rod surface).
V	Drawn to final wire sizes (10% R.A. per pass) from a preheat temperature** of $400^{\circ}\text{C}$ , and dies heated to $\sim 400^{\circ}\text{C}$ . Cargyl used as lubricant.

\* Hand swaged first 2 passes.

\*\* Dynamically preheated in a 1.22 meter electric furnace, while drawing at 1.22 to 3.69 meters/min.

The variables investigated in developing the preliminary fabrication schedule for W-Hf-C are summarized in Table VI, and a brief discussion of the effect of these variables on wire production is given below.

## 1. Swaging Conditions

### Use of Cladding

Attempts were made to swage sections of as-received extrusion bars with and without the use of molybdenum cladding. The as-received bar sections which were swaged without cladding either broke up or split badly after one or more swaging passes (10-50% R.A.). However, bars fabricated with the original cladding intact were swaged a number of passes ( $\sim 95\%$  R.A.) without severe surface cracks developing\*.

In some cases, sections of rods which were swaged to smaller sizes with the original cladding intact and were re-clad for further swaging after surface conditioning. These rod sections were also apparently free from surface cracks after they were swaged to a size suitable for wire drawing.

### Surface Conditioning

The two methods employed for surface conditioning were centerless grinding and immersion in a fused sodium nitrate salt bath. Because of the brittleness of the W-Hf-C alloy at heavy rod sizes, centerless grinding results in very large material losses. The losses occurred either when swage-straightening the bars for grinding, and/or during the grinding operation.

On the other hand, the use of a fused salt bath proved to be a very effective means of removing the rough surface from the as-received extrusion bars or swaged rods (Fig. 4). The utilization of this technique was perhaps the most important step in the development of a fabrication schedule for wire production.

### Preheat Temperatures

A preheat temperature of  $\sim 1800^{\circ}\text{C}$  (minimum) is required for initial swaging of as-received extrusion bars. This temperature can be gradually reduced to  $\sim 1600^{\circ}\text{C}$  after about 50% R.A. and to  $\sim 1100^{\circ}\text{C}$  after  $\sim 90\%$  R.A.

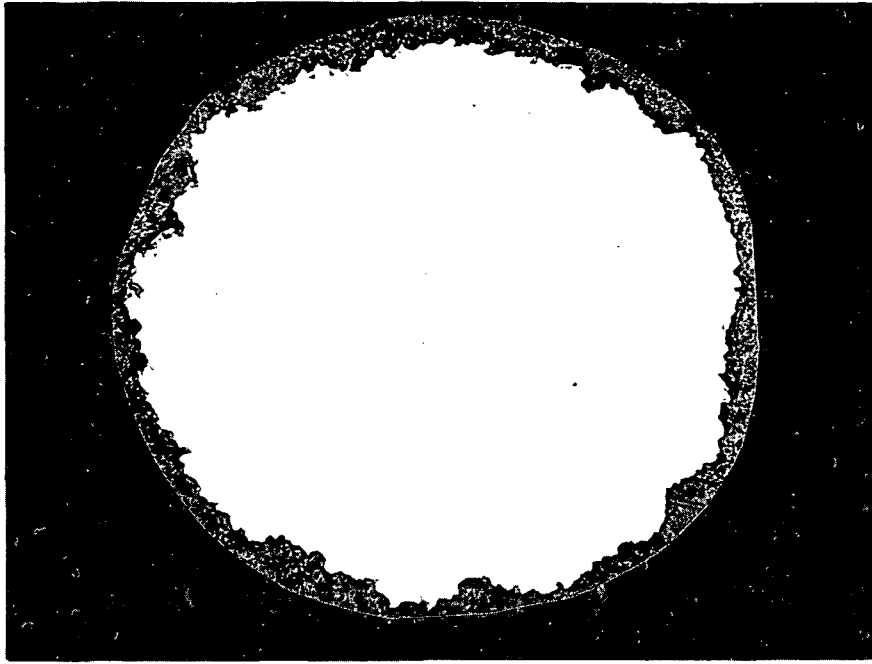
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\* Internal cracks were found in sections taken from the rods after swaging, but these cracks could have been present before swaging.

Table VI

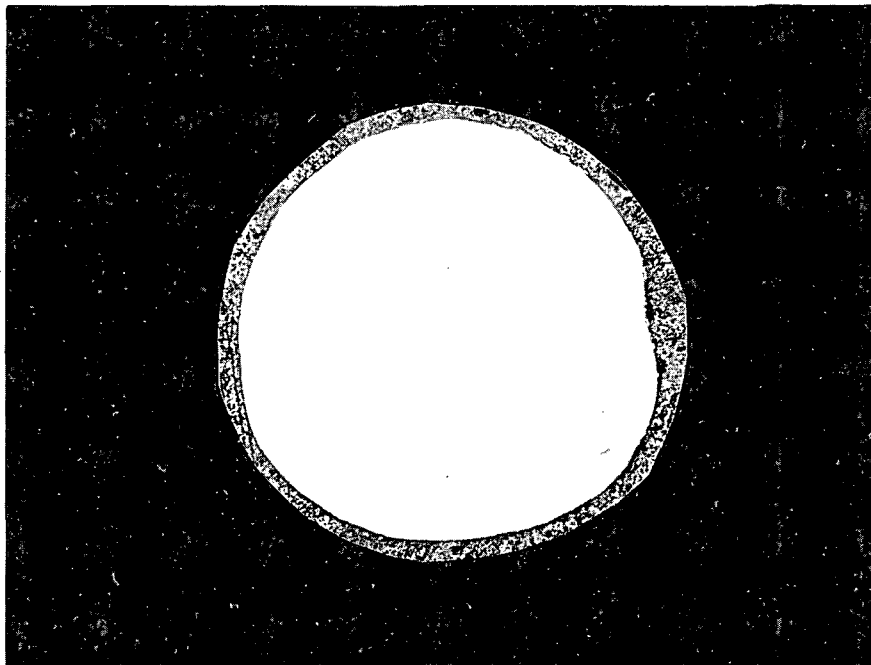
Summary of Variables Investigated in Developing  
A Fabrication Schedule for W-Hf-C (Task I)

<u>Operation</u>	<u>Extent of Investigation</u>
1. Swaging	
a. Use of Cladding	Swaged As-Clad, Unclad & Reclad
b. Surface Conditioning	Centerless Grind. & Fused Salt Bath
c. Preheat Temperatures	1850°C to 1100°C
d. In-Process Anneals	2000°C and 2200°C
e. Red. in Area Per Pass	10% to 30%
2. Wire Drawing	
a. Preheat Temperatures	1100°C to 700°C
b. Capstan Temperature	Room Temperature to 200°C
c. Die Temperature	400°C
d. Red. in Area Per Pass	10% to 35%
e. Drawing Speed	3.69 meters to 9.77 meters/min.
f. Lubricants	Cargyl



(a)

8.5X



(b)

8.5X

Fig. 4 Photomicrographs of a Swaged Rod of W-Hf-C:  
(a) Before and (b) After Removal of Surface  
in a Fused Salt Bath.

## In-Process Anneals

Anneals were performed at various intervals during swaging. An anneal of 1 hour at 2200°C caused the alloy to recrystallize with a loss in ductility during subsequent fabrication. An anneal at 2000°C for 10 to 20 minutes was also employed; however, this anneal is a recovery or stress relieving type anneal and improved the fabricability of the alloy in subsequent swaging steps.

## Reductions in Area Per Pass

Reductions in area of 10% to 30% per pass were employed without an apparent detrimental effect on wire fabrication.

## 2. Wire Drawing Conditions

### Preheat Temperatures

Initial preheat temperatures for drawing were about 1100°C and were decreased gradually to about 700°C at smaller wire drawing sizes.

### Capstan Temperature

The capstan (0.9 meter diameter) used for drawing from about 2.54 mm diameter must be heated to about 200°C to prevent wire breaks at heavy ( $>0.76$  mm diameter) wire sizes. At smaller wire sizes ( $<0.76$  mm diameter), the wire was drawn with an unheated 30.4 cm diameter capstan.

### Die Temperature

Dies were preheated to  $\sim 400^\circ\text{C}$  in an electrically heated die block.

## Reductions in Area Per Pass, Drawing Speeds and Lubricants

The reductions in area, wire speeds and the lubricant employed (Table VI) were typical for drawing tungsten.

## 3. Production of Wire for Task I Evaluation

Approximately 26-40 meters of wire at sizes of 0.25 mm to 0.51 mm diameter were produced by the fabrication

schedule reported in Table VII\*. The material supplied was produced by first swaging as-received extrusion bars with the original cladding to  $\sim 8.62$  mm diameter (core). Next, the cladding was chemically removed, and the swaged rod was immersed in a fused salt bath to remove  $\sim 2.54$  mm from the diameter. Afterwards, the rod was re-clad in 6.35 mm diameter molybdenum tubing and swaged to  $\sim 2.54$  mm diameter (clad). The cladding was removed, and the rods ( $\sim 1.78$  mm diameter) were drawn to wire by essentially standard wire drawing techniques. It is significant to note that the rods swaged to 2.54 mm diameter were ductile enough to bend  $\sim 90^\circ$  at room temperature.

5. Evaluation of Wire Properties of ASTAR-811C, B-88 and W-Hf-C at Sizes of 0.25 mm, 0.38 mm and 0.51 mm Diameter - Task 1

a. Chemical Analyses

Chemical analyses were made on samples of the alloys at various stages of fabrication. The results showed that the composition of heavy rod samples and 0.51 mm diameter wire were essentially the same. Therefore, only the results for the 0.51 mm diameter wire analyses are compared later (Table VIII) with the vendors reported average analysis of the starting ingots. The only case in which a large discrepancy exists is in the case of the oxygen content of W-Hf-C ingots and wire; the wire having an oxygen content (0.0039%) which is almost an order of magnitude higher than the average value reported for the starting ingot (0.0006%).

b. Tensile Properties

Tensile tests were made at room temperature, 1093°C, and 1204°C on wires of the three alloys at various sizes, and the average results (2 or more tests) are reported in Tables IX, X and XI. It is apparent that W-Hf-C has the highest strength at all test temperatures. However, on a strength to density basis, B-88 is superior to W-Hf-C at room temperature and is almost equal to W-Hf-C at elevated temperatures. On the same basis of strength to density, ASTAR-811C is inferior to B-88 and W-Hf-C under all test conditions.

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\* Of the wire produced, between 15 and 26 meters of wire was delivered to NASA - Lewis in a straightened and cleaned condition.

Table VII

Fabrication Schedule for Producing 0.51 mm, 0.38 mm  
and 0.25 mm Diameter W-Hf-C Wire Under Task I

Step No.

- I Swaged\* from  $\sim 2.54$  cm to 1.13 cm diameter (clad dimensions) from preheat temperature of  $1825^{\circ}\text{C}$  (initial) to  $\sim 1535^{\circ}\text{C}$  (final). Rod annealed for 20 minutes at  $2000^{\circ}\text{C}$  at 1.58 cm diameter.
- II Cladding removed ( $\sim 9.02$  mm core diameter) and W-Hf-C rod diameter reduced to  $\sim 6.20$  mm diameter by immersion in a fused sodium nitrate salt bath.
- III Swaged to  $\sim 5.61$  mm diameter ( $\sim 8\%$  to  $15\%$  R.A. per pass) from a preheat temperature of  $\sim 1650^{\circ}\text{C}$ , then cleaned and re-clad in 6.35 mm I.D. x 8.64 mm O.D. molybdenum tubing.
- IV Swaged to  $\sim 2.41$  mm diameter (18-28% R.A. per pass) from preheat temperatures of  $1500^{\circ}\text{C}$  (initial) to  $1100^{\circ}\text{C}$  (final).
- V Cladding removed ( $\sim 1.78$  mm core diameter) then drawn to final sizes (10-27% R.A. per pass) from preheat temperatures of  $\sim 900^{\circ}\text{C}$  (initial) to  $\sim 700^{\circ}\text{C}$  (final) and a die temperature of  $\sim 400^{\circ}\text{C}$ . Cargyl was used as a lubricant and wire drawing speeds of 1.22 meters to 1.83 meters/min. were employed.

\* Hand swaged from starting size to 1.13 cm diameter.

Table VIII

Chemical Analyses of Starting Materials  
and 0.51 mm Diameter Wire for the Various Alloys

<u>Alloy</u>	<u>Composition, Weight Percent</u>							<u>Bal.</u>
	<u>W</u>	<u>Hf</u>	<u>Re</u>	<u>C</u>	<u>O</u>	<u>N</u>	<u>H</u>	
ASTAR-811C (Ingot)	8.3	0.86	1.12	0.024	0.0030	0.0013	-----	Ta
ASTAR-811C (Wire)	8.2	0.91	1.13	0.027	0.0058	0.0026	-----	"
B-88 (Ingot)	27.8	2.12	-----	0.058	0.0084	0.0018	-----	Cb
B-88 (Wire)	28.3	1.94	-----	0.059	0.010	0.0029	-----	"
W-Hf-C (Ingot)	-----	0.37	-----	0.027	0.0006	0.0003	0.0004	W
W-Hf-C (Wire)	-----	0.37	-----	0.030	0.0039	0.0009	0.0016	"

Table IX

Tensile Properties\* of ASTAR-811C at Various  
Temperatures and Wire Sizes

<u>Test Temp. (°C)</u>	<u>Wire Size (mm)</u>	<u>0.2% Y.S. MN/M<sup>2</sup></u>	<u>Ult.Str. MN/M<sup>2</sup></u>	<u>Ult.Str. Density (meters)</u>	<u>Total** % Elong.</u>
22	0.25	1075	1356	8189	5.5
"	0.38	1013	1303	7868	6.0
"	0.51	911	1539	9289	8.1
1093	0.25	383	571	3452	5.0
"	0.38	500	868	5240	14.1
"	0.51	521	738	4458	9.8
1204	0.25	257	334	2019	8.4
"	0.38	469	635	3845	16.2
"	0.51	283	391	2360	44.5

\*  $(\text{MN/M}^2)/6.89 \times 10^{-3} = \text{PSI}$ .

\*\* 10.15 cm (4 inch) Estimated Gage Length.

Table X

Tensile Properties\* of B-88 at Various Temperatures  
and Wire Sizes

<u>Test Temp. (°C)</u>	<u>Wire Size (mm)</u>	<u>0.2% Y.S. MN/M<sup>2</sup></u>	<u>Ult.Str. MN/M<sup>2</sup></u>	<u>Ult.Str. Density (meters)</u>	<u>Total % Elong.</u>
22	0.25	742	1219	12047	6.6
"	0.38	1041	1601	15812	14.4
"	0.51	917	1515	14963	13.8
1093	0.25	259	319	3152	5.3
"	0.38	408	642	6340	9.2
"	0.51	433	638	6307	6.9
1204	0.25	239	312	3076	4.8
"	0.38	292	461	4552	8.5
"	0.51	332	471	4646	10.9

\* (MN/M<sup>2</sup>)/6.89 x 10<sup>-3</sup> = PSI.

\*\* 10.15 cm (4 inch) Estimated Gage Length.

Table XI

Tensile Properties\* of W-Hf-C at Various  
Temperatures and Wire Sizes

<u>Test</u> <u>Temp.</u> <u>(°C)</u>	<u>Wire</u> <u>Size</u> <u>(mm)</u>	<u>0.2%</u> <u>Y.S.</u> <u>MN/M<sup>2</sup></u>	<u>Ult.Str.</u> <u>MN/M<sup>2</sup></u>	<u>Ult.Str.</u> <u>Density</u> <u>(meters)</u>	<u>Total**</u> <u>%</u> <u>Elong.</u>
22	0.25	915	1662	8743	0.9
"	0.38	1357	1964	10335	1.1
"	0.51	1311	1973	10381	3.6
1093	0.25	579	968	5095	1.4
"	0.38	536	1082	5695	2.2
"	0.51	709	1114	5860	2.3
1204	0.25	586	1026	5395	1.4
"	0.38	528	1092	5748	2.3
"	0.51	570	1061	5537	2.1

\*  $(\text{MN/M}^2)/6.89 \times 10^{-3} = \text{PSI}$ .

\*\* 10.15 cm (4 inch) Estimated Gage Length.

### c. Stress-Rupture Properties

Constant load (internal loaded) stress-rupture tests were performed both in ultra high vacuum ( $\sim 1 \times 10^{-8}$  torr) at Westinghouse Astronuclear Laboratories, and at a nominal vacuum of  $\sim 10^{-6}$  torr at this laboratory. The results obtained for the three alloys are tabulated in Table XII. A logarithmic plot of the data in Table XII, in terms of the time to rupture versus applied stress, results in the following approximate values for the stress to produce rupture in 100 hours at 1093°C for the various alloys\*.

<u>Alloy</u>	<u>Stress** for 100 Hour Creep Rupture, MN/M<sup>2</sup></u>	<u>Creep Stress Density (Meters)</u>
B-88	69 ( 10.0)	683
ASTAR-811C	138 ( 20.0)	833
W-Hf-C	1055 (153.0)	5563

It is quite obvious from these results that W-Hf-C is vastly superior to B-88 or ASTAR-811C from the standpoint of stress-rupture properties under the conditions tested, even when judged on a strength to density basis. The data above also show that the alloy ASTAR-811C has a slight advantage over B-88 from the standpoint of creep stress to density ratio.

### d. Optical Microscopy and Hardness Measurements

Preparation of metallographic samples of all alloys was made by first grinding the samples through 4/0 emery paper, and afterwards polishing on a hard, low nap polishing cloth with a slurry made up of Linde B Alumina Powder ( $0.05 \mu\text{m}$ ) in a 3% chromic acid solution. Samples of B-88 and ASTAR-811C were etched in a solution of 1 part HF (49%): 1 part HNO<sub>3</sub> (conc.): 2 parts glycerine. Samples of W-Hf-C were etched in a solution of 10 gms KOH and 10 gms K<sub>3</sub>Fe(CN)<sub>6</sub> in 100 mls of H<sub>2</sub>O.

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\* All of the wire produced in Task I contained splits of varying lengths.

\*\* Numbers in parenthesis are for stress in KSI.

Table XII

Stress-Rupture Properties of As-Drawn Wires of  
the Various Alloys at 1093°C

<u>Alloy</u>	<u>Nominal Wire Diameter, mm</u>	<u>Stress*, MN/M<sup>2</sup></u>	<u>Time to Rupture, hrs.</u>
ASTAR-811C	0.51	586 ( 85.0)	0.1
"	"	448 ( 65.0)	2.4
"	"	276 ( 40.0)	22.9
"	"	207 ( 30.0)	27.5
"	0.38	207 ( 30.0)	23.5
"	0.25	138 ( 20.0)	51.4
"	"	103 ( 15.0)	172.0
B-88	0.51	338 ( 49.0)	1.0
"	"	276 ( 40.0)	0.5
"	"	207 ( 30.0)	3.3
"	"	103 ( 15.0)	17.0
"	0.38	310 ( 45.0)	0.3
"	"	103 ( 15.0)	23.8
W-Hf-C	0.51	689 (100.0)	180**
"	"	689 (100.0)	167**
"	0.38	1103 (160.0)	48.6
"	"	1069 (155.0)	67.3

\* Numbers in Parenthesis are for Stress in KSI.

\*\* No Fracture.

All of the microstructural data reported below are for samples produced by the fabrication schedules reported in Tables II, V and VII, respectively. Hardness data are recorded along with the microstructures for all conditions except final production wire, which is too small for macro-hardness testing.

(ASTAR-811C): The microstructure of the as-received (6.35 mm diameter swaged rods) of ASTAR-811C consisted of slightly elongated grains, about  $40\mu\text{m}$  wide by  $120\mu\text{m}$  long (Fig. 5a).

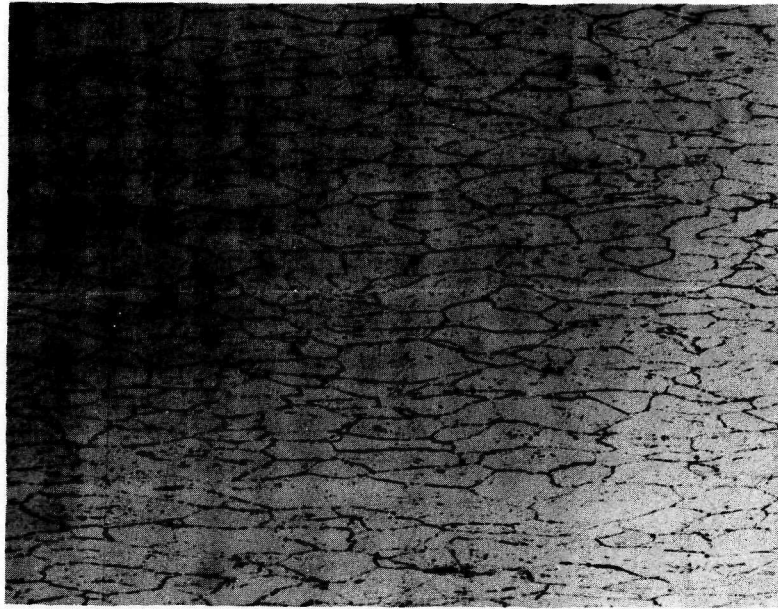
The microstructure of the alloy after swaging to  $\sim 1.70$  mm diameter consisted of fine fibrous grains. However, it was characteristic of the swaged microstructures that the fiber pattern changed abruptly as shown in Fig. 5b.

The in-process anneals employed had the effect of lowering the hardness of the swaged alloy from  $\sim 410$  VHN to  $\sim 324$  VHN, but otherwise had no effect on the microstructure (stress-relief).

The microstructure of the alloy in the final drawn condition consisted of fibrous grains (too small to resolve by optical microscopy) and in addition, what appears to be stringers of second phase particles (Fig. 6a). Recrystallization of the alloy results in the formation of equiaxed grains of  $\sim 20\mu\text{m}$  diameter (Fig. 6b). However, annealing for short times (2 minutes at  $1800^\circ\text{C}$ ) does not homogenize the alloy, i.e. the "stringers of second phase particles" along former fiber boundaries are not removed.

(B-88): The microstructures of B-88 and ASTAR-811C were similar in many respects. The as-received alloy had a fibrous microstructure which was fairly uniform (Fig. 7a). However, after swaging the alloy to  $\sim 1.78$  mm diameter by the schedule reported in Table V, sharp disruptions in the fibrous microstructure were evident (Fig. 7b). The microstructure of the as-drawn wire (Fig. 8a) was characterized by very fine fibers and what appears to be second phase particles aligned along the fiber boundaries.

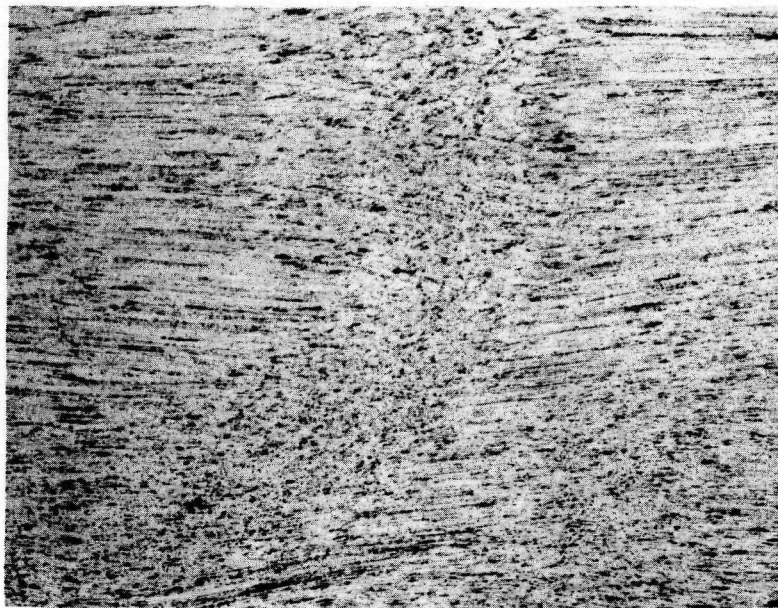
Annealing the wire ( $\frac{1}{2}$  minute at  $1500^\circ\text{C}$ ) after working had no effect on the microstructure but lowered the hardness from about 380 VHN to 360 VHN. The swaged alloy can be recrystallized by annealing for 2 minutes at  $1800^\circ\text{C}$ . This results in an equiaxed grain structure of  $\sim 30\mu\text{m}$  diameter grains (Fig. 8b) and a hardness of  $\sim 320$  VHN.



(a)

325 VHN

100X

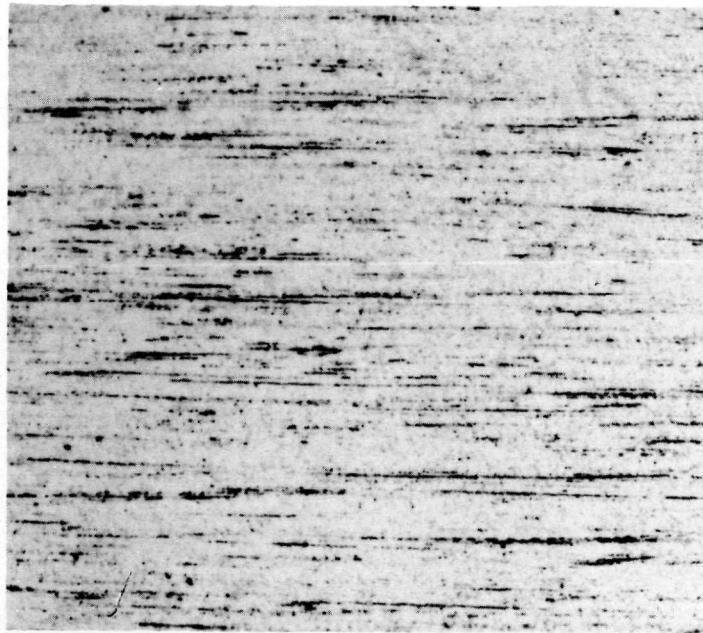


(b)

410 VHN

100X

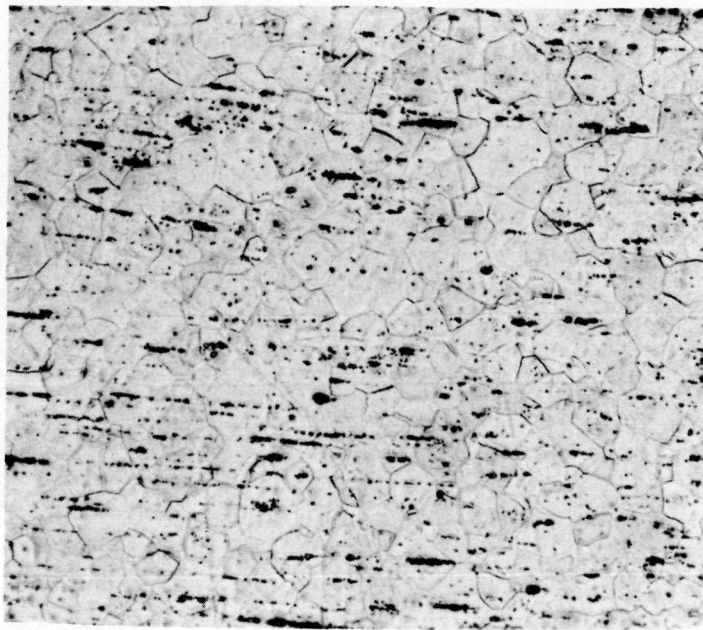
Fig. 5 Optical Microstructure of ASTAR-811C.  
(a) As-Received and (b) As-Swaged to  
1.70 mm Diameter.



(a)

450 VHN

400X

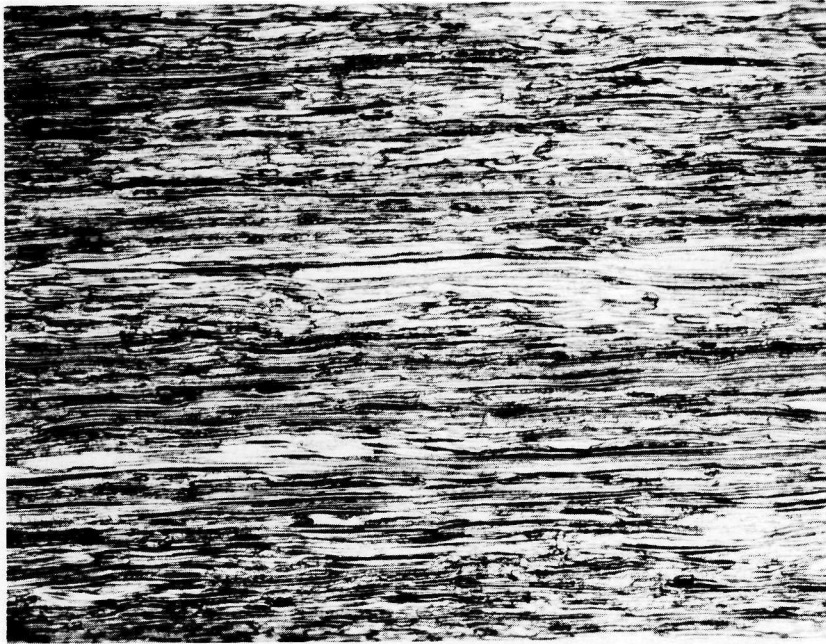


(b)

310 VHN

200X

Fig. 6 Optical Microstructures of ASTAR-811C.  
(a) As-Drawn to 0.38 mm Diameter and  
(b) Annealed 2 Minutes at 1800°C.



(a)

350 VHN

100X

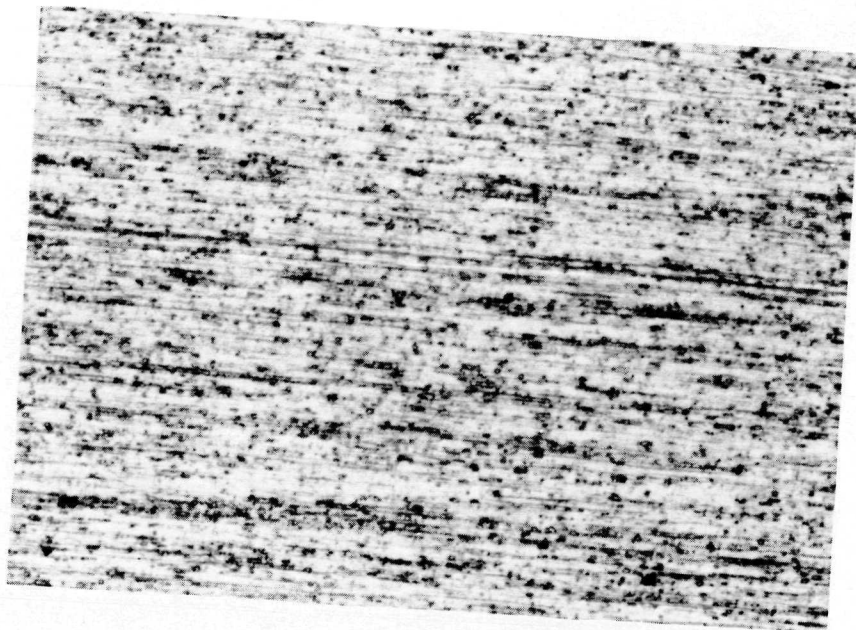


(b)

390 VHN

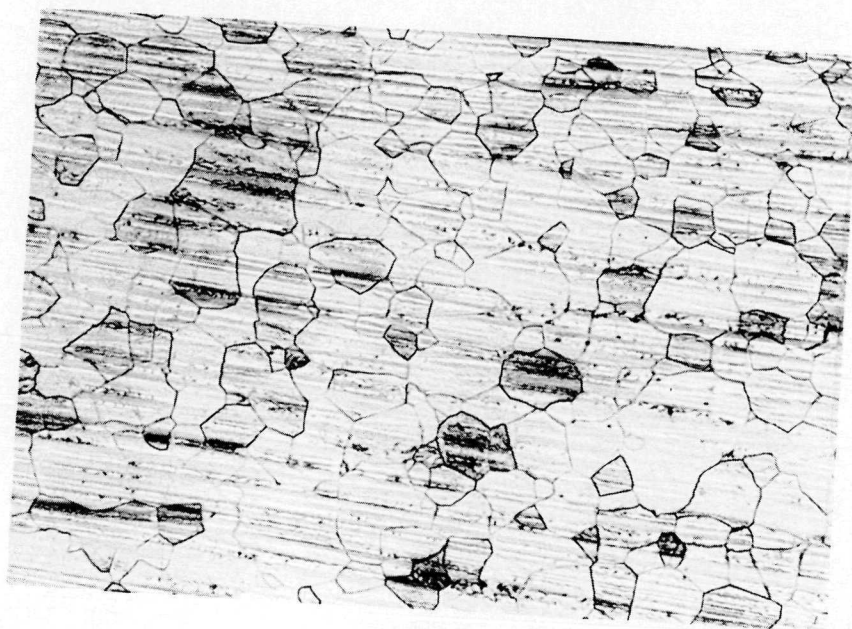
100X

Fig. 7 Optical Microstructures of B-88.  
(a) As-Received and (b) As-Swaged to  
1.78 mm Diameter.



(a)

400X



(b)

320 VHN

200X

Fig. 8 Optical Microstructures of B-88.  
(a) As-Drawn to 0.46 mm Diameter and  
(b) Annealed 2 Minutes at 1800°C.

(W-Hf-C): The microstructure of the as-received alloy consisted of highly elongated grains, varying in width from about  $20\mu\text{m}$  to as large as  $250\mu\text{m}$  (Fig. 9a) and appeared to be a single phase alloy. After swaging the alloy to 2.54 mm diameter, the grain structure consisted of very fine fibers (Fig. 9b), which became even finer in the as-drawn condition (Fig. 10a). The in-process anneals (20 minutes at  $2000^{\circ}\text{C}$ ) had no effect on the grain structure of the alloy but reduced the hardness from  $\sim 560$  VHN to  $\sim 490$  VHN. The alloy can be recrystallized by annealing for 1 hour at  $2200^{\circ}\text{C}$ , and results in the formation of slightly elongated irregular shaped grains (typical of secondary recrystallization) with an average width of  $\sim 70\mu\text{m}$  (Fig. 10b) and a hardness value of  $\sim 405$  VHN.

#### e. Electron Transmission Microscopy

The details of specimen preparation of the various alloys for electron transmission microscopy (ETM) studies are reported in Appendix B. Specimens of ASTAR-811C and B-88 were prepared from swaged 1.78 to 2.04 mm diameter rods which were stress-relieved for 30 minutes at  $1204^{\circ}\text{C}$  to prevent cracking during specimen preparation. Specimens of W-Hf-C were prepared from 1.78 mm diameter swaged rod, and also from 0.38 mm diameter wire. The swaged rod was annealed for 72 hours at  $1093^{\circ}\text{C}$ , whereas the wire was annealed for 50 hours at  $1232^{\circ}\text{C}$  prior to preparing the specimens. A summary of the observations for the various alloys is given below:

The structure of B-88 was found to consist of elongated fibrous grains with a diameter of  $\sim 0.3\mu\text{m}$  in the transverse direction and 5 to  $10\mu\text{m}$  in the longitudinal direction (Fig. 11a). Carbide precipitates were present as  $\sim 0.05\mu\text{m}$  diameter platelets within the fiber grains, but the high dislocation density prohibited examination of small precipitates at the fiber boundaries (Fig. 11b). Many of the dislocations appear to be stabilized by pinning or decoration with precipitates, and much debris was noted in the boundaries.

The ASTAR-811C structure was not quite as elongated and fibered as the B-88 alloy (Fig. 12a). However, some areas did appear to be fibrous (Fig. 12b), but these areas did not represent the predominant structure. The dislocation density within subgrains of ASTAR-811C was much lower than observed in B-88 and few precipitates were noted in the ASTAR-811C alloy. Likewise, the grain boundaries and subgrain boundaries were relatively free of debris.

The substructure of the W-Hf-C alloy in the swaged and stress-relieved condition was characterized by highly elongated fibers about  $1.0\mu\text{m}$  in width (Fig. 13). Many precipitates were evident, some being as large as about  $0.5\mu\text{m}$  and other as small as  $0.02\mu\text{m}$  (Fig. 14). The larger precipitates appear as lenticular platelets and appear to have been broken during fabrication. However, it is believed that what is observed is more likely an electron diffraction contrast effect (possibly caused by lattice strains). This observation is deduced from the fact that the "contrast effect" in the particles is the same regardless of the orientation of the particle with respect to the specimen axis. It is also evident from Fig. 14 that very little debris is present within subgrain boundaries, although in some areas simple types of dislocation networks can be seen. The substructure of 0.38 mm diameter wire (stress-relieved, 50 hours at  $1230^{\circ}\text{C}$ ) is shown in Fig. 15. The fiber width has decreased to about  $0.18\mu\text{m}$ , and no increase in the amount of debris within the fibers is apparent.

## 6. Selection of Two Alloys for Task II

Based on the mechanical property data presented earlier, the selection of W-Hf-C for further development work in Task II was an obvious choice. This alloy has superior stress-rupture properties and also good tensile strength properties in comparison to the remaining two alloys when judged from the standpoint of strength to density ratios.

The choice of the second alloy was not quite as obvious since B-88 has a better tensile strength to density ratio but a lower creep stress to density ratio than ASTAR-811C. Therefore, the final selection was guided mainly by the intended end use of the composite materials. This will involve (in some cases) components of fixed design; and in such instances, the total weight of the particular component is of the greatest importance. It was primarily on this basis that B-88 was selected as the second alloy for Task II, since the use of this alloy rather than ASTAR-811C (in components of fixed design) would result in a lighter member with little or no sacrifice of strength properties.

### B. Task II

#### 1. Optimization of Fabrication Schedules for B-88

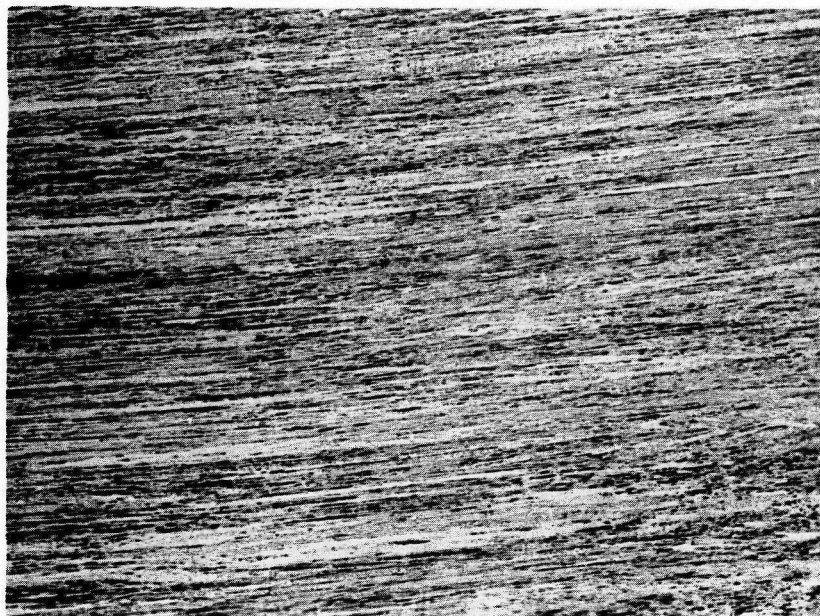
The optimization of the fabrication schedule employed for producing B-88 wire was performed with two different objectives in mind: (a) to improve the creep-rupture properties of



(a)

510 VHN

40X



(b)

580 VHN

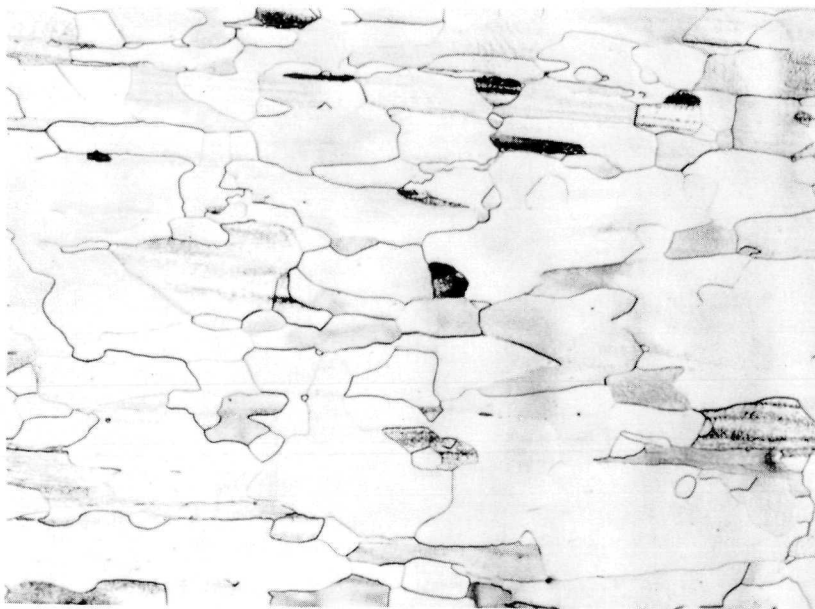
100X

Fig. 9 Optical Microstructures of W-Hf-C.  
(a) As-Received and (b) As-Swaged to  
0.25mm Diameter.



(a)

400X

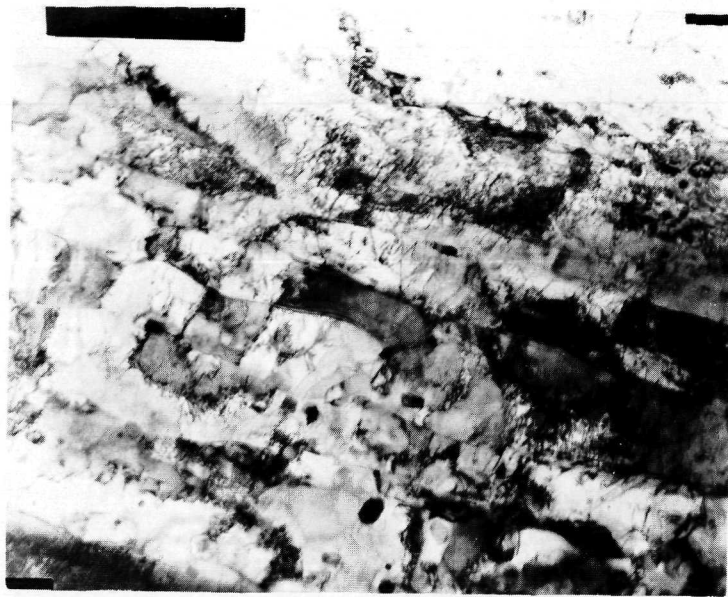


(b)

405 VHN

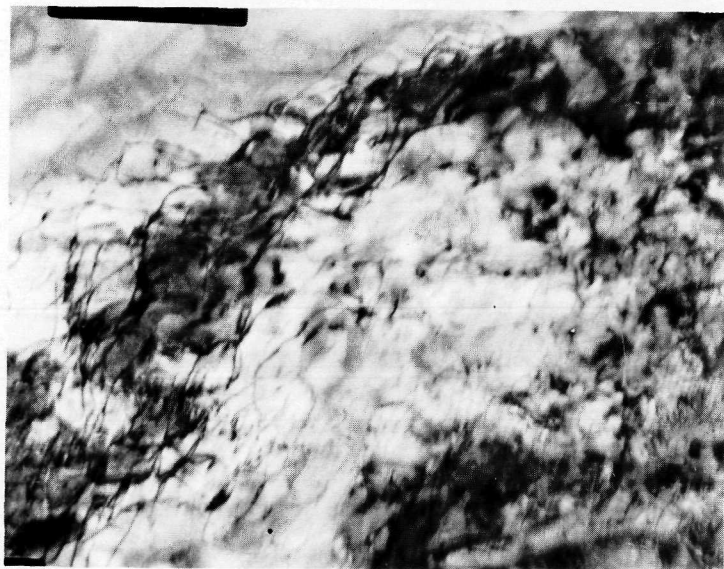
100X

Fig. 10 Optical Microstructures of W-Hf-C.  
(a) As-Drawn to 0.38 mm Diameter and  
(b) 1.64 cm Diameter Annealed 1 Hour at  
2200°C.



(a)

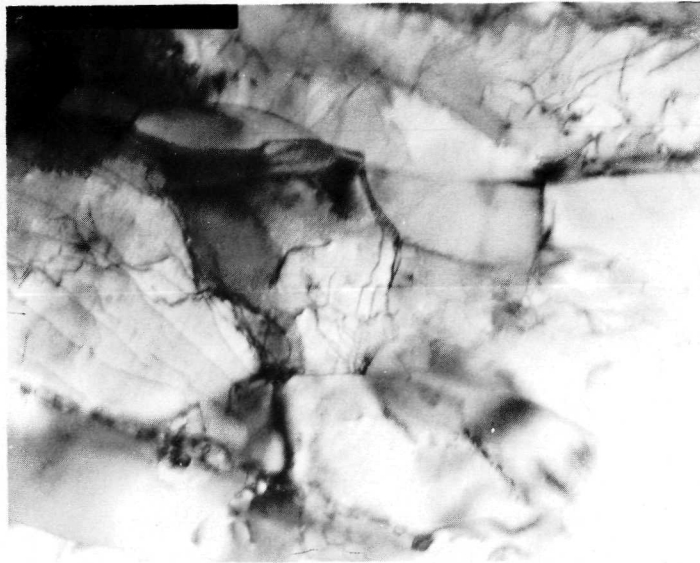
1  $\mu$



(b)

.25  $\mu$

Fig. 11 Transmission Electron Micrographs of 1.91 mm Diameter Swaged B-88 Rod Stress Relieved 30 Minutes at 1204°C. (a) Cell Size and (b) Dislocation Tangles at Grain Boundaries.



(a)

.25 $\mu$



(b)

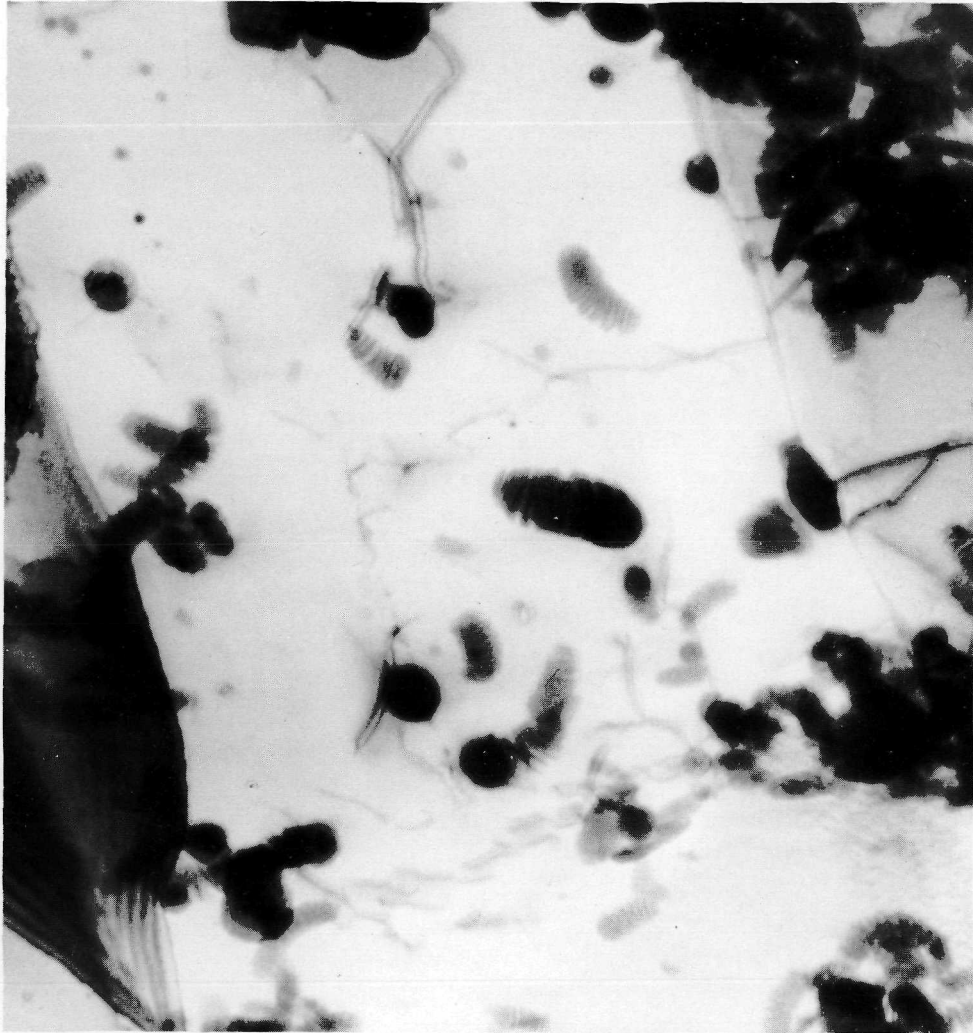
1 $\mu$

Fig. 12 Transmission Electron Micrographs of ASTAR-811C 1.91 mm Diameter Swaged Rods Stress Relieved 30 Minutes at 1204°C. (a) Typical Subgrains and (b) Fibered Subgrains.



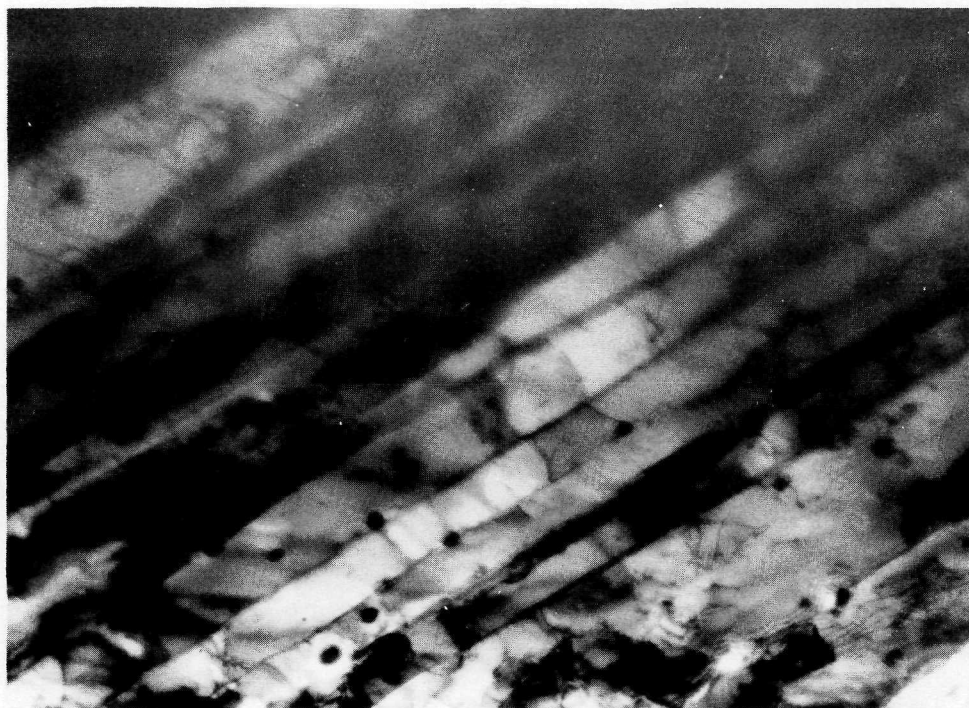
.5 $\mu$   
┌──────────┐

Fig. 13 Transmission Electron Micrograph of W-Hf-C  
1.78 mm Diameter Swaged Rod Annealed 72 Hours  
at 1093°C.



.25 $\mu$

Fig. 14 Transmission Electron Micrograph of W-Hf-C  
1.78 mm Diameter Swaged Rod Annealed 72 Hours  
at 1093°C.



.5 $\mu$   
┌──────────┐

Fig. 15 Transmission Electron Micrograph of W-Hf-C  
0.38 mm Diameter Wire Annealed 50 Hours at  
1232°C.

the finished wire (0.51 mm diameter), and (b) to reduce the number of splits which had previously been detected in the Task I production wire during its evaluation. By decision of the Contract Manager, the major emphasis was placed on the latter objective; however, enough work was directed towards the first objective to establish that annealing would increase the creep-rupture strength of B-88 wire.

a. Effect of Annealing on the Stress-Rupture Strength of B-88

Work previously performed at Westinghouse Astronuclear Laboratories (2) had shown that annealing B-88 at increasingly higher temperatures improved its stress-rupture life (SRL). Therefore, a decision was made to evaluate the effect of annealing on the SRL of 0.51 mm diameter wire.

Initially, short sections of wire were annealed in situ in a vacuum radiation furnace at temperatures of 1700°C to 1800°C for ~2.5 minutes, and then cooled to 1093°C. Stress-rupture (SR) tests were then performed by applying an external load through a bellows type vacuum seal with the test load corrected for the effect of vacuum pressure. The results clearly indicated that annealing improved the SRL of B-88\*. Therefore, a system was designed and constructed by the Westinghouse R and D Center, Pittsburgh, for continuously annealing 0.51 mm diameter wire up to 1925°C in vacuo (see Appendix C). Also, the creep testing apparatus employed was modified to permit SR testing at this laboratory with internally applied loads. A comparison of results between externally loaded and internally loaded specimens indicated that at small test loads, the externally loaded test results were not accurate. Therefore, only the results obtained on internally loaded specimens are reported in Table XIII. In addition, the data in Table XIII includes SR tests made on as-worked B-88 wire which had been split tested and shown to be free of splits prior to SR testing.

The results (Table XIII) show that the elimination of splits does not improve the SRL of as-worked B-88 wire (compare results in Tables XII and XIII). On the other hand, annealing above the recrystallization temperature improves the SRL,

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\* Room temperature tensile strength was reduced: 0.2% Y.S. = 928 MN/M<sup>2</sup>, Ult. Str. = 931 MN/M<sup>2</sup>, % Elong. = 10.7%.

Table XIII

Effect of Annealing on the Stress-Rupture  
Properties of 0.51 mm Diameter B-88 Wire at 1093°C

<u>Annealing*</u> <u>Temperature, °C</u>	<u>Applied</u> <u>Stress, MN/M<sup>2</sup></u>	<u>Time to</u> <u>Rupture, hrs.</u>
As-Worked	338	0.97
"	310	0.25
1825	345	8.22
1925	345	20.08

\* All anneals performed dynamically in vacuo with wire at temperature for approximately 7 seconds.

with higher SRL at the higher annealing temperature. Wire which was continuously annealed at both 1825°C and 1925°C was delivered to NASA - Lewis for further evaluation.

b. Effect of Fabrication Schedule on Split Formation and Brittleness in B-88 Wire

The variables investigated with regard to reducing splits in the finished wire involved variations in: (1) swaging temperatures, (2) in-process anneals and (3) wire drawing temperatures. The effect of these variables on wire quality are summarized as follows:

1. Effect of Swaging Temperature

The swaging temperature was maintained at approximately 1100°C in swaging eight rods to 2.54 mm diameter (clad dimension). As previously noted, however, the cladding ruptures locally; in most instances, at larger rod diameters (3.82-4.07 mm diameter). The first two rods swaged were ductile after cleaning and were drawn to 0.51 mm diameter by the drawing schedule previously established (400°C preheat) without breaks. However, three out of six of the remaining rods were completely brittle or partially brittle even after removing 0.25-0.38 mm from the diameter. The conclusion drawn was that the swaging temperature must be reduced from 1100°C to ~650°C after the molybdenum cladding ruptures in order to prevent local contamination that embrittles the wire.

2. Effect of In-Process Anneals

The previously developed fabrication schedule (Task 1) employed in-process anneals at 1100°C during swaging, and an inert-atmosphere anneal at 1500°C after swaging to ~2.54 mm diameter. Two rods were swaged by the same schedule except that the 1100°C in-process anneals were omitted. These rods were ductile after annealing at 1500°C and after cleaning. The conclusion drawn was that the 1100°C anneals could be eliminated.

A third rod was swaged as above and the 1500°C anneal was eliminated. This rod was ductile after cleaning, but split tests made with an eddy current device indicated that numerous splits were present after only two drawing passes. The conclusion drawn was that it would be beneficial to retain the 1500°C anneal at the finished swaged size.

### 3. Effect of Wire Drawing Temperature

An eddy current crack detector was used to determine the apparent density of splits in a section of wire at a large wire diameter ( $\sim 1.32$  mm diameter). The split trace indicated that the number of splits at that size was small and not too severe in depth. The wire was then drawn by the standard schedule, namely:  $400^{\circ}\text{C}$  preheat,  $400^{\circ}\text{C}$  die temperature and 10 to  $\sim 20\%$  R.A. per pass. The wire was again examined for splits at  $\sim 1.07$  mm diameter, and the number of splits had increased in drawing.

A section of wire that had been annealed at  $1500^{\circ}\text{C}$  was drawn at preheat temperatures of  $600^{\circ}\text{C}$ - $800^{\circ}\text{C}$  and split traces made at 1.32 mm, 1.07 mm and 0.51 mm diameter. No severe splits were indicated at any size. However, it was found that the surface condition which developed during drawing at the higher temperatures could not be removed by practical cleaning methods.

Another section of a rod that had the  $1500^{\circ}\text{C}$  anneal was then drawn at a preheat temperature of  $500^{\circ}\text{C}$ . Split analysis at  $\sim 1.0$  mm diameter revealed that one end had a high density of splits, but no splits on the other end. A second split trace, made at 0.51 mm diameter, showed that no splits had formed in the ductile section of the wire. Furthermore, the wire could be cleaned in the usual manner.

### 2. Production and Evaluation of 0.51 mm Diameter B-88 Wire Under Task II

Based on the optimization studies discussed previously, the fabrication schedule reported in Table XIV was employed to produce  $\sim 2,389$  meters of 0.51 mm diameter B-88 wire. The most important difference between the Task II production schedule and the production schedule employed in Task I was the increase in the preheat temperature from  $400^{\circ}\text{C}$  (Task I) to  $500^{\circ}\text{C}$  (Task II).

Split tests were made on a number of spools of Task II production wire and revealed that the average number of splits was greatly reduced (Fig. 16). In Fig. 16, splits are indicated by a sharp deflection of the pen on the strip chart recorder. The magnitude of the deflection of the pen is proportional to the depth of the split (up to a split depth equal to the wire radius). The length of the split and the length of wire is determined by ratio of the speed of the wire through the test head

Table XIV

Fabrication Schedule for Producing 0.51 mm Diameter  
B-88 Wire Under Task II

<u>Step No.</u>	<u>Operation</u>
I	Swaged* from 8.61 mm to 3.66 mm diameter (clad diameter) from a preheat temperature of $\sim 1100^{\circ}\text{C}$ (10% to 20% R.A. per pass).
II	Swaged to 2.39 mm diameter (10%-20% R.A. per pass (from a preheat temperature of $\sim 650^{\circ}\text{C}$ ).
III	Annealed clad rods for 5 seconds at $\sim 1600^{\circ}\text{C}$ then chemically removed cladding ( $\sim 1.78$ mm core diameter).
IV	Removed 0.10 to 0.20 mm from rod diameter by acid cleaning, then oxidized rods for 4 minutes at $450^{\circ}\text{C}$ .
V	Drawn to $\sim 0.51$ mm diameter (10% to 20% R.A. per pass) from a preheat temperature of $\sim 500^{\circ}\text{C}$ ** and die temperature of $\sim 400^{\circ}\text{C}$ . Cargyl used as lubricant.

\* Hand Swaged Two Passes.

\*\* Rods Dynamically Heated While Drawing Through a 1.2 Meter Electric Furnace at Speeds of 1.2-5.5 Meters/Min.

to the speed of the recorder. Thus, at a typical wire speed of  $\sim 19.2$  m/min and a chart speed of 6.0 cm/min, 2.5 cm of chart is equivalent to 8.0 meters of wire.

Except for the stress-rupture tests reported previously (III, B, 1), the only other evaluation made of mechanical properties were four tensile tests made at  $1316^{\circ}\text{C}$ . The average value of the 0.2% yield strength was  $83.7 \text{ MN/M}^2$  ( $\pm \sim 10.5 \text{ MN/M}^2$ ) and an average ultimate strength of  $116 \text{ MN/M}^2$  ( $\pm \sim 10.5 \text{ MN/M}^2$ ). The elongation was increased considerably but could only be estimated from one test in which an  $\sim 1.28$  mm gage was chemically introduced in the test specimen, resulting in an "apparent" total elongation of 138%.

### 3. Optimization of the Fabrication Schedule for W-Hf-C

The material yield of W-Hf-C wire produced in Task I was less than 10% of the starting material. Therefore, the optimization of the fabrication schedule for producing 0.38 mm diameter wire was concerned entirely with improving the material yield of this alloy. The heaviest material losses were incurred in swaging to 2.54 mm diameter, therefore, a study was made of the effect of in-process anneals, preheat temperatures and the use of cladding in swaging to 2.54 mm diameter.

#### a. Use of Cladding in Swaging W-Hf-C Extrusion Bars

Initially, it was attempted to swage as-received molybdenum clad extrusion bars to a smaller size for re-cladding so as to have the benefit of the original cladding during swaging. Two sections of extrusion bars were swaged from a starting size of  $\sim 2.8$  cm diameter to  $\sim 1.78$  cm diameter (clad dimensions) from preheat temperature of  $\sim 1850^{\circ}\text{C}$  (initial) decreasing to  $\sim 1700^{\circ}\text{C}$  (final) with 10%-20% R.A. per pass. At this size, the cladding was removed for re-cladding the W-Hf-C bar ( $\sim 1.52$  cm core diameter) in 1.65 cm I.D. molybdenum tubing. However, the W-Hf-C bars were found to be split so severely along their entire length that they had to be scrapped. End sections were then cut from two as-received bars, and it was found that center cracks (cracks not extending to the surface of the cross section) were present on the ends of both bars. Therefore, it was decided to remove the cladding from all bars before swaging so as to distinguish between cracks which were developed because of swaging conditions and cracks already present in the as-extruded bars.

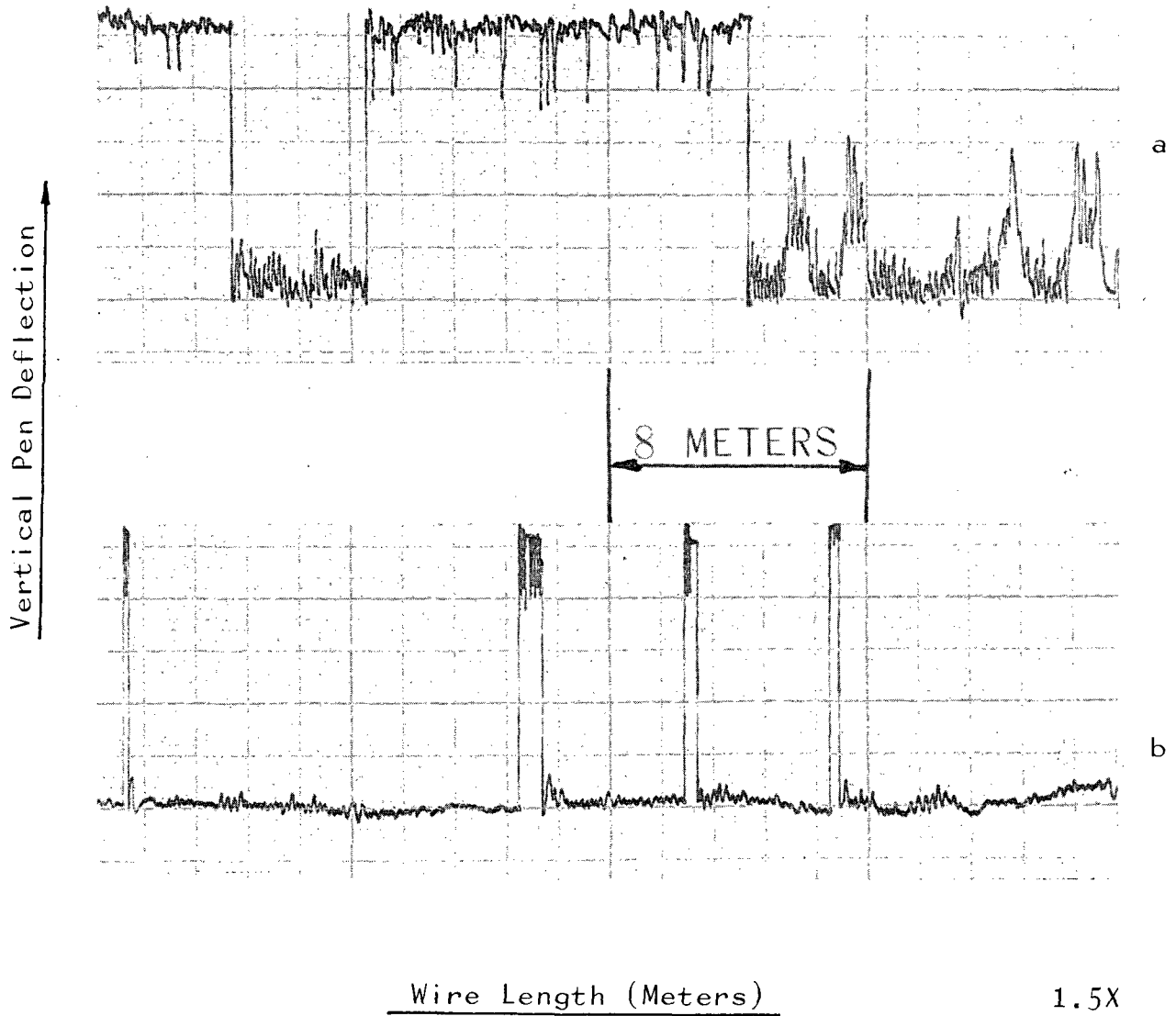


Fig. 16 Reproductions of Strip Chart Recordings Indicating Splits (Vertical Pen Deflections) Typical of B-88 Wire Produced Under: a. Task I and b. Task II.

All of the bars were found to have center cracks on at least one end, and some of the bars had cracks extending from the surface to the center of transverse cross sections\*. The various conditions observed on the longitudinal surface of the extrusion bars after removal of the cladding are depicted in Fig. 17. Characteristically, all of the extrusion bars had a heavily striated surface as shown in the close-up of Fig. 17a. Also, the nose end was generally larger than the tail end and in some cases locally "gouged" areas were present such as shown in the close-up of Fig. 17b. The surface striations were removed by immersing the bars in a fused sodium nitrate salt bath, however, several swaging passes ( $\sim 10\%$  R.A. per pass) were usually required before the bar diameter was uniform along its entire length.

Two fabrication schedules were employed in the production of W-Hf-C wire in Task II. The first schedule required re-cladding the extrusion bars after swaging to  $\sim 1.60$  cm diameter. The second schedule involved the fabrication of wire without re-cladding the extrusion bars for swaging.

#### b. Effect of Preheat Temperature for Swaging

Two extrusion bars were swaged to  $\sim 1.78$  cm diameter (10% to 20% R.A. per pass) from a preheat temperature of  $\sim 1830^\circ\text{C}$  to determine if increasing the swaging temperature would eliminate the severe longitudinal cracks. Both bars split severely at  $\sim 1.78$  cm diameter and were scrapped. Thus, increasing the swaging temperature to about the maximum possible did not in itself eliminate the problem of splitting.

#### c. Effect of In-Process Anneals

As previously mentioned, annealing for 1/2 hour at  $2000^\circ\text{C}$  reduces the hardness of W-Hf-C to  $\sim 485$  VHN without causing recrystallization. A section of an as-extruded bar was annealed for 1/2 hour and swaged one pass (0% up to  $\sim 20\%$  R.A. along the total length of the bar because of the variation in starting size) from a preheat temperature of  $\sim 1830^\circ\text{C}$ . A hardness test taken from the nose end (larger end) of the bar revealed that the hardness had increased to  $\sim 560$  VHN after one

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\* Heavy electropolishing was required to reveal cracks.

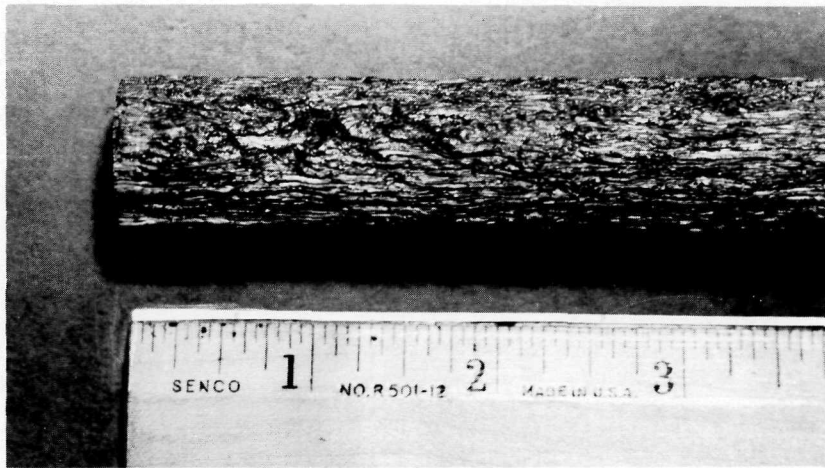
swaging pass. Therefore, the bars were annealed for 1/2 hour at 2000°C before and after each pass when swaging to  $\sim 1.60$  cm diameter (reclad size) or when swaging bars without cladding to  $\sim 1.16$  cm diameter. By eliminating the effect of work hardening (annealing after each swaging pass) at heavy bar sizes, it was possible to swage the extrusion bars to  $\sim 2.54$  mm diameter without developing severe longitudinal splits (even though center cracks were present throughout most bar sections).

#### 4. Production and Evaluation of 0.38 mm Diameter W-Hf-C Wire Under Task II

As stated previously, two fabrication schedules were employed in the production of 0.38 mm diameter W-Hf-C wire in Task II: (1) reclad at  $\sim 1.60$  cm diameter or (2) swage to 2.54 mm diameter without recladding. The latter fabrication schedule is preferred, because the swaged bar sections in most cases were longer than the only molybdenum tubing available for recladding (2.54 cm O.D. x 1.65 cm I.D. x 76.5 cm length), making it necessary to crop the ends of the bars before recladding. As a result, the material yield of bars swaged with and without recladding was about the same, but the labor required to produce the wire was greater for the reclad bars.

A total of 2,994 meters of 0.39 mm diameter wire was produced and delivered to NASA - Lewis in a straightened and cleaned condition from three different extrusion bars employing the fabrication schedules reported in Tables XV and XVI. The overall material efficiency for the three bars was  $\sim 48\%$  material yield and ranged from  $\sim 43\%$  to  $\sim 57\%$ . The highest yield was obtained on a bar section which was swaged without recladding.

Two tensile tests were made at 1316°C on the 0.39 mm diameter W-Hf-C production wire in Task II. The average value of the 0.2% yield strength was 542 MN/M<sup>2</sup> ( $\pm 0$ ), and the average value of the ultimate strength was 1082 MN/M<sup>2</sup> ( $\pm \sim 14$  MN/M<sup>2</sup>). The elongation was estimated to be  $\sim 2.1\%$  based on an assumed four inch gage length.



(a)



(b)

Fig. 17 Photographs Representative of the Surface Conditions Observed on W-Hf-C Extrusion Bars After Removal of the Molybdenum Cladding: (a) Heavily Striated and (b) Locally Gouged Area with an Enlarged Nose End.

Table XV

Fabrication Schedule for Production of 0.38 mm Diameter

W-Hf-C Wire from Reclad Extrusion Bars (Task II)

Step No.

- I Removed cladding from as-received extrusion bars ( $\sim 22.9$  mm core diameter) and then removed  $\sim 2.0$  mm ( $\sim 15\%$  by weight) from the diameter of the bar by immersion in a fused salt bath.
- II Swaged\* to  $\sim 16.0$  mm diameter (15% to 22% R.A. per pass) from a preheat temperature of  $\sim 1830^{\circ}\text{C}$ . Bars were annealed  $\frac{1}{2}$  hour at  $2200^{\circ}\text{C}$  before and after each swaging pass.
- III Bars were reclad in 16.51 mm I.D. x 25.4 mm O.D. molybdenum tubing and then swaged to  $\sim 5.08$  mm diameter (20% to 33% R.A. per pass) from preheat temperatures of  $1750^{\circ}\text{C}$  (initial to  $\sim 1350^{\circ}\text{C}$  (final)\*\*).
- IV Cladding removed ( $\sim 4.19$  mm core diameter) and the rod was annealed for  $\sim 2$  minutes at  $\sim 1950^{\circ}\text{C}$  then swaged to  $\sim 2.54$  mm diameter (25% to 35% R.A. per pass) from preheat temperatures of  $\sim 1300^{\circ}\text{C}$  (initial) to  $\sim 1200^{\circ}\text{C}$  (final).
- V Drawn to 0.39 mm diameter (10% to 36% R.A. per pass) from preheat temperatures of  $\sim 950^{\circ}\text{C}$  (initial) to  $\sim 700^{\circ}\text{C}$  (final). Rods were preheated in a 0.61-0.91 m gas-air burner at speeds of 5.49-13.72 m/min. Cargyl was used as a lubricant.

\* Hand swaged at sizes above  $\sim 11.86$  mm diameter.

\*\* The molybdenum cladding failed prematurely on one bar section at  $\sim 10.08$  mm diameter. The cladding was removed and the W-Hf-C rod ( $\sim 8.13$  mm diameter) was annealed 2 minutes at  $\sim 1950^{\circ}\text{C}$  then swaged to 4.19 mm diameter (25% R.A. to 35% R.A. per pass) from preheat temperatures of  $\sim 1400^{\circ}\text{C}$  (initial) to  $\sim 1200^{\circ}\text{C}$  (final). The remainder of the fabrication schedule for this rod was the same as given in Steps IV and V, above.

Table XVI

Fabrication Schedule for Production of 0.38 mm Diameter  
W-Hf-C Wire from Unclad Extrusion Bars (Task II)

Step No.

- |     |   |
|-----|---|
| I   | Removed cladding from as-received extrusion bars ( $\sim 22.86$ mm core diameter) and then removed $\sim 2.0$ mm ( $\sim 15\%$ by weight) from the diameter of the bar by immersion in a fused salt bath.   |
| II  | Swaged to $\sim 14.10$ mm diameter (10% to 20% R.A. per pass) from a preheat temperature of $\sim 1830^{\circ}\text{C}$ . Bars were annealed $\frac{1}{2}$ hour at $2000^{\circ}\text{C}$ before and after each swaging pass.   |
| III | Swaged* to $\sim 2.54$ mm diameter ( $\sim 20\%$ to 32% R.A. per pass) from preheat temperatures of $\sim 1800^{\circ}\text{C}$ . (initial) to $\sim 1200^{\circ}\text{C}$ (final). Bars were annealed $\frac{1}{2}$ hour at $2000^{\circ}\text{C}$ at 11.56 mm diameter and 2 minutes at $1950^{\circ}\text{C}$ at 5.10 mm diameter. |
| IV  | Drawn to 0.39 mm diameter ( $\sim 10\%$ to 36% R.A. per pass) from preheat temperatures of $\sim 950^{\circ}\text{C}$ (initial) to $\sim 700^{\circ}\text{C}$ (final). Rods were preheated in a 0.61-0.91 m gas-air burner at speeds of 5.49-13.72 m/min. Cargyl was used as a lubricant.   |

\* Hand swaged at sizes above 11.56 mm diameter.

## IV. Discussion of Results

### A. Development of Fabrication Schedules and Production of Wire

In developing a fabrication schedule for producing wire from each of the alloys, the research effort was guided mainly by these considerations: (1) the prior experience that had been acquired elsewhere in the development of the alloy systems, (2) a general awareness of the limitations imposed because of problems intrinsic to the particular base alloy composition, and (3) the specific experience gained from prior research efforts concerned with the development and fabrication of high strength refractory metal alloys<sup>(4)</sup>.

The approaches taken in establishing a fabrication schedule for producing wire from each of the alloys were similar in certain respects, and the steps taken were approximately in the following order:

#### Swaging

Determine a suitable swaging temperature.

Determine whether cladding was required.

Determine if in-process anneals were required.

Determine if surface conditioning was required.

#### Wire Drawing

Determine a suitable drawing temperature.

Determine a suitable lubricant.

Determine if in-process anneals were required.

Determine if surface conditioning was required.

Determine the effect of wire drawing speed and % reduction in area per pass.

In Task I, the evaluation of each step in the fabrication schedule was pursued only to the extent necessary to produce a maximum of 30 meters of wire at the required wire sizes. Similarly, in Task II only specific problem areas could be more

thoroughly evaluated; and therefore, the possible effects of all of the processing steps involved in fabrication of wire could not be fully evaluated. However, in the following, the main problems encountered in developing a fabrication schedule for producing 0.25 mm to 0.51 mm diameter wire from each alloy will be outlined, and the possible causes and solutions (where known) will be discussed.

## 1. ASTAR-811C

This alloy was only investigated in Task 1. The alloy was swaged at room temperature with no difficulty, however, frequent wire breaks occurred in drawing. The breaks were primarily brittle fractures, although the wire in general was very ductile. The reasons for the locally brittle areas were not identified, but the three most likely causes are suspected of being: (1) non-homogeneities in the wire due to impurities (or local segregation) in the starting composition, (2) excessive die friction (poor lubrication), and (3) excessive local contamination when the wire was oxidized to provide a base for the lubricant.

Some evidence is available to support each of these possibilities. The wire chattered badly in the die when the drawing speed was increased or after drawing a number of passes at the same speed (die friction). It was also observed that the wire became generally brittle when the temperature for oxidizing the wire before drawing was increased above 500°C. On the other hand, the microstructure shown in Fig. 5b (showing abrupt changes in the fiber pattern across the entire cross section) would tend to suggest that local changes in composition may have been present in the starting material, since the patterns observed are not typical of homogeneous alloys.

## 2. B-88

The main problems encountered in fabricating this alloy were the following: (1) local embrittlement during wire drawing, (2) removal of the defective surface after swaging, and (3) elimination of splits in the finished wire. The possible causes for the first problem cited are the same as previously discussed for ASTAR-811C (including the fact that the microstructure of the swaged rod had areas suggestive of non-homogeneities in the composition - Fig. 7b). The second problem was solved by chemically cleaning the rods before drawing, however, the method employed involved a somewhat hazardous operation (which was also costly from the standpoint of the labor involved). The third problem

was also satisfactorily reduced in Task II by increasing the drawing temperature from 400°C to 500°C. Drawing at slightly higher temperatures ( $\sim 600^\circ\text{C}$ ) reduced even further the split density, but caused a surface oxide to form which could not be removed by practical production cleaning methods. Therefore, under present wire drawing conditions, the control of temperature is very critical.

### 3. W-Hf-C

The most severe problem in fabricating W-Hf-C was the poor condition of the as-received extrusion bars. Even after the problem of removing the rough surface was solved by cleaning in a fused salt bath, the internal flaws undoubtedly contributed to the difficulty of swaging the bars at large sizes. In addition, the high work hardening rate of the alloy was likewise a factor in the problem of fabricating W-Hf-C. The solution to this problem was to swage the bars at a high swaging temperature ( $\sim 1830^\circ\text{C}$ ), and then stress relieve the bars after each swaging pass until sufficient "cold" working had improved the fabricability of the alloy. The effect of "cold working" on the structure of the alloy was not thoroughly investigated, however, it was shown by ETM that a very fine ( $1.0\ \mu\text{m}$  transverse diameter) highly elongated substructure was present after swaging to  $\sim 0.25$  mm diameter. On the other hand, it is unlikely that the same type of substructure was present in the starting material.

#### B. Mechanical Properties of the Alloys at Wire Sizes of 0.25 mm to 0.51 mm Diameter

All three of the materials investigated are precipitation hardening type alloys. However, the role played by the precipitates in creep-rupture strengthening is different in the cases of ASTAR-811C and B-88, as compared to the case of W-Hf-C. The first two alloys had higher creep-rupture strength properties (but lower tensile properties) in the recrystallized condition\*, than in the as-worked condition. On the other hand, recrystallizing W-Hf-C destroys its high strength properties (5). Thus, it appears that the improved creep-rupture strength of ASTAR-811C and B-88 is due to a

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\* Although ASTAR-811C was not included in Task II, a stress-rupture test was made on a specimen of the alloy after annealing for 1 hour at  $1650^\circ\text{C}$ .

lower mobile dislocation density in the recrystallized condition, whereas W-Hf-C achieves its strength properties by a fine sub-structure which is stabilized by precipitates.

## V. Summary of Results

Fabrication schedules were developed for producing 0.25 mm, 0.38 mm and 0.51 mm (0.010, 0.015 and 0.020 inch) diameter wire from the alloys ASTAR-811C, B-88 and W-Hf-C. The mechanical properties of the wire were evaluated by tensile testing (room temperature, 1093°C and 1204°C) and also by 100 hour stress-rupture tests at 1093°C. From the overall experience gained and fabricating and evaluating the alloys, the following conclusions can be made.

### 1. Fabricability of the Respective Alloys

ASTAR-811C can be fabricated from 0.64 cm (0.25 inch) diameter wrought bar stock to sizes as small as 0.025 cm (0.010 inch) diameter wire at room temperature. The wire produced contained a large number of splits suggesting, therefore, that higher wire drawing temperatures (less than 700°C) would be desirable.

B-88 requires elevated temperatures (700°C to 1100°C) for fabrication into wire. Swaging of 0.64 cm (0.025 inch) diameter bar stock began at 1100°C and required the use of a cladding and in-process recovery anneals for successful wire drawing. Drawing temperatures of 400°C resulted in a large density of splits. The split levels were substantially reduced by drawing at 500°C.

W-Hf-C was extremely difficult to fabricate because of intrinsic brittleness of the starting molybdenum clad material and the poor quality of the as-received extrusion bars. Surface conditioning of the bars was accomplished by immersion in a fused salt bath after the cladding was removed. The initial swaging conditions required high preheat temperatures, approximately 1825°C, and in-process recovery anneals (30 minutes at 2000°C) after each swaging pass to 1.27 cm (0.50 inch diameter, 10 to 20% reduction in area per pass). After the initial swaging passes were accomplished, it was possible to reduce gradually the preheat temperature to approximately

1200°C while swaging to 0.25 cm (0.1 inch) diameter. Subsequently, the alloy was drawn to size by essentially standard techniques employed in commercial tungsten wire fabrication. This alloy is as ductile as commercial tungsten wire after working to 0.25 to 0.51 mm (0.010 to 0.020 inch) diameter.

## 2. Evaluation of Mechanical Properties

The W-Hf-C wire was found to have vastly superior stress-rupture properties (3 to 4 times stronger) at 1093°C in comparison to B-88 or ASTAR-811C wire. This strength advantage is realized on an absolute as well as a relative (strength/density) basis. On the other hand, the relative room temperature tensile strength of W-Hf-C is poorer than that of the other two alloys. However, its tensile strength is slightly better at elevated temperatures (1093°C and 1204°C) when compared on the same relative basis. ASTAR-811C is marginally better than B-88 in stress-rupture strength at 1093°C when compared on a relative basis. On the contrary, B-88 has marginally better relative tensile properties from room temperature to 1204°C than ASTAR-811C.

## VI. Recommendations for Future Work

### ASTAR-811C and B-88

The question of homogenizing the composition of arc-cast ASTAR-811C and B-88 needs further investigation. Also, further studies of wire drawing conditions (lubrication and drawing temperatures) will be needed to reduce the material loss (and cost) in producing wire from these alloys.

### W-Hf-C

An investigation of the extrusion parameters employed in producing this alloy is needed. An alternative approach might be to explore the production of this alloy by powder metallurgy techniques.

## APPENDIX A

### Summary of Vendor's\* Report on Production of Starting Materials

#### 1. ASTAR-811C

##### a. Ingot Consolidation

Double A.C. Vacuum Arc Melted

##### b. Billet Extrusion

3" diameter ingots machined to 6.90 cm diameter and canned in mild steel. Extruded at WPAFB; die size 2.84 cm diameter, 60 conical, ratio 6:8:1, facing  $ZrO_2$ , lubricant-billet-0010 glass, 7.60 mm thick-container and die-Fiske 604D, container temperature-260°C, billet extrusion temperature-1260°C, argon atmosphere, 1 hour soak.

##### c. Bar Swaging

1. Extruded bars cropped, chemically cleaned after cladding removed.

2. Swage straightened from 2.96 cm diameter to 2.69 cm diameter, 1204°C preheat.

3. Centerless ground to 2.55 cm diameter, vacuum annealed 1 hour at 1620°C.

4. Swaged from preheat temperatures of 540°C (initial) to R.T. (1.09 cm diameter) with gradually decreasing temperatures. Rods pickled and annealed 1 hour at 1620°C, 1370°C during swaging to 1.09 cm diameter continued swaging to 6.85 mm diameter at room temperature. Annealed 1 hour at 1370°C and 1315°C during swaging. Centerless ground to 6.70 mm diameter.

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\* Westinghouse Astronuclear Laboratories, Pittsburgh, Pennsylvania.

2. B-88\*

- a. Ingot Consolidation  
Double A.C. Vacuum Arc Melted
- b. Billet Extrusion  
7.25 cm diameter ingots machined to 6.85 cm diameter and canned in molybdenum. Extruded at WPAFB; die size 3.18 cm diameter, 90° conical, 6:1 ratio, facing ZrO<sub>2</sub>, lubricant-billet 7052 glass, 7.60 mm thick-container and die-Fiske 604D, container temperature 260°C, billet extrusion temperature-1820°C, 25 minute soak, induction heated under argon gas.
- c. Bar Swaging
  1. Extruded bars nose and tail cropped, swaged to ~3.18 cm to 2.10 cm diameter. Core size ~1.75 cm diameter.
  2. Clad removed and re-clad in 2.54 cm O.D. x 3.18 mm wall, evacuate and seal.
  3. Swage at 1200°C to a core diameter of 1.30 cm diameter, remove clad and pickle in 50% HCl.
  4. Wrap in Ta foil and anneal 1 hour at 1600°C in vacuum (~1 x 10<sup>-5</sup> torr).
  5. Re-clad in mild steel (1.90 cm O.D. x 2.54 mm wall), evacuate and seal.
  6. Swage at 1200°C to a core diameter of 9.70 mm diameter, then repeat steps 3 and 4.
  7. Re-clad in mild steel (1.27 cm O.D. x 1.52 mm wall) and swage to core diameter of 7.36 mm.
  8. Centerless grind to 6.25 mm diameter.

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\* All billets extruded at WPAFB except one billet extruded at NASA - Lewis.

### 3. W-Hf-C

#### a. Ingot Consolidation

Single A.C. vacuum arc melted (electrodes were made up of pressed and sintered tungsten, hafnium wire and carbon cloth--in string form).

#### b. Billet Extrusion\*

6.35 cm diameter billets ground to 6.10 cm diameter and canned in molybdenum (7.35 cm diameter x 15.2 cm long with a 60° nose end shape). With the exception of one billet, container and die lubricant was Fiske 604D, billets were bare. Die size 2.77 cm and 2.75 cm for 7.95:1 and 8:1 extrusion ratios, respectively, 90° conical with ZrO<sub>2</sub> facing. Billet extrusion temperatures varied from 2065°C to 2200°C.

## APPENDIX B

### Summary of Preparation of Specimens for Electron Transmission Microscopy

#### 1. B-88 ( $\sim 1.78$ mm Diameter)

Wire was stress relieved for 30 minutes at  $1204^{\circ}\text{C}$  (to avoid splitting during thinning) in a clear plastic, and ground in longitudinal direction from both sides of mount until  $\sim 0.25$  mm thick, then ground to a final thickness of  $\sim 0.13$  mm on 240 to 600 grit water-cooled silicon carbide paper.

Specimens (3.17 mm long x width of wire section) punched, and then mounted in a PVC holder, then thinned in a dual jet electropolishing apparatus.

Specimens were thinned in a solution consisting of 5%  $\text{H}_2\text{SO}_4$ , 2.5% HF (50%) and 92.5% Methanol. Solution was maintained at  $-74^{\circ}\text{C}$  by immersing the electrolyte container in a dry ice and methanol bath. The current density was  $\sim 200$  ma/cm<sup>2</sup> at  $\sim 20$  to 40 volts during thinning (5 to 10 minutes).

#### 2. ASTAR-811C

Preparation of specimens for thinning was the same as reported for B-88. The specimens were also thinned in an apparatus similar to that used for B-88. The electrolyte consisted of 85%  $\text{H}_2\text{SO}_4$  (conc.) and 15% HF (48%). The thinning bath was maintained at  $60^{\circ}\text{C}$ , while thinning at a current density of 250 to 300 ma/cm<sup>2</sup> at 10 to 20 volts (2-4 minutes).

#### 3. W-Hf-C

##### a. Approximately 2.54 mm Diameter Swaged Rod

Specimens pre-annealed for 72 hours at  $1100^{\circ}\text{C}$  prior to mounting in clear plastic. Polished longitudinally on alternating sides with 1/0 polishing paper to  $\sim 0.10$  mm thick ribbon. Specimens about 3.17 mm long were punched out and placed in a PVC holder and thinned in a dual jet electropolishing apparatus. The electrolyte consisted of 3% NaOH at room temperature. The current density for thinning was 320-380 ma/cm<sup>2</sup> (3-5 minutes).

b. Approximately 0.51 mm Diameter Wire

Specimens were annealed for 50 hours at 1230°C before mounting in an opaque epoxy resin (Armstrong A-12 resin with Armstrong P-32 coloring reagent added, 1:1). The wires were polished on alternating sides to 0.10 mm to 0.18 mm thick with 1/0 polishing paper. The specimens were punched out and placed in the same apparatus for thinning as described above. The electrolyte consisted of 3% NaOH at room temperature. The current density for thinning was approximately 400 ma/cm<sup>2</sup> (3-5 minutes).

## APPENDIX C

### Continuous Vacuum Annealing of 0.02" Diameter

#### B-88 Wire at Temperatures Between 1800°C and 1925°C

The essential features of the vacuum furnace (see Fig. C1) consisted of a 30.5 cm diameter x 76.2 cm high bell jar mounted on a base plate connected to a 10.15 cm oil diffusion pump. A vertical shaft enters the vacuum chamber through a seal in the base plate. The shaft can be raised and lowered, and also, it can be rotated in the counterclockwise direction. The shaft drives a 10.15 cm diameter capstan which acts as the take-up for the wire. The wire is guided from a brass feed reel (equipped with a spring-back tension device) through a 30.5 cm long x 2.54 cm O.D. x 1.91 cm I.D. molybdenum furnace muffle. The muffle is induction heated by a vertical 13.7 turn induction coil, 26.7 cm long x 3.80 cm I.D. carrying 10 KHz current.

In operation, the (cleaned and straightened) wire was threaded through the furnace, and the unit then pumped to a vacuum pressure of  $\sim 2 \times 10^{-5}$  torr. The muffle was heated to temperature (1800°C to 1925°C, TT) in  $\sim 40$  minutes, at which time the vacuum read  $\sim 10^{-5}$  torr. The take-up drum was then rotated at a rpm corresponding to  $\sim 1.92$  meters/minute wire speed. During the run, the vacuum worsened to  $\sim 4 \times 10^{-4}$  torr, except near the end of the run, in which the pressure increased so high that the gage was turned off (poor cleaning of ends).

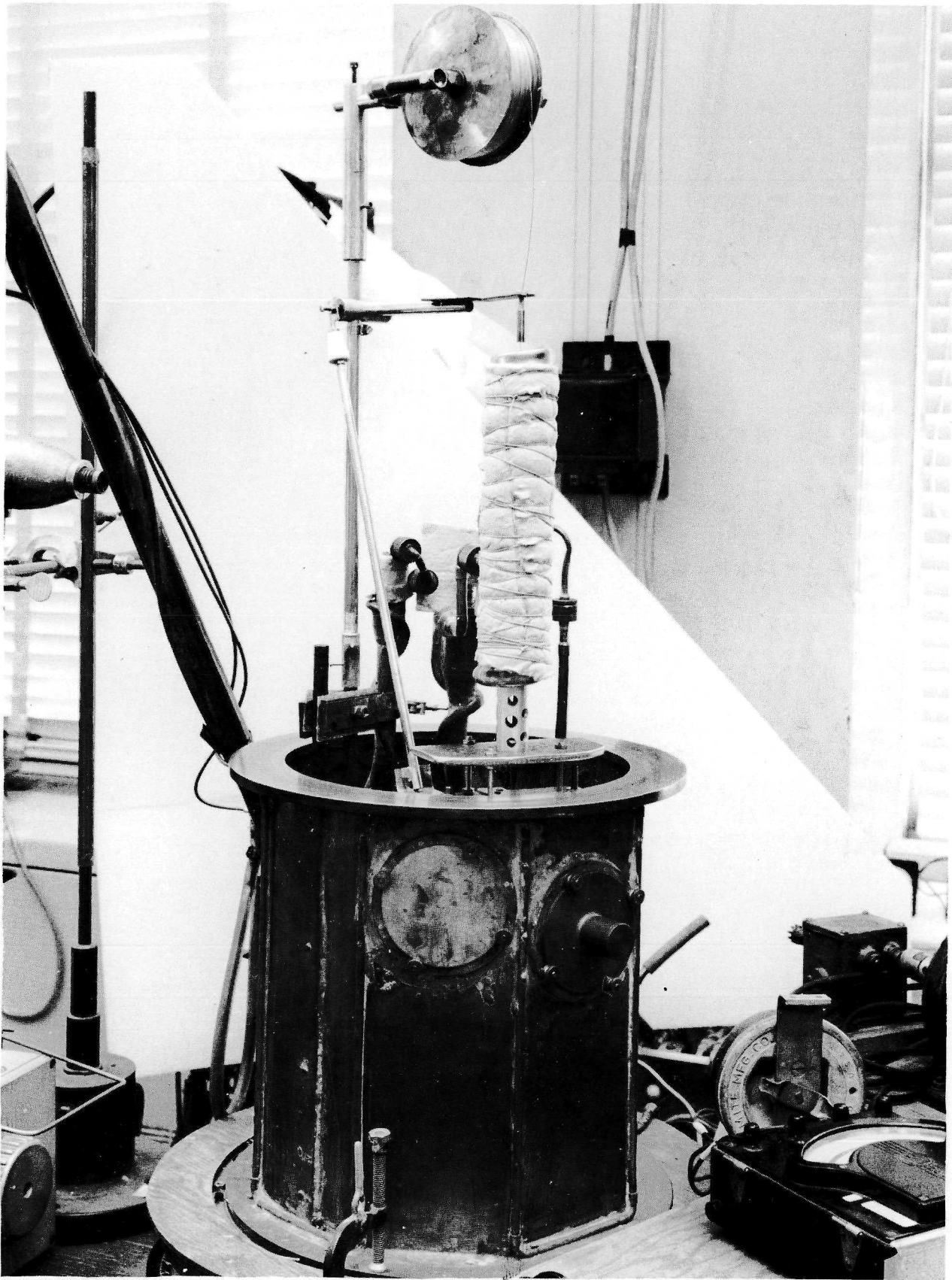


Fig. C1 Photograph of Vacuum Furnace Used for Continuously Annealing 0.51 mm Diameter B-88 Wire.

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