EVALUATION OF HIGH STRENGTH TUNGSTEN BASE ALLOYS

Charles R. Cook
TRW Inc
Equipment Laboratories

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AIR FORCE SYSTEMS COMMAND
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FOREWORD

This Summary Technical Report covers the work accomplished in the period from July 1, 1963 to September 30, 1965.

This contract AF 33(657)-11239 with TRW Inc., was initiated under Physical Metallurgy Branch Project Nr 7351, "Research on Refractory Alloys of Tungsten, Tantalum, Molybdenum, and Columbium". The program is under the technical direction of Mr. James T. Gow of the Physical Metallurgy Branch, Metals and Ceramics Division, A.F. Materials Laboratory, Wright-Patterson Air Force Base, Ohio.

F.N. Lake of the Materials and Processes Department, TRW Equipment Laboratories, was engineer in charge, L.J. Stefan was the engineer responsible for the execution of the program and C.R. Smeal contributed to the final evaluation of the program and report preparation. C.R. Cook, Research Supervisor, was responsible for program management at TRW.

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This technical report has been reviewed and is approved.

I. Perlmutter
Chief, Physical Metallurgy Branch
Metals and Ceramics Division
AF Materials Laboratory

ABSTRACT

A deformation study and evaluation of high strength tungsten-base alloys was accomplished. The two alloys studied included the solid solution strengthened 68% W-20% Ta-12% Mo alloy and the dispersed-phase strengthened W-12% Cb-0.29% V-0.12% Zr-0.07% C alloy. The vacuum melting plus centrifugal casting technique was used for the consolidation of the solid solution alloy. A new type of ingot mold was developed to cast 3-inch diameter ingots. High temperature extrusion was successfully accomplished to provide 68% W-20% Ta-12% Mo bar for evaluation. Studies of upset and side forging, rolling and swaging of the solid solution alloy were made. Upset forging was the only successful technique for secondary working. The extruded bar was used to determine recrystallization behavior, tensile and creep rupture properties. The one-hour recrystallization temperature for the 68% W-20% Ta-12% Mo bar was 3300°F. The 3000°F, 3500°F and 4000°F tensile strengths of the recrystallized bar were approximately three times those of unalloyed tungsten.

The W-12% Cb-(V, Zr, C) alloy was consolidated by AC arc melting due to arc instability in DC melting. The three-inch diameter ingots could not be produced without cracking. High temperature extrusion produced sufficient sound bar to evaluate tensile properties. This dispersed phase strengthened alloy exhibited the highest strengths at elevated temperatures known to have been reported to date for refractory metals.

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I INTRODUCTION

General review of the history of refractory metal technology reveals continuing recognition by the Department of Defense of the increasing needs for superior high temperature refractory alloys in both bar and sheet form for aerospace design concepts.

Recent programs sponsored by the Air Force Materials Laboratory, Research and Technology Division, have shown considerable promise for extending the service temperature of many metallic systems.

This program was initiated to study several promising refractory metal alloy systems more thoroughly and to provide scale-up verification for the better alloys. The study was planned to include the evaluation of process refinements leading to bar and sheet products of high strength alloys developed earlier in the Air Force sponsored program[1,2].

The promising compositions selected for study comprised solid solution strengthened ternary W-Ta-Mo and W-Ta-Cb alloys and the dispersed phase strengthened W-12Cb-.29V-.12Zr-.07C alloy. The alloys exhibited extremely high strengths at high temperatures with reasonable ductility as shown in Table I. These single tests of as-extruded bars from small arc melted ingots represented the highest strengths reported to date for refractory metals.

There are four major program areas involved in this study: consolidation of the high strength refractory compositions; primary breakdown of the castings by extrusion; secondary deformation studies leading to bar and sheet products; and metallurgical evaluation of the alloys at all stages to characterize them and to resolve the problem areas.

A major problem encountered early in the program in the attempts to arc melt larger ingots necessitated a modification in the plan. The revised program provided for a study of two alloys aimed at developing maximum useful high temperature strengths. hese studies were divided into the following phases:

Phase A: Evaluation of the solid solution strengthened 68W-20Ta-12Mo alloy.

Phase B: Evaluation of the dispersed phase strengthened W-12Cb-0.29V-0.12Zr-0.07C alloy.

The first year's effort was summarized in ML TDR 64-271[3] in September 1964. This report is a summary of all the results of the program including data and illustrations from the above report as appropriate.

TABLE I

REFRACTORY METAL ALLOYS SELECTED FOR EVALUATION

Nominal Composition (Wt.%)	3000°F Ultimate Tensile Strength (a) (psi)	3000°F Elongation (a) %
68W-20Ta-12Mo	67,000	16
20W-68Ta-12Mo	55,000	24
68W-20Ta-12Cb	48,000 (b)	4 (b)
44W-44Ta-12Cb	54,000	7
W-12Cb-.29V-.12Zr-.07C	57,000 (at 3500°F)	43

(a) Data reproduced from Reference 2, representative of bar stock as extruded from ingots consumable electrode vacuum arc cast to the approximate nominal compositions shown above.

(b) Fractured at the fillet portion of the specimen gage.

II SUMMARY

A development study has been conducted with selected solid solution and dispersed phase strengthened tungsten base alloys. The two promising high strength alloys selected for this program were 68% W-20% Ta-12% Mo and W-12% Cb-0.29% V-0.12% Zr-0.07% C. The requirements of the program were to determine the scale-up possibilities, the processing conditions for producing bar and sheet products and to characterize these alloys by mechanical property evaluation of the finished forms.

The program has established that the scale-up from the 1 1/4" diameter ingot size to 3" diameter (and larger) ingots of these compositions is a major problem. Modifications in centrifugal casting techniques were developed to consolidate the 68W-20Ta-12Mo composition. Difficulty in maintaining a stable arc for DC arc melting of the W-12Cb-0.29V-0.12Zr-0.07C for centrifugal casting prevented the application of the technique for this alloy. AC vacuum arc melting was then employed for this alloy.

High temperature extrusion was successfully accomplished with the 68W-20Ta-12Mo alloy. A total of ten billets of this composition were extruded to round bar at reduction ratios of 4.5 - 6.4 to 1 within a temperature range of 3500°F to 3960°F. Two extrusions of the W-12Cb dispersed phase alloy were made at reduction ratios of 3.2 and 4.0 to 1 at temperatures of 3950°F and 4150°F, respectively.

Secondary working studies including upset forging, side forging, rolling and swaging were made with the 68W-20Ta-12Mo alloy extruded bar. These studies indicated that the material could be upset to approximately 50% reduction in height in both the wrought and recrystallized conditions. The side forging, rolling, and swaging studies indicated that the alloy could not be successfully deformed to make bar or sheet products by these techniques.

Evaluation of the extruded bar showed that the one-hour recrystallization temperature of the 68W-20Ta-12Mo alloy was 3300°F and for the W-12Cb-0.29V-0.12Zr-0.07C alloy was over 4100°F. Tensile tests of extruded 68W-20Ta-12Mo bar were made in both wrought and recrystallized conditions. The W-12Cb dispersed phase alloy was tested in tension in the as-extruded, partially recrystallized condition. Both alloys exhibit high strengths, e.g., yield strengths at 3000°F of 43,000 psi and 65,000 psi for the solid solution and dispersed phase alloys, respectively.

III Solid Solution Alloy - 68W-20Ta-12Mo

A. Consolidation

1. Melting

The initial work on the ternary alloy systems W-Ta-Mo and W-Ta-Cb selected for study in this program was accomplished with small ingots melted in a unique furnace[2]. The work in the current program has shown that the uncooled molybdenum crucible mold with the tungsten sheet liner in which the 1 1/4" diameter ingots were melted in the earlier program was a wise choice. The slow cooling rate afforded by allowing the ingot to cool within the solid molybdenum mold, which was well above incandescent temperature during the melt cycle, along with the small ingot size prevented cracking due to stresses generated during solidification and cooling. However, it was determined from discussions with several experienced refractory metal melting sources that construction and operation of a large furnace for melting the ingots required in this program would constitute too great an undertaking for the current effort.

In this program, the initial melting was accomplished in a consumable electrode vacuum arc furnace with a water cooled copper crucible. The ingots of the four compositions selected for study from the W-Ta-Mo and W-Ta-Cb systems (shown in Table I) were cracked severely after cooling. In addition, several of the ingots showed a high degree of porosity. The cracking was concluded to be a result of inability of the alloys to withstand the stresses resulting from contraction during cooling through the lower temperatures, i.e., from about 1500°F to ambient.

After reappraisal of potential melting techniques, the centrifugal casting technique was concluded to offer the best possibility for obtaining homogeneous, dense, defect-free ingots. Oregon Metallurgical Corporation (Oremet) had developed a furnace for centrifugally casting refractory metals and had demonstrated this capability in earlier programs with the 85 tungsten-15 molybdenum alloy. With the recognition that more development effort was required for the consolidation phase than had been anticipated, the decision was made to concentrate the effort on the highest strength solid solution alloy - 68W-20Ta-12Mo.

The centrifugal casting process was applied initially with a single ingot mold of solid graphite. The process consisted essentially of preparing an electrode by melting a cluster of high purity starting materials in an AC vacuum consumable electrode furnace. After machining the outer surface of the ingot so produced, it became the electrode for consumable DC vacuum arc melting into a tiltable water cooled copper skull crucible from which the alloy was cast into a rotating graphite mold.

The first centrifugal casting attempt failed to produce an ingot. During casting the molten metal prematurely solidified in the funnel and the sprue cavity and thereby failed to enter the mold. This premature solidification resulted from miscalculation of the superheat temperature required to maintain fluidity during pouring. Apparently, the fluidity of the 68W-20Ta-12Mo alloy is less than that of 85W-15Mo alloy, upon which the first superheat estimate

for the experimental alloy was based. Universal Cyclops also reported that 68W-20Ta-12Mo required more power to maintain a molten pool during vacuum arc melting than did unalloyed tungsten.(4)

Several changes were made to obtain a better pour with this alloy. The height of the melting crucible above the supporting trunnion (tilt point) was increased by six inches to allow the crucible to pour directly through the hole in the funnel, rather than on the funnel lip. The inside depth of the six inch diameter crucible was increased by four inches to increase the metal pool volume. This attempt was successful in producing an ingot, identified as ingot 1A. The ingot as-received, with sprue attached, is shown in Figure 1. The melting parameters for this and later heats are shown in Table II.

Due to the low ductility of this alloy at room temperature, the ingot was removed from the sprue by electrical discharge machining in an effort to prevent the formation of cracks during machining and/or the propagation of any cracks that may have existed in the as-cast material. Several cracks and a solidification shrink cavity were visible on the as-sectioned surface, Figure 2.

After removal of the sprue, the ingot was given a 3000°F stress relief anneal for 4 hours to eliminate any stresses that may have resulted from solidification. To minimize the possibility of thermal stress cracking during the anneal, a 4 1/2 hour heating cycle and a 6 hour cooling cycle were employed. The stress relief anneal was performed in a resistance heated vacuum furnace. At no time during the heating cycle was the absolute pressure in the furnace above 1×10^{-5} mm Hg.

After the stress relief anneal, sections were removed from both ends of the ingot for chemical analysis and metallographic examination. At this point it was visually apparent that the ingot was cracked. Ultrasonic examination revealed that the cracks extended the total length of the ingot.

Because no extrusion data were available for this alloy it was considered advisable to extrude the ingot to determine pressure, temperature, and extrusion ratio relationships for a guide in processing subsequent ingots To accomplish this the ingot was machined and ground to its maximum size, 2.5" diameter by 4.25" long. Photographs of the finish ground extrusion billet, both with and without die penetrant, reveal the cracks encountered (Figure 3).

After detailed discussion of the procedures used for the initial casting attempts, a modified technique was developed with Oremet for the second casting of the 68W-20Ta-12Mo alloy. A 9" diameter copper crucible was used to increase the system volume so that two billets could be poured in a single heat. The single solid graphite mold was replaced with two thin-wall graphite cylinders, one 1/2" and the other 3/4" thick. These mold modifications were intended to result in a much slower, more uniform cooling rate and thereby prevent the cracking encountered in the previous attempt. Further, they allowed two ingots to be cast during the pour. A sketch of the new mold is shown in Figure 4. The thin mold graphite liners are less conductive than the solid graphite while the vacuum and reflectors minimize the cooling rates; the two liner thicknesses were used for comparison of results. A typical

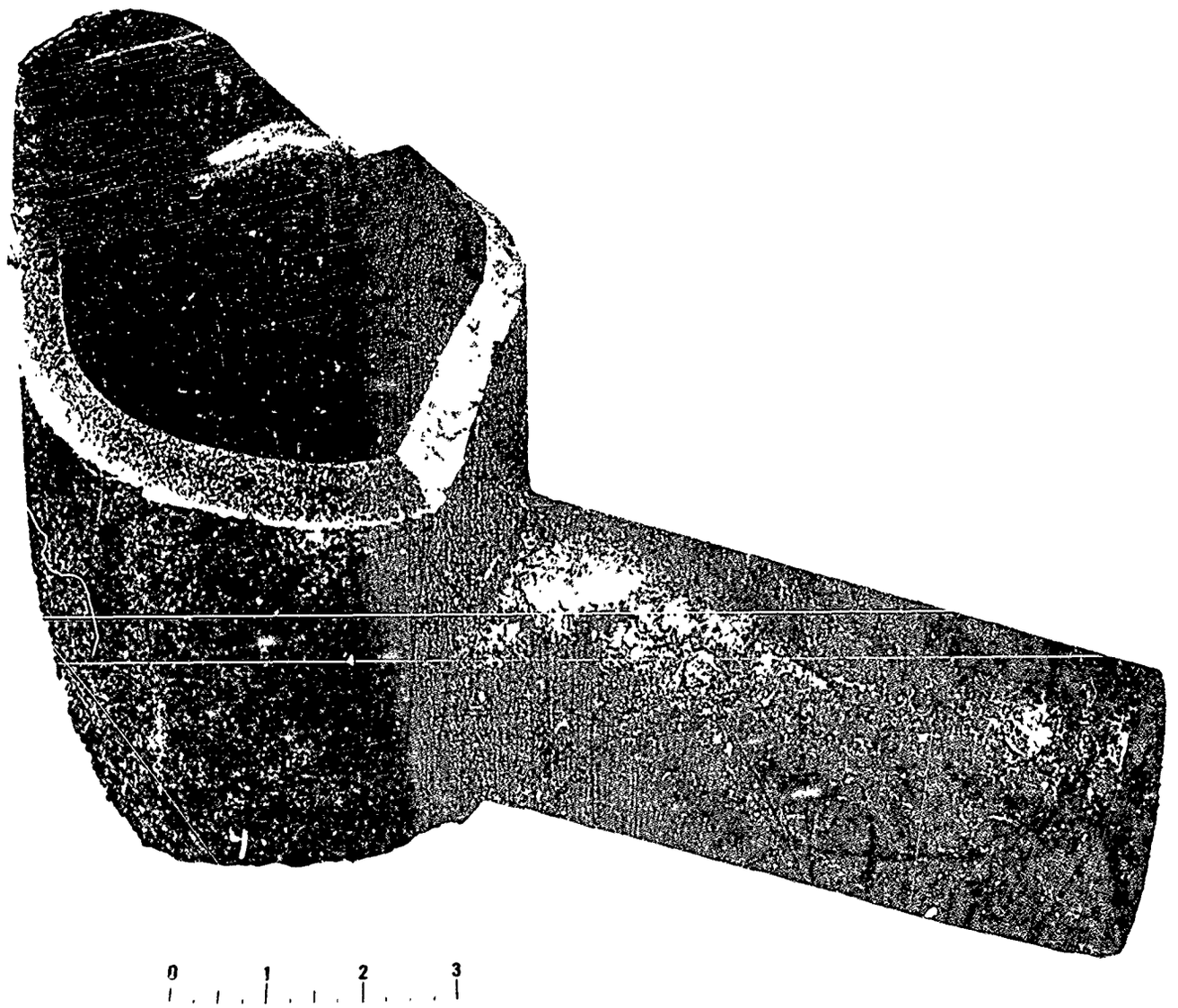


Figure 1. Ingot and Sprue of 68W-20Ta-12Mo Alloy Ingot 1A, as Cast by Oregon Metallurgical Corp.

TABLE II

MELTING PARAMETERS FOR CENTRIFUGALLY CAST 68W-20Ta-12Mo INGOTS

Melt No.	Billet Nos.	Melt Time Minutes	Voltage Avg.	Amperage Avg.	Furnace Pressure Microns	Mold Thickness Inches	Casting Weight lbs.
1	-	9.5	29	19,000	45	Solid	mis-run
2	1A	9.5	29	19,000	20	Solid	77.6
3	2A,3A	14.5	33	28,000	3000	1/2&3/4	68.6
4	4A,5A	12.0	30	28,000	20	1/2	85.3
5*	6A,7A	6.5	34	28,000	40	1/2	111.0
6	8A,9A	5.7	36	28,000	30	1/2	99.8
7	10A,11A	8.0	34	29,000	40	1/2&5/16	104.5

* This melt was processed twice because the sprue was not completely filled on the first pour. It was then crushed and remelted and cast as shown.

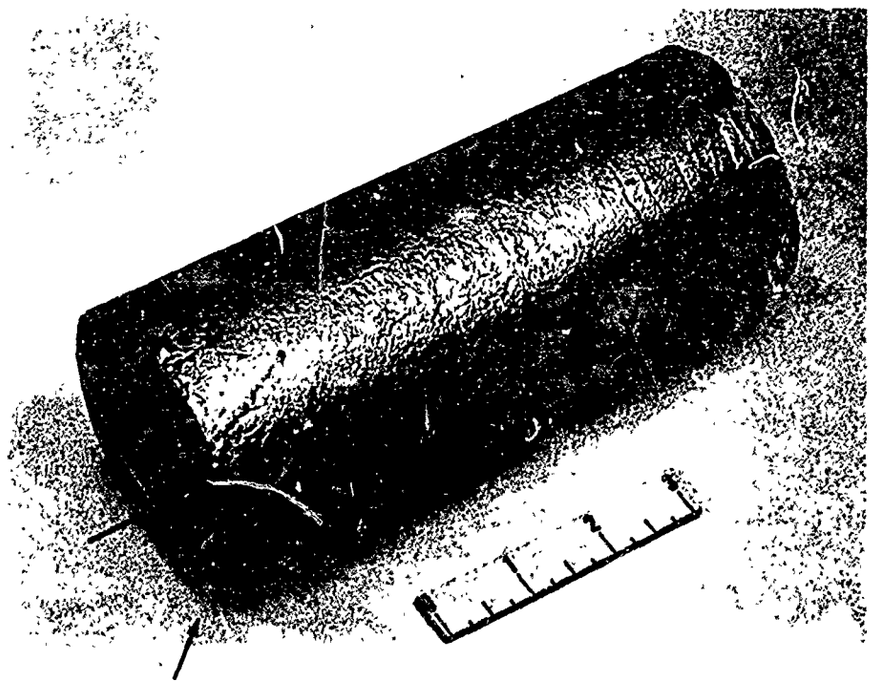
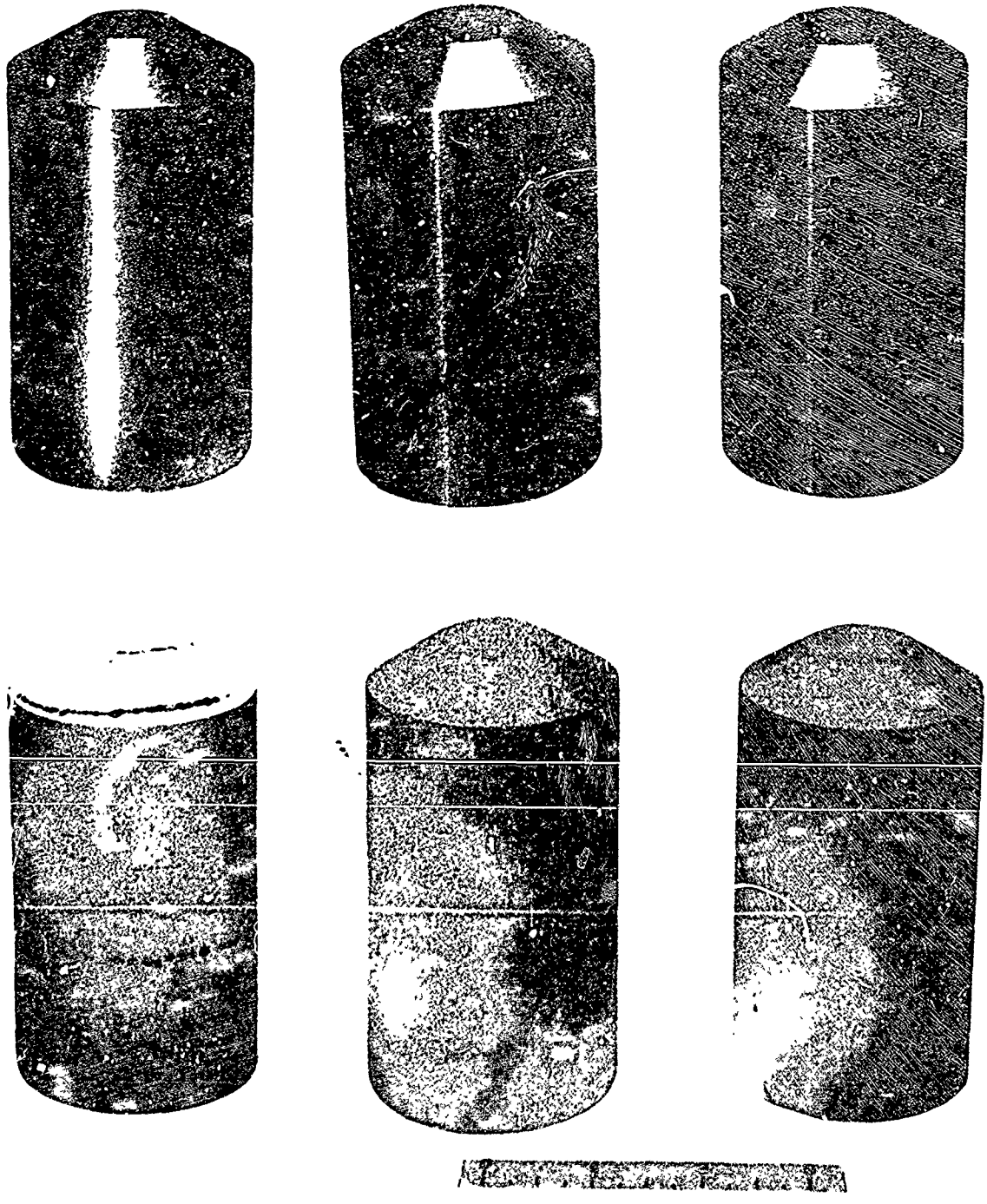


Figure 2. Billet 1A, 68W-20Ta-12Mo Alloy as Sectioned from the Sprue by Electrical Discharge Machining. The Shrink Cavity and Cracks are Apparent on the Machined Surface.



Billet 1A Billet 2A Billet 3A

Figure 3. Centrifugal Cast Billets 1A, 2A, and 3A, 68%W-20%Ta-12%Mo. (a) As Finished Ground, (b) with Die Penetrant Showing Crack Pattern.

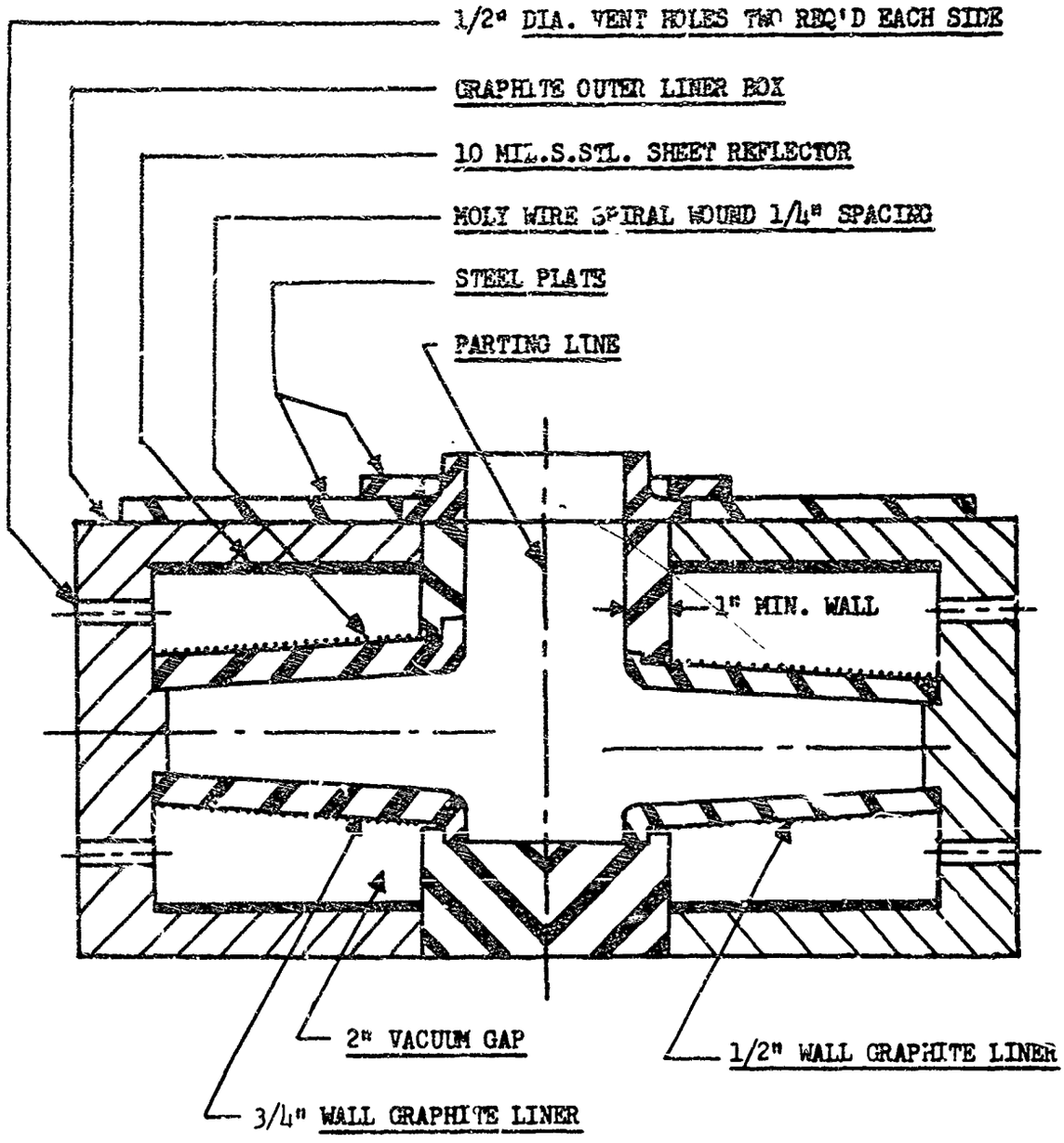


Figure 4. New Thin Wall Mold Configuration for Centrifugal Casting Billets 2A and 3A, 68W-20Ta-12Mo Alloy.

work statement outlining material processing, before, during, and after casting appears in the Appendix. The casting attempt was successful except that a slightly short pour resulted from a vacuum loss within the furnace near the end of the melting cycle (see Table I). The ingot cast in the 1/2" thick mold was identified as 2A and that cast in the 3/4" thick mold as 3A. The two ingots and sprue appear in Figure 5.

The experience with the first ingot indicated that the alloy could be handled without resorting to extreme precautions to prevent cracking. Therefore, ingots 2A and 3A were removed from the sprue with an abrasive cut-off wheel. After the ingots were removed from the sprue they were given a 3000°F stress equalization anneal for 4 hours, sectioned for metallographic and chemical analyses, machined and ground to extrusion billet size (2.5" diameter by 4.25" long), and ultrasonic and dye penetrant inspected. Upon sectioning to billet length both ingots were found to contain large shrink cavities. This was attributed to the short pour and a resultant lack of molten metal in the sprue cavity during ingot solidification.

Ultrasonic inspection of the two billets revealed that both contained cracks, but billet 2A, which was cast in the 1/2" thick graphite mold, had a sound section for 1 3/4" on the end opposite the sprue. These results indicated that this new mold which was designed to reduce the solidification and cooling rates did result in less cracking in the ingots. The two billets, as-ground and with dye penetrant developer, are also shown in Figure 3.

To obtain additional stock for further rolling and mechanical property evaluation of the 68W-20Ta-12Mo alloy, a fourth centrifugal casting consisting of two extrusion billets was made. The material used in this casting was obtained from the skull and sprue of the third casting and recycle material from the second casting. The production of a partially sound ingot during the third casting attempt in the 1/2" thick graphite mold as compared to the unsound ingot produced in the 3/4" thick graphite mold indicated that the 1/2" thick mold with the slower, more uniform cooling and solidification rate would have a greater potential of producing a sound ingot. Therefore, for this fourth casting, the 1/2" thick mold was employed for both ingots.

The pour was successful in producing two ingots identified as 4A and 5A shown in Figure 6. After the ingots were removed from the sprue, they were processed in a manner identical to ingots 1A, 2A and 3A, i.e., they were given a 3000°F stress equalization anneal for four hours, sectioned transversely to provide end slices for metallographic and chemical analyses, machined and ground to extrusion billet size, and ultrasonic and dye penetrant inspected.

Upon sectioning, both ingots were found to contain large shrink cavities. Ultrasonic inspection revealed that the shrink cavities extended approximately 2/3 of the way into the ingots. It also revealed that ingot 5A was cracked from end to end. Ingot 4A, although partially cracked, contained approximately 2" of sound material on the end opposite the sprue.

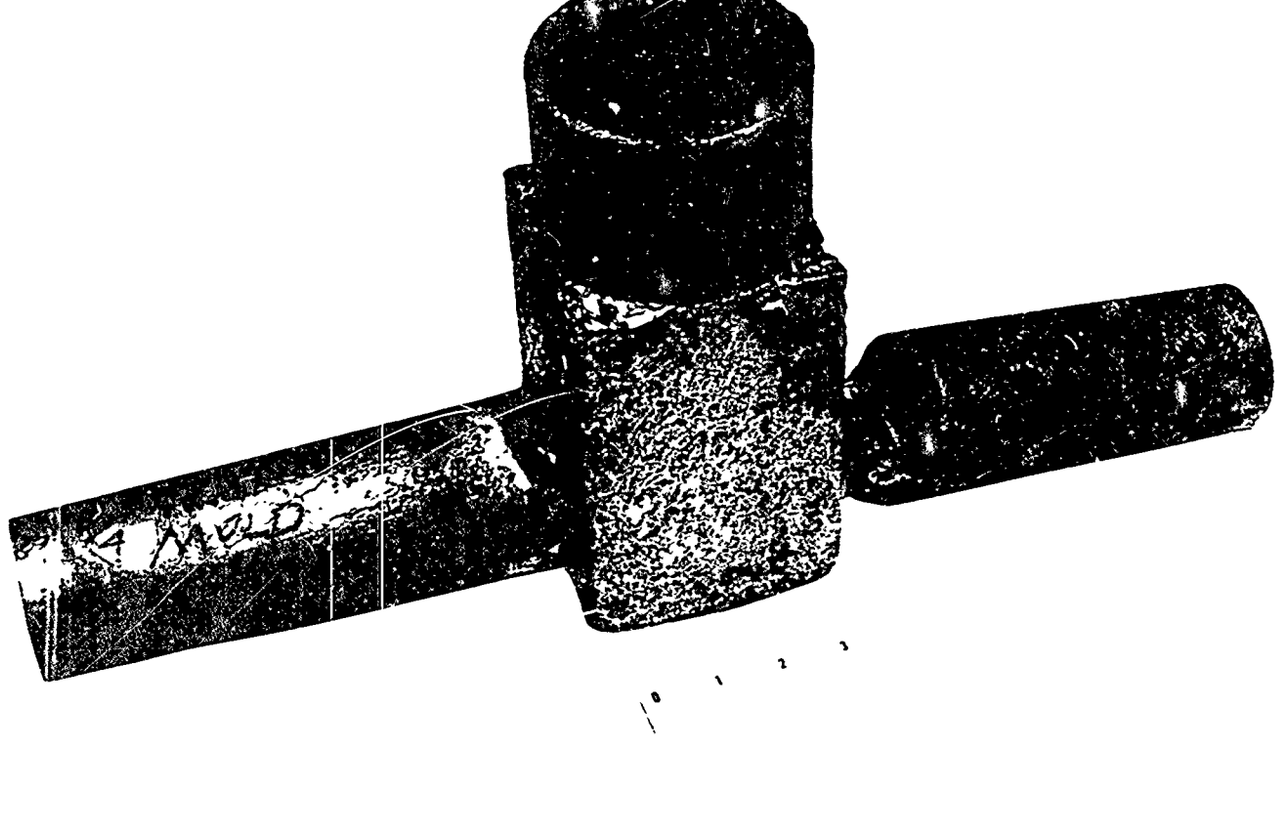


Figure 5. Ingots and Sprue of The Centrifugal Cast 68W-20Ta-12Mo Alloy. Billet 2A was Machined from the Ingot on the Right and Billet 3A from the Left.

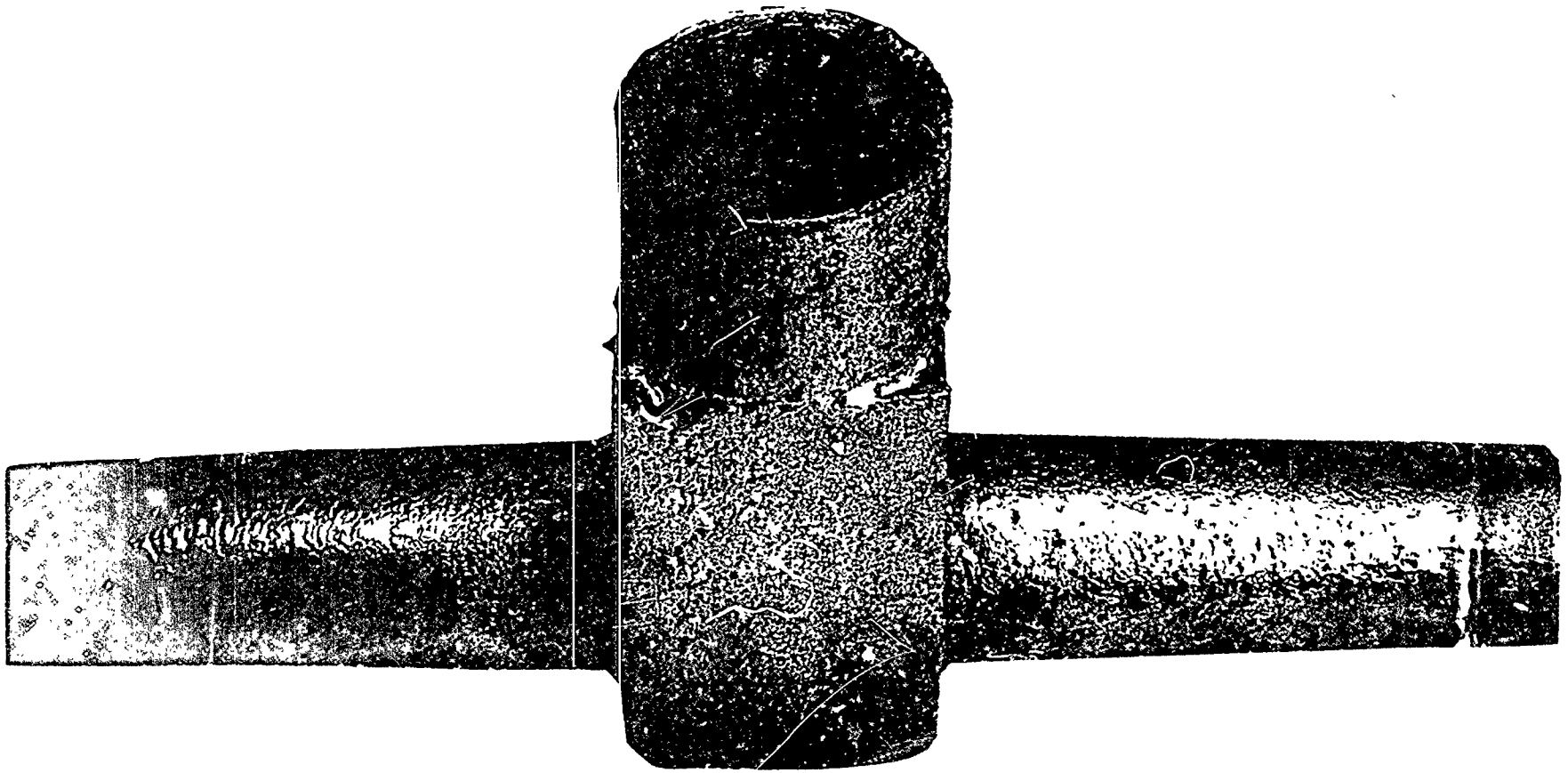


Figure 6. As-Cast Ingots 4A and 5A with Sprue Attached. Produced During the Fourth 68W-20Ta-12Mo Alloy Centrifugal Casting Pour.

Previous examination of ingots revealed that the cracking apparently was associated with the shrink cavity present. Ingots that exhibited the most shrinkage were excessively cracked. This difficulty could be eliminated by filling the sprue with molten metal so that it would act as a hot top and would feed the shrinkage cavity as the ingots solidified. An increased charge weight was used in the fifth centrifugal casting (of two ingots, 6A and 7A) in an effort to fill the sprue, and thereby minimize the shrinkage and cracking found in previous ingots. Both the ingots were cast in 1/2" thick graphite molds to produce slow uniform solidification and cooling.

Oremet offered to remelt and recast this heat when the amount of metal in the first pour was less than anticipated. The second attempt was successful in producing a casting containing the added metal in the sprue. Visual examination of these two ingots, 6A and 7A, after grinding revealed only slight cracking and no shrink cavities. The shrink was all in the sprue portion. Inspection by visual, ultrasonic and die penetrant techniques indicated that ingot 7A was free of all defects; it contained no shrink cavity or cracks. Ingot 6A was free of shrink cavity but contained a large radial crack which could have been caused by rough handling during shipment.

On the strength of the successful casting of ingot 7A, an additional casting was produced using the same conditions. Visual examination of these two ingots, identified as 8A and 9A, after grinding showed minor shrink cavities with associated cracking on the sprue ends. In Table II, it can be noted that this casting weight was slightly less than for 6A and 7A.

The final casting of this alloy was made with only one modification in procedure, i.e., the wall thickness of one of the two mold liners was reduced to 5/16". The ingot produced in this thinner graphite mold liner showed no visual or die penetrant indication of surface cracks and appeared free of internal defects in the ultrasonic inspection. Two views of this casting from which ingots 10A and 11A were removed are shown in Figure 7. Ingot 11A was cast in the 5/16" thick graphite liner. It is significant to note that this series of modifications in melting procedures not only provided material for deformation studies and evaluation at each step, but also culminated in the production of a sound ingot from which a major portion of the mechanical property data were obtained.

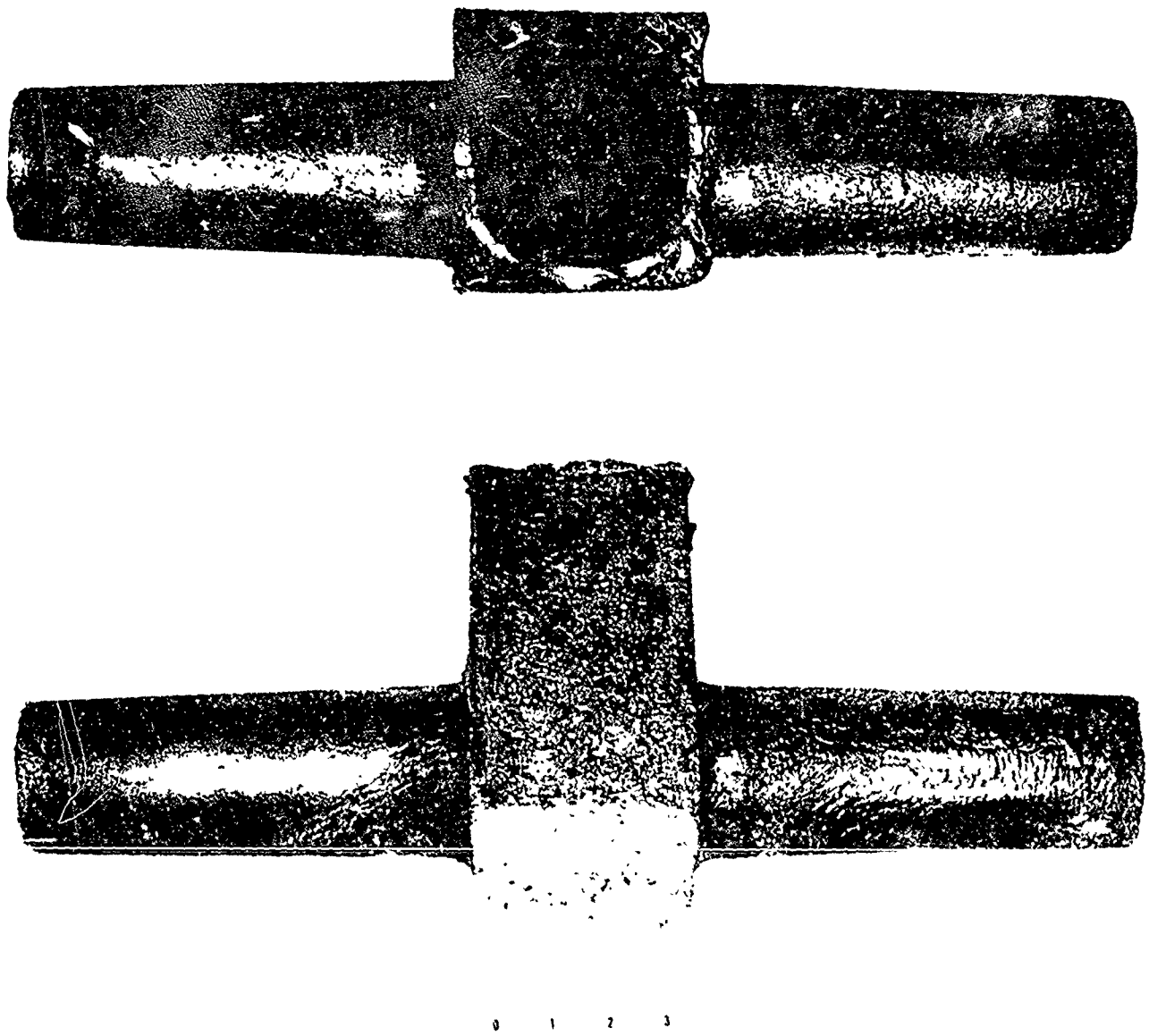


Figure 7. Top and Bottom Views of Final 68W-20Ta-12Mo Casting (Ingots 10A and 11A).

2. Chemical Analyses

Consolidation of the refractory metal alloys to produce sound ingots is one task. A second and equally important task is that of meeting the specifications for both interstitial elements and intentional alloying additions.

Complete chemical analyses representative of each centrifugal casting attempt have been obtained. The major alloying elements, tungsten, tantalum, and molybdenum, as well as interstitial elements were within limits which are considered acceptable.

Due to the low yield obtained by vacuum centrifugal casting of small ingots it is desirable to recycle as much material as possible from previous attempts. To accomplish this, sprues, electrode stubs, skulls, and machining chips from earlier attempts were crushed and blended with the required quantity of new material to prepare the electrode for each new attempt. For this reason analysis of interstitials was determined for each ingot produced. Complete chemical analyses of all of the 68W-20Ta-12Mo alloy cast in the program are listed in Table III along with the analytical techniques employed.

The amount of interstitial contamination encountered during recycle casting appears to be low. The carbon content, which would be expected to show the greatest increase due to contamination from the graphite molds, decreased with each recycle and was held at a low level until the thinnest mold was used in the final casting. A very fine pinhole leak in the crucible developed at the end of the third melt (ingots 2A and 3A) and resulted in oxygen contamination up to 80 ppm.

The interstitial level of the fourth melt (ingots 4A and 5A) was much lower than in the previous melts even though the charge material was largely recycle from the previous melt. The oxygen content was reduced by approximately 50%. The carbon content, which during the third pour had been reduced by 22% (from the second pour), was reduced even further to 16 ppm. These results, shown graphically in Figure 8, confirm that recycling of this alloy by a double vacuum arc melting and centrifugal casting technique can produce ingots within close chemical composition tolerances and with low interstitial levels.

The fifth melt produced ingots 6A and 7A. The oxygen level for the fifth melt (ingots 6A and 7A) was lower than that in all previous castings. In addition the total interstitial content of these two ingots was lower than for any previous ingot. The average oxygen and carbon levels for this casting were both 22 ppm.

The final two castings produced billets 8A, 9A, 10A and 11A and provided more evidence that the melting and casting procedures yielded good control over the major alloying elements. The interstitial level of billets 8A and 9A was about equivalent to the preceding melt. In the last melt which utilized a thinner graphite mold for ingot 11A the oxygen content increased to the level shown in the fourth melt while the carbon was the highest of any of the melts produced. The carbon increase is assumed to have resulted from a longer exposure of the alloy ingot to the graphite mold at elevated temperatures. The thinner mold acted as less of a heat sink while the tungsten alloy mass cooled in the vacuum chamber.

TABLE III

CHEMICAL ANALYSES OF CENTRIFUGALLY CAST 68W-20Ta-12Mo ALLOY

	First Melt (%)	Billet 1A (%)	Billet 2A (%)	Billet 3A (%)	Billet 4A (%)	Billet 5A (%)	Billet 6A (%)	Billet 7A (%)	Billet 8A (%)	Billet 9A (%)	Billet 11A (%)	Analytical Technique
Tungsten	66.00	68.42	67.60	68.44	67.30	67.70	68.65	68.21	67.80	67.00	67.27	Wet
Tantalum	21.05	18.19	19.22	19.16	18.27	17.90	18.86	18.92	18.65	19.55	19.40	"
Molybdenum	13.10	12.92	12.88	12.20	14.05	13.90	12.19	12.65	13.04	12.96	13.03	"
	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	
Nitrogen	10	10	10	2	10	10	10	10	10	10	10	Micro Kjeldahl
Oxygen	15	26	63	80	38	42	21	23	19	22	43,44	Inert Gas Fusion
Hydrogen	5	2	3	1	1	1	4	2	2	1	14	Vacuum Fusion
Carbon	37	32	25	25	16	16	24	19	15	16	90,60	Leco Conductometric
	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	(ppm)	
Al	300	400	300	300	10	10	10	10	10	10	10	Spectrographic
Ca	10	10	30	20	10	10	30	30	70	30	50	"
Cr	--	--	--	--	10	10	1	1	1	1	1	"
Co	50	50	10	20	--	--	--	--	--	--	2	"
Cu	--	--	10	--	6	3	10	10	8	10	10	"
Fe	10	10	10	10	20	20	30	30	10	10	20	"
Pb	--	--	20	20	30	30	30	30	30	30	2	"
Ni	--	--	60	60	20	20	10	10	10	10	1	"
Si	300	250	60	20	20	20	20	20	10	10	8	"
Sn	10	10	--	--	--	--	--	--	1	1	1	"

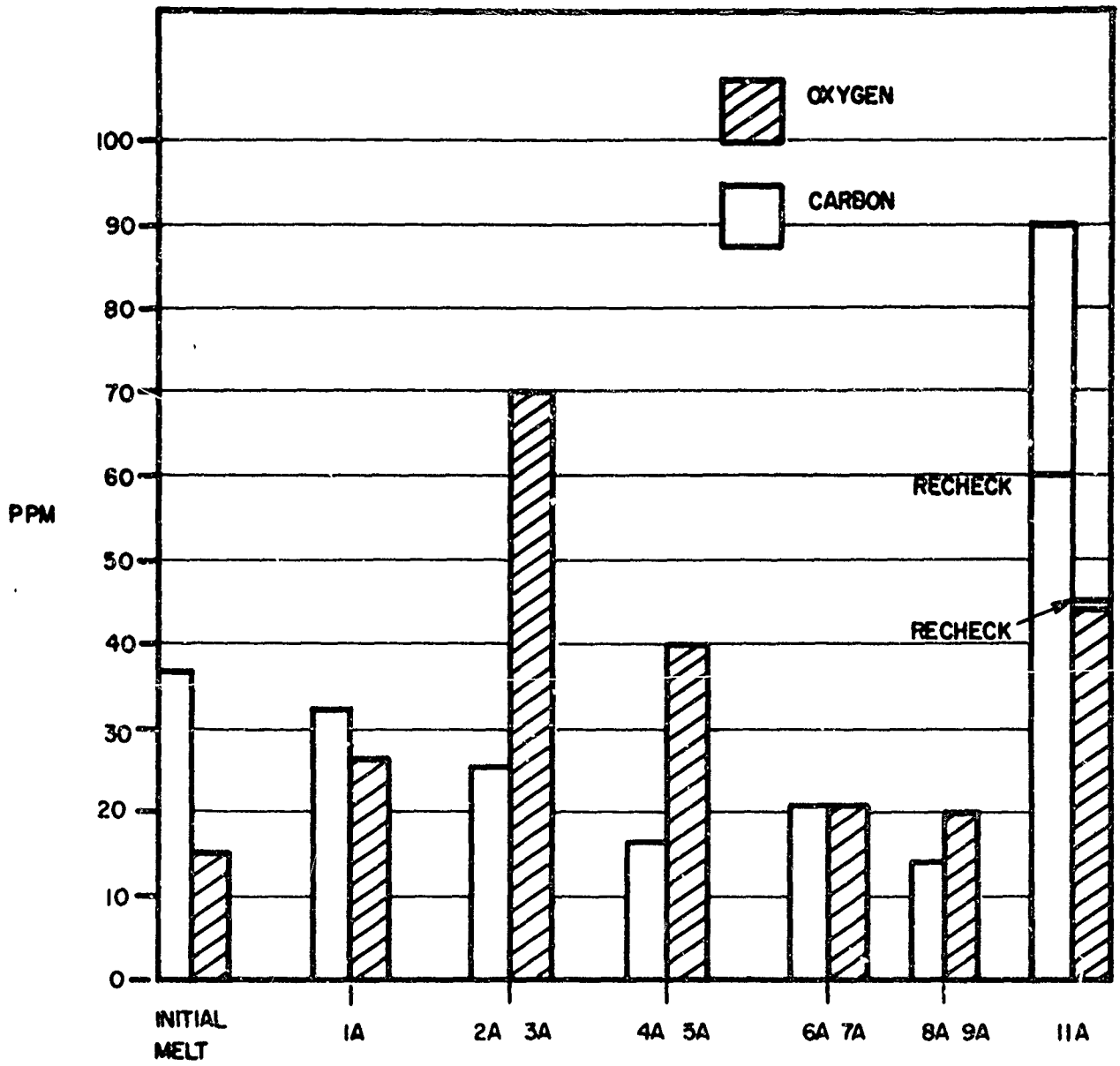


Figure 8. Average Carbon and Oxygen Analyses for Successive Centrifugal Casting Heats Using Recycle Material.

3. Ingot Microstructures

Photomicrographs of the first three ingots appear in Figure 9. The structures of all three consisted of clean, equiaxed grains approximately ASTM 4 in size. A minor amount of very fine porosity was found throughout the structure. Examination of samples from both ends of the ingots revealed that the structures were uniform throughout, with some coring existing within the as-cast grains. Photomicrographs showing the effect of 3000° and 4000°F anneals on the as-cast structure are shown in Figure 10. The coring appears to have been minimized by the 4000° treatment. To quantitatively determine the magnitude of the coring and the effects of various heat treatments a series of electron microprobe traverses were conducted on material from the second casting heat (ingot 2A)*. The material evaluated was: (1) as-cast, (2) as-cast plus a vacuum anneal for 1 hour at 3700°F, (3) as-cast plus a vacuum anneal for 1 hour and 4 hours at 4000°F. The results of this study are tabulated in Table IV. The data show that the coring was not as severe as indicated by etching response and that it could be reduced by long time anneals in the 4000°F temperature range. No change was observed after 1 hour at 3700°F. The photomicrographs in Figure 11 show the as-cast structure at high magnifications. The differential etching response certainly appears to be on a microscale rather than a macro segregation effect.

It is known in the metal processing field that heat treatment alone will not completely remove coring, but that combinations of both working and heat treatment are required. Examination of the microstructures of the as-extruded material revealed that the coring had been eliminated to the point that it was undetectable by etching response. This indicates that the high extrusion temperature and the working associated with the extrusion process are adequate to eliminate the coring observed in the as-cast structure. The long times and high temperatures required to reduce the observed coring without working make it impractical in consideration of the minor amount of improvement observed, particularly when the coring is eliminated in the extrusion operation.

Metallographic examination of the 68W-20Ta-12Mo alloy ingots 4A and 5A in the as-cast condition revealed clean equiaxed grain structures approximately ASTM 2-3 in size. Photomicrographs of this structure appear in Figure 12. Unlike ingots 2A and 3A, no porosity was found within the structure. The slightly larger grain size and smaller amount of porosity in these ingots as compared with the first three heats are a result of a slower cooling rate with the thinner graphite mold liner. Examination of samples from both ends of the ingots revealed that the structure was uniform throughout. Electron photomicrographs of the as-cast structure revealed that the material was exceptionally clean. For example, the typical grain boundary shown in Figure 12 at 10,000X magnification has only a minor amount of a second phase.

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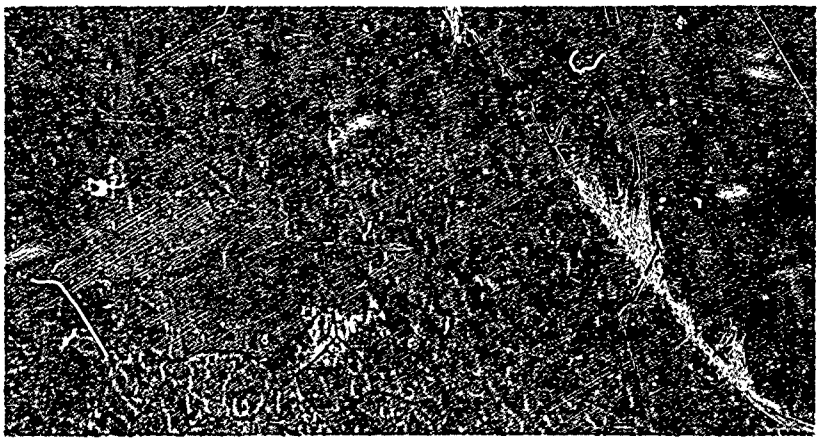
*The microprobe data were supplied by Mr. J. Gow of the Air Force Materials Laboratory.



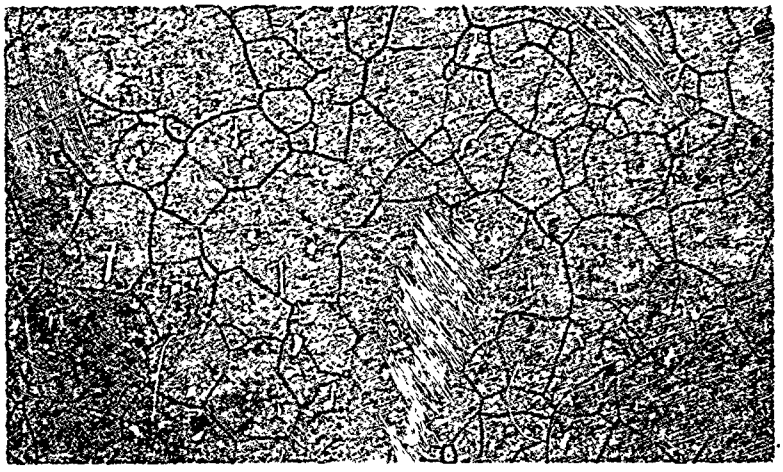
Ingot 1A

Ingot 2A

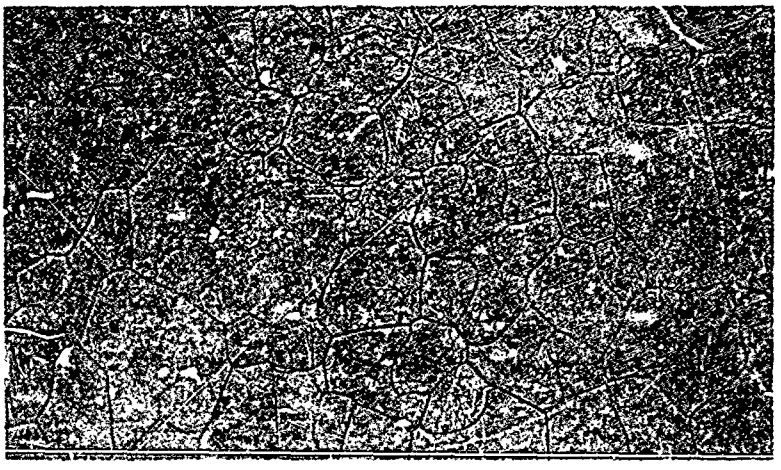
Ingot 3A

Figure 9. As Cast Structure of Ingots 1A, 2A, and 3A Centrifugal Cast 68W-20Ta-12M . 100X

a



b



c

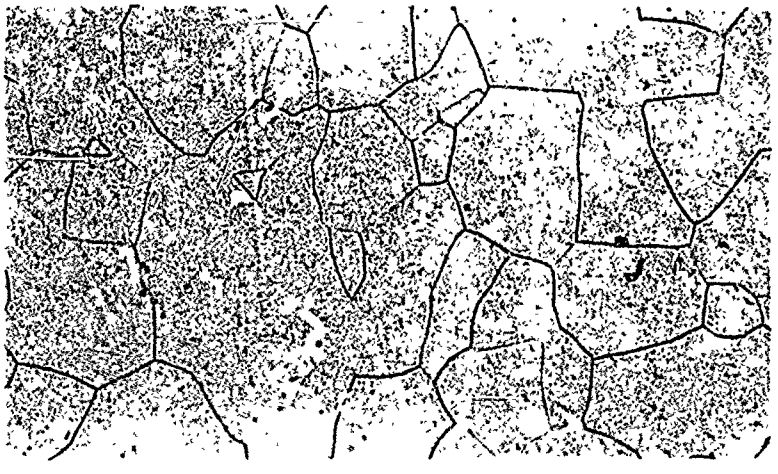
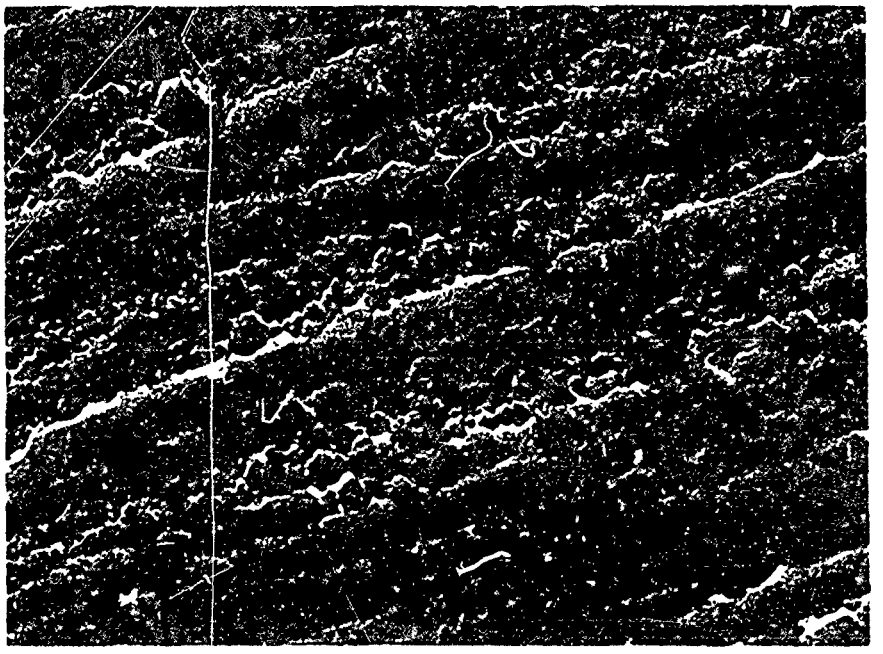


Figure 10. Microstructure of 68W-20Ta-12Mo Alloy from Centrifugal Cast Billet #1 (a) As Cast (b) As Cast Plus 4 Hour, 3000°F Stress Equilization Anneal. (c) As Cast Plus 4 Hour, 4000°F Stress Equilization Anneal. 100X

TABLE IV

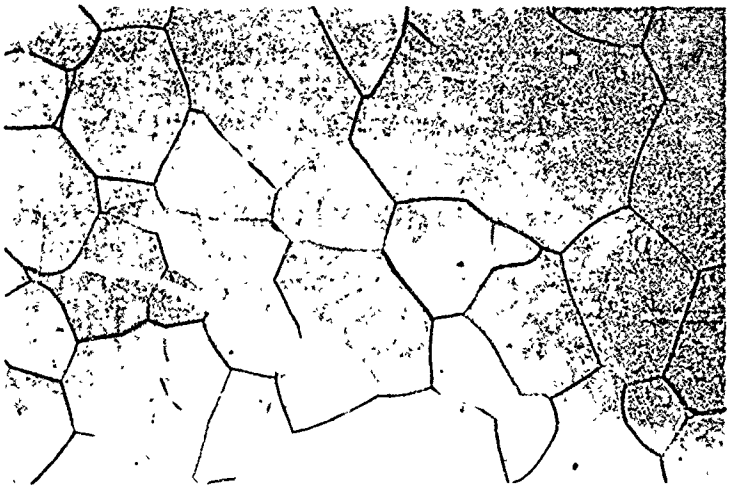
MICROPROBE ANALYSES OF SECOND HEAT OF 68W-20Ta-12Mo FOR CORING STUDY

Condition	Percent Change From Nominal Analyses		
	W	Ta	Mo
As Cast	± 5	± 2	± 3
1 hour - 3700°F	± 5	± 2	± 3
1 hour - 4000°F	± 3.5	± 1.5	± 2
4 hours- 4000°F	± 3	± 1	± 2



20,000X

Figure 11. Electron Photomicrograph of the 68W-20Ta-12Mo As-Cast Cored Structure.



Billet 4A
100X

Billet 5A
100X

Billet 5A
10,000X

Figure 12. Photomicrographs of 60W-20Ta-12Mo Billets 4A and 5A As-Cast and a Typical Grain Boundary.

Typical photomicrographs of the as-cast structure representative of ingots 6A, 7A, 8A, and 9A appear in Figure 13. The microstructure of these ingots consisted of a clean, essentially single phase structure of about ASTM 2 in grain size.

The microstructure of the final 68W-20Ta-12Mo casting is represented by ingot 11A as shown in Figure 14. The grain size appears to be about the same or slightly finer than that shown for the previous casting. The higher magnification shows a few spherical particles along the grain boundary. The structure does not show obvious defects nor massive grain boundary precipitates which would be plastic flow deterrents. The microstructure is uniform throughout thus presenting a good structure with which to begin deformation studies.

B. Deformation Studies

1. Extrusion

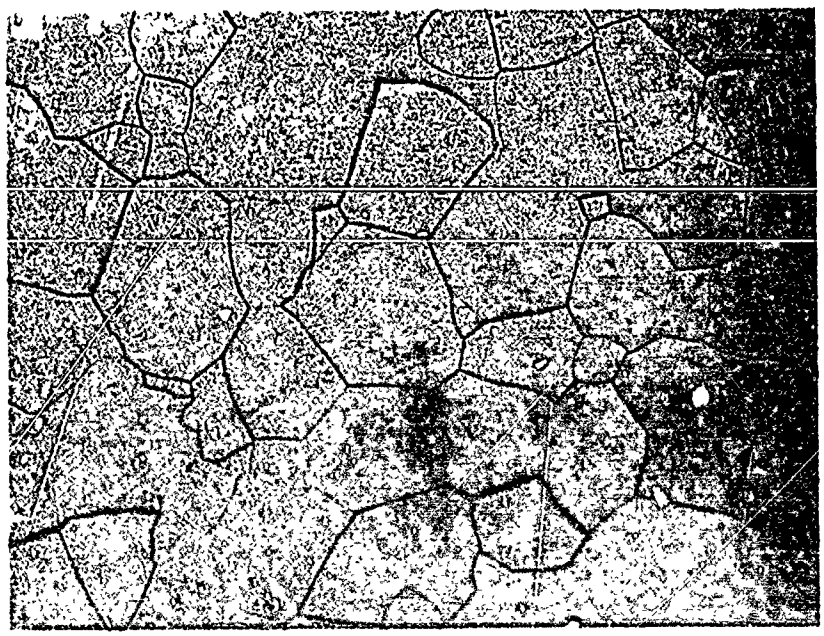
Extrusion is the optimum process for initial breakdown of the castings in order to obtain bar stock suitable for metallurgical and mechanical property evaluation and for subsequent secondary working studies. Ultra-high temperature extrusion was utilized to accomplish this breakdown. Extrusion was conducted with the TRW 700-ton Loewy fully instrumented horizontal press. Heating was conducted in a quick opening 30 KW argon filled induction furnace capable of heating 3" diameter billets above 4000°F. The extrusion dies were prepared from AISI H-21 die steel and were heat treated to a hardness of 52-54 R_c. Die entrance and orifice surfaces were coated with a .020" thick layer of zirconia to minimize die wash.

The 68W-20Ta-12Mo centrifugal cast ingots which had been finish ground to 2.5" diameter by 4.25" long as shown in Figure 15 were canned to 3.06" diameter. The cans were prepared by machining cavities the exact size of the billets in 3.06" diameter Mo-0.5% Ti bars, Figure 16. Interior and exterior surfaces of the forward can ends were machined to match the 120° included angle employed both for the die entrance and the billet noze geometry. Prior TRW experience during extrusion of canned refractory metal billets at lower temperatures had shown that these matching configurations facilitated uniform co-extrusion. Butt end caps were TIG welded to the cans in an argon filled chamber.

Lubrication practice employed during extrusion consisted of spraying a 1 to 1 by weight mixture of sodium silicate and graphite on both the press liner and the die surfaces.

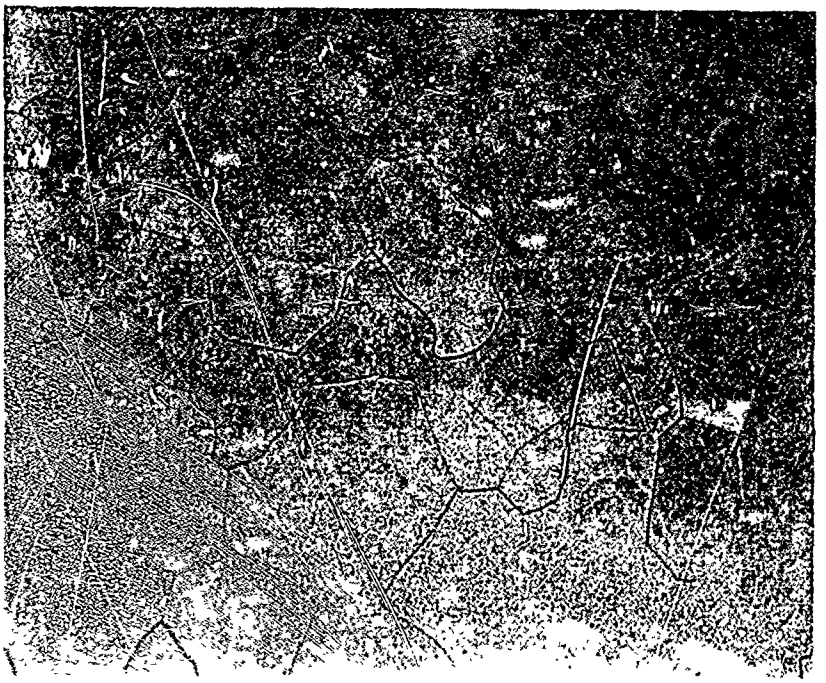
It was initially anticipated that the difference in strength at the extrusion temperature between the Mo-0.5% Ti cans and the 68W-20Ta-12Mo alloy billets might result in poor co-extrusion. If this happened it would not have been possible to obtain the minimum 4 to 1 area reduction considered necessary to obtain relatively uniform working throughout the extrusion cross section. For this reason the first three canned billets were extruded at higher total (billet plus can) area reduction ratios. The ratios employed were 5 to 1 for billet 1A, 6 to 1 for billet 2A, and 6.4 to 1 for billet 3A.

8A

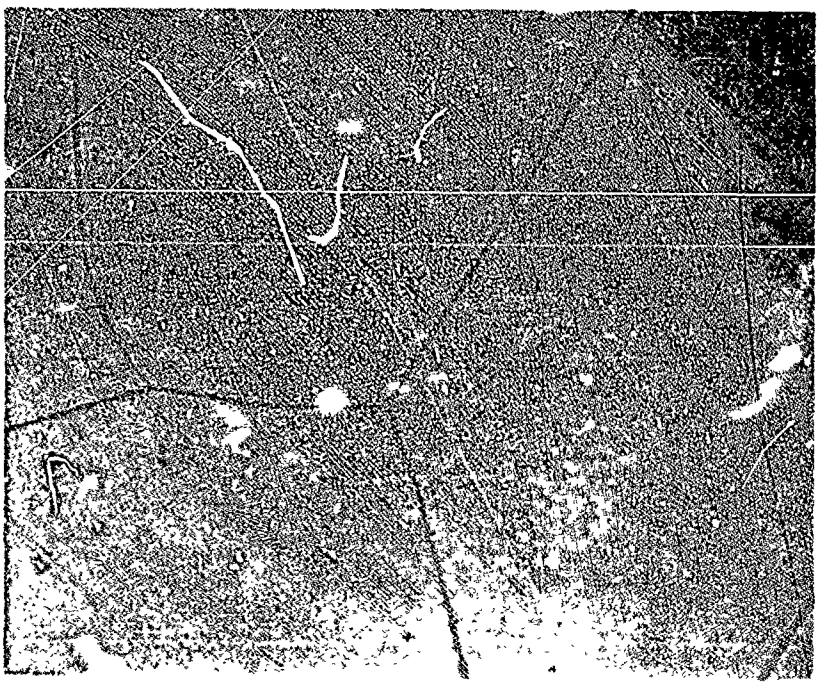


9A

Figure 13. Microstructure of As-Cast Ingots #8A and 9A, 68W-20Ta-12Mo Alloy. 100X



100X



1000X

Figure 14. Microstructure of Ingot 11A From Final 68W-20Ta-12Mo Casting, as Cast.

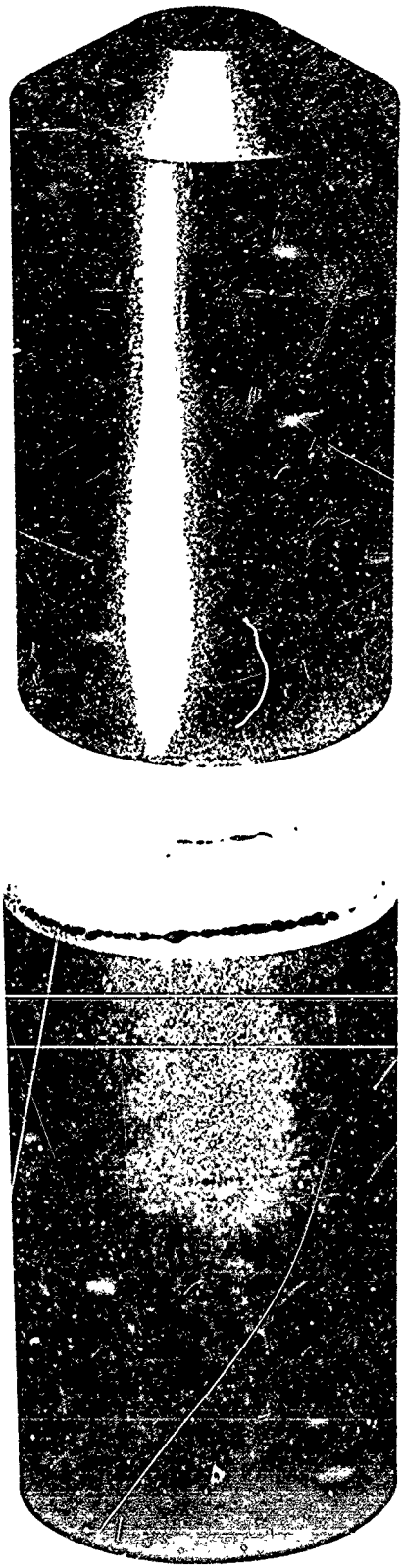


Figure 15. Centrifugal Cast Billet 1A, 68W-20Ta-12Mo. (a) As Finished Ground, (b) With Die Penetrant Showihg Crack Pattern.

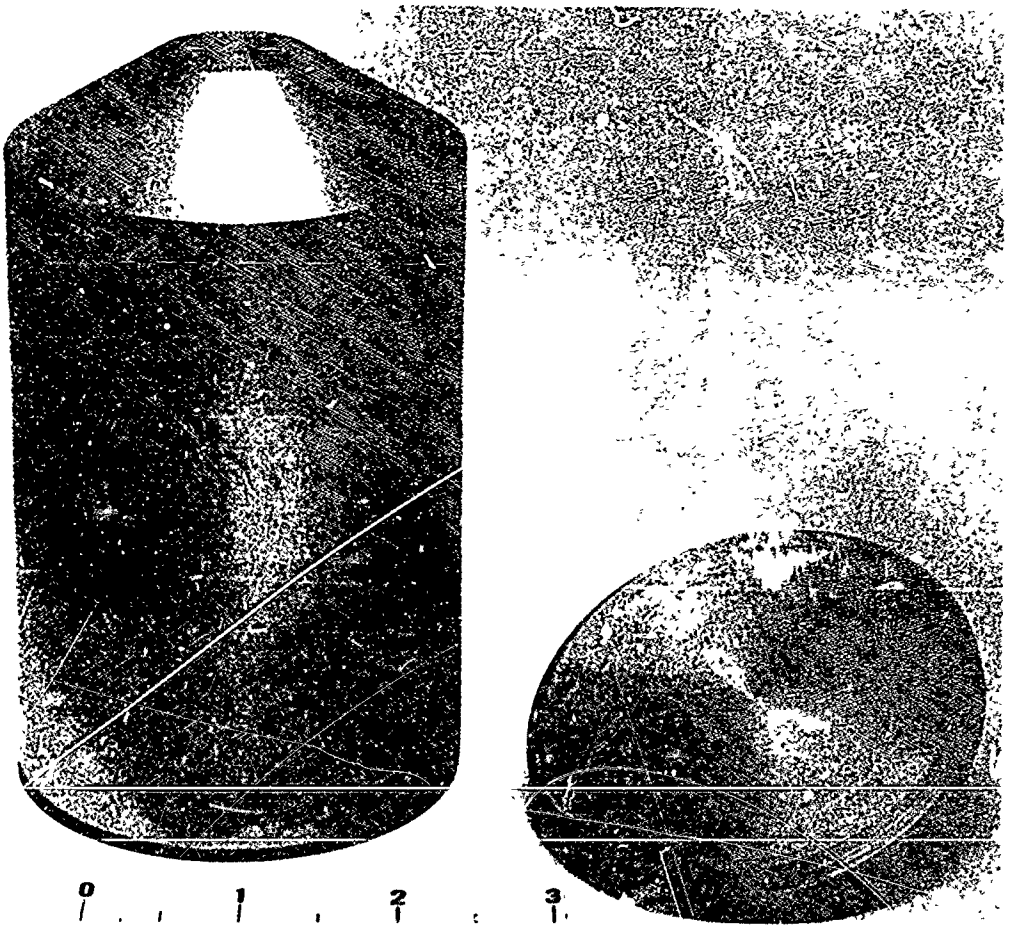


Figure 16. Typical Finished Machined Mo-0.5%Ti Can and Cap within which the Centrifugal Cast 68W-20Ta-12Mo Billets were Sealed and Extruded.

The canned billets were slowly induction heated to extrusion temperature in argon, allowed to soak for 10 minutes, and extruded. Billet temperatures were measured with W-3% Re versus W-26% Re thermocouples. The thermocouple wires used were certified to generate within ±1% of a standard emf.

All three billets were successfully extruded, Figure 17. The extrusion data are listed in Table V. The Mo-0.5% Ti surfaces were excellent and diameter measurement indicated virtually no variation from nose to tail of each extrusion. Further, the extrusion dies showed no indication of die wash.

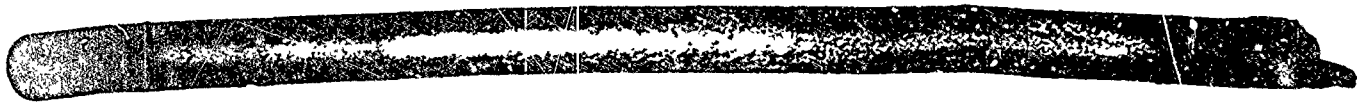
After extrusion the three bars were cropped, sectioned, and examined to evaluate the effects of the high temperature conversion step upon the structure and the crack patterns observed in the billets. This examination revealed that the cracks in the starting billets were to a great extent eliminated by the high pressures and temperatures associated with the extrusion process.

After the extrusions were cropped each was cut into sections from which samples were taken for further evaluation. Bar 1A was cut as shown in Figure 18 into three 2" long sheet bars, thirteen recrystallization samples, and two sections from which tensile specimens were machined. Bar 2A was sectioned into sheet bars to be used in rolling studies, Figure 19. Bar 3A was cut into sections for 3000, 3500, and 4000°F creep rupture tests. Each of these as-sectioned surfaces were ground, polished, etched, dye penetrant inspected, and examined at 100X for defects. Only a few minor defects were found in bars 1A and 3A and bar 2A was free of defects. In bar 1A minor microporosity was found in the rear portion of the extrusion. Bar 3A contained a few spots of microporosity in the forward end.

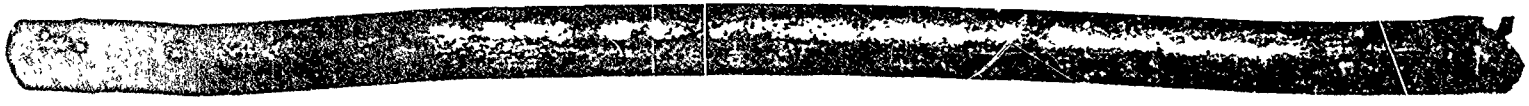
This section-by-section examination also revealed that the Mo-0.5% Ti can material had uniformly co-extruded throughout the length of each extrusion. Dye penetrant and metallographic inspection of the interface between the two materials subsequently indicated that a metallurgical bond had been formed. These excellent co-extrusion results are apparent in Figure 19.

Metallographic examination of the three bars was conducted to determine the type of structure produced. Bar 1A had a microstructure that was 40 to 60% recrystallized, while bars 2A and 3A were almost completely wrought. Photomicrographs of the as-extruded structures appear in Figure 20. It is probable that the differences in microstructures is a result of slightly higher reduction ratios for 2A and 3A and some retardation of recrystallization for these two bars as a result of the higher oxygen contents (Table III).

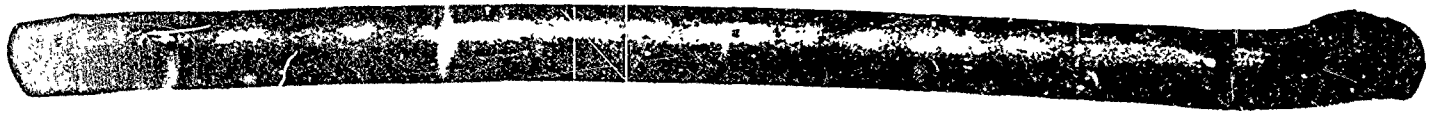
In an effort to avoid even partial recrystallization of the extrusions as experienced with the first three billets with extrusion temperatures of about 3950°F, billets 4A and 5A were extruded at lower temperatures (3710° and 3500°F respectively). The extrusion pressures as shown in Table V indicated that 3700°F was about the minimum temperature for this reduction and this alloy in the 700-ton press. The extrusions were much like the first three in appearance as can be seen in Figure 21.



1A



2A



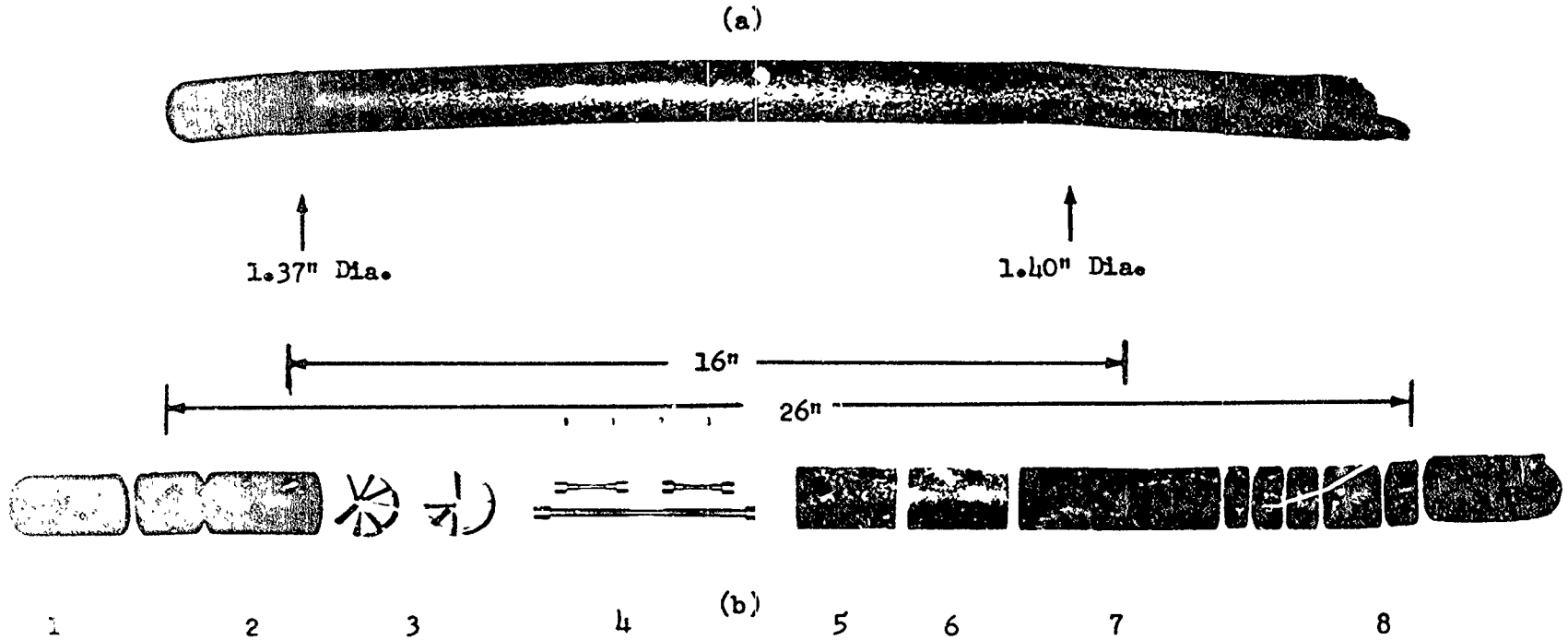
3A

Figure 17. Photograph of 68W-20Ta-12Mo Extrusion No. 1A, 2A and 3A.

TABLE V

EXTRUSION CONDITIONS FOR 68W-20Cr-12Mo ALLOY

	Extrusion Number										
	1A	2A	3A	4A	5A	6A	7A	8A	9A	10A	11A
Temp(°F)	3960	3955	3950	3710	3800	3900	3750	3500	3690	3900	3870
Ext.ratio	5:1	6:1	6.4:1	5.5:1	5.5:1	5:1	5.5:1	5:1	5:1	6:1	4.5:1
Ram Speed	14.6	11.2	9.6	7.0	9.8	Rect. -	11.0	7.4	6.8	No Chart	13.0
Break-through pressure	124,000	140,000	149,500	177,500	156,000	-	138,000	165,000	179,000		110,000
Running pressure	119,500	126,500	138,000	170,000	144,500	-	129,000	152,000	161,000		96,000
Micro-structure	0.6Rx recryst.	0.1Rx	0.15Rx	Wrought	Wrought	-	0.05Rx	0.10Rx	0.05Rx	0.2(nose) 0.05(butt)	0.01(nose)Rx 0.10 (butt)Rx
Remarks	good	good	good	good	good	Press stalled	good	fract.	fract.	good	good



Legend

1 - Nose
2 - Sheet Bar #1
3 - Recrystallization Samples
4 - Tensile and Creep Specimens
5 - Sheet Bar #2
6 - Sheet Bar #3
7 - Tensile and Creep Specimens
8 - Tail Containing Pipe

Figure 18. (a) Photograph of 68W-20Ta-12Mo Extrusion #1,
(b) Photograph showing how extrusion was selected for evaluation.

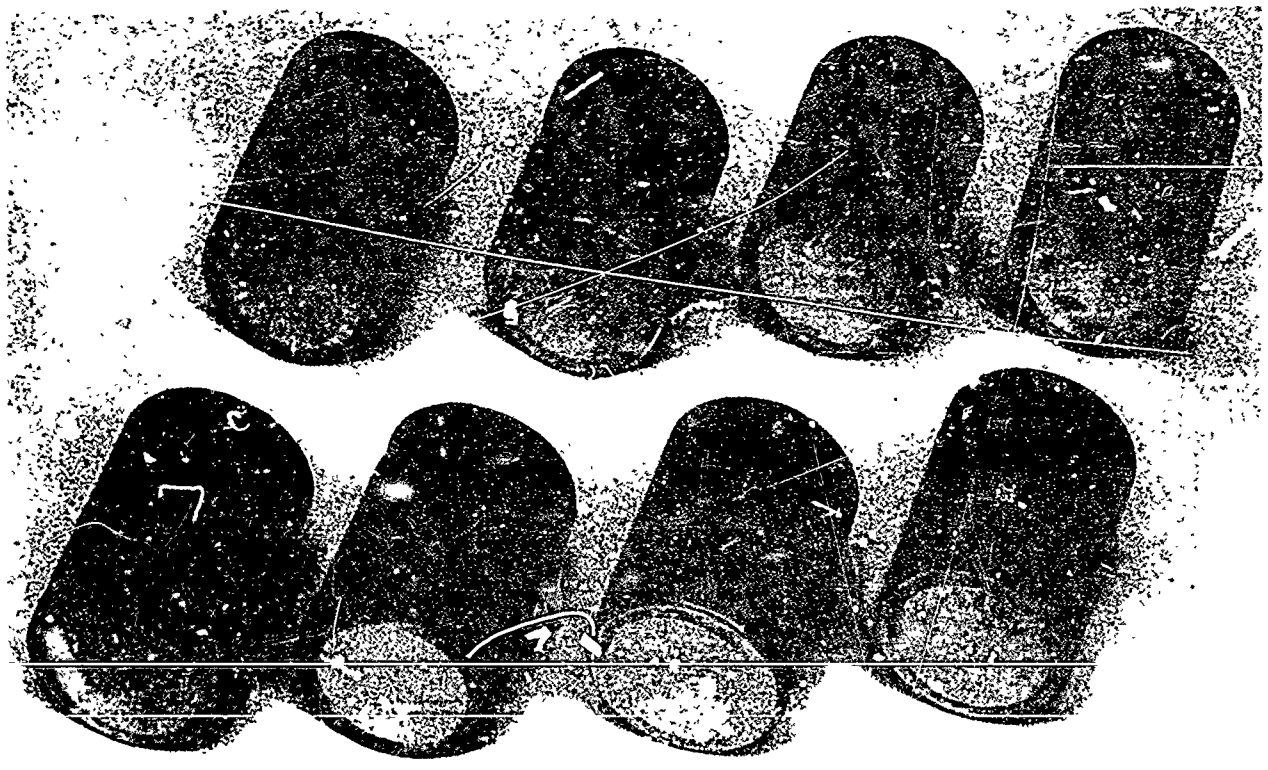
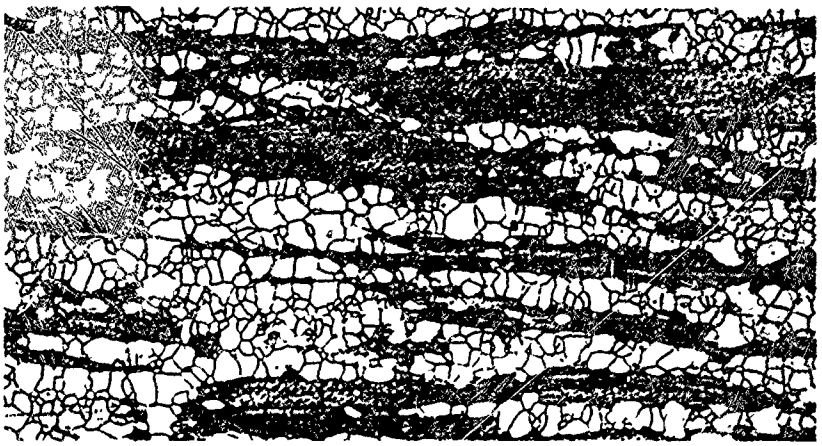
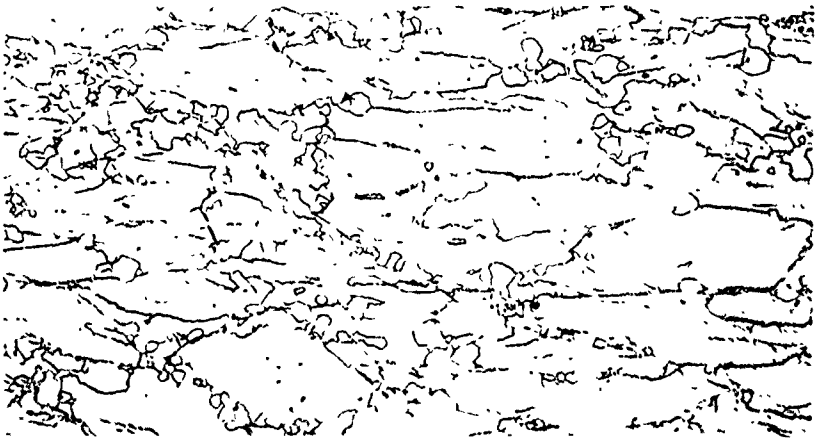


Figure 19. Sheet Bars for Rolling Development Sectioned from Extrusion 2A. The Mo-0.5%Ti Coextruded Can Material Remains on the Surfaces.



Extrusion 1A

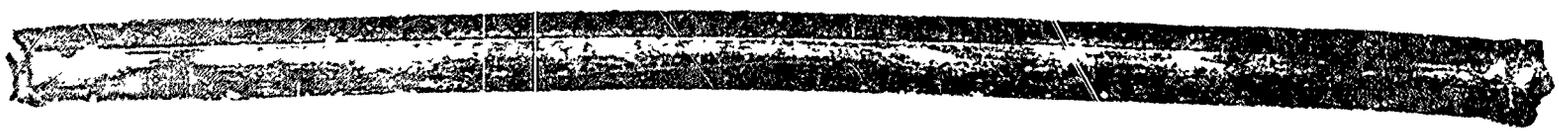
Extrusion 2A



Extrusion 3A

Figure 20. Representative Microstructures of Three 68W-20Ta-12Mo Extrusions, Longitudinal. 100X

4A



5A

Figure 21. Photographs of 68W-20Ta-12Mo Extrusions 4A and 5A.

Metallographic evaluation of extrusions 4A and 5A showed the microstructure was very nearly a true wrought structure. Both bars were examined and the microstructures in Figure 22 are typical of both.

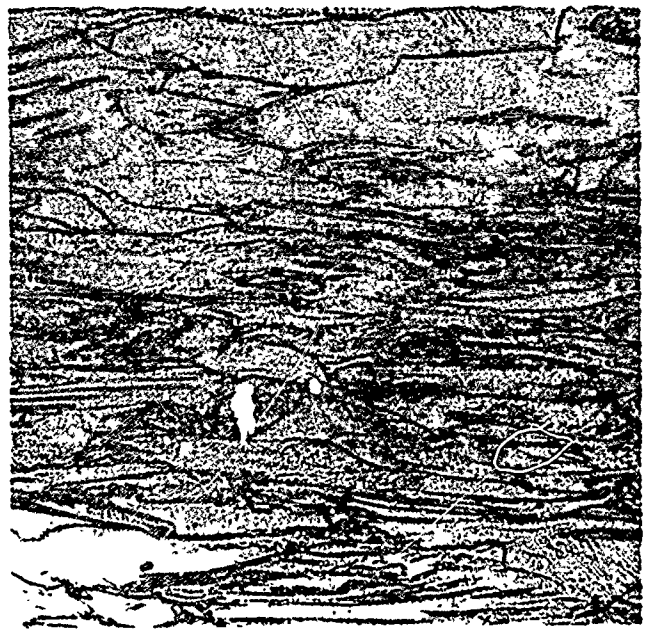
An attempt was made to extrude billet 6A to a rectangular (0.8" x 1.25") bar. This shape would facilitate rolling studies. However, the press stalled when only a minor portion of the billet had extruded into this cross section. The increased pressure requirements for the extrusion of this section are made apparent by considering that this 5 to 1 reduction stalled the press at a billet temperature of 3900°F while 4A was successfully extruded to rod using a 5.5 to 1 ratio at 3710°F.

Billet 7A was successfully extruded to a round with a 5.5 to 1 reduction at 3750°F. The extrusion surface quality and dimensional uniformity was comparable to previous extrusions. The microstructure resulting is shown in Figure 23. The initiation of recrystallization is apparent. This observation and the fact that the extrusion pressures were lower than those obtained with 5A at 3800°F indicates that the bar was probably at a higher temperature than planned.

Billets 8A and 9A were scheduled for extrusion at a 5 to 1 reduction at 3700°F to provide additional wrought material. The two billets were sound throughout approximately half of their lengths. The cracks were apparent on the surfaces and ultrasonic inspection revealed internal porosity or defects as shown in Figure 24. The extrusion press failed to go into high pressure when the ram advanced on 8A thus necessitating reheating of this billet. Difficulty was experienced in reaching 3700°F during reheating. The billet was extruded at 3500°F. The bar fractured in several places during the extrusion. Since the oxygen and carbon contents of this ingot were low by comparison with other heats and the microstructure, Figure 13, was similar to other heats it was concluded that the fractures resulted from the use of a lower extrusion temperature than for previous extrusions. Although 8A fractures in several places there was material available for tensile tests and microstructural evaluation. Photomicrographs of the as-extruded structure are shown in Figure 25.

The induction heating of billet 9A was interrupted after four minutes when the thermocouple malfunctioned. At this time the temperature was estimated to be 650°F. After replacement of the thermocouple the billet was heated normally to 3690°F for extrusion. This extrusion was also fractured as shown in Figure 26. The fracture mode indicated that cracks in the ingot propagated during extrusion. This problem may have resulted from the heating and cooling immediately before extrusion as a result of the thermocouple failure. The fact that both 8A and 9A, the only unsatisfactory round extrusions, came from the same heat is not thought to be the significant factor.

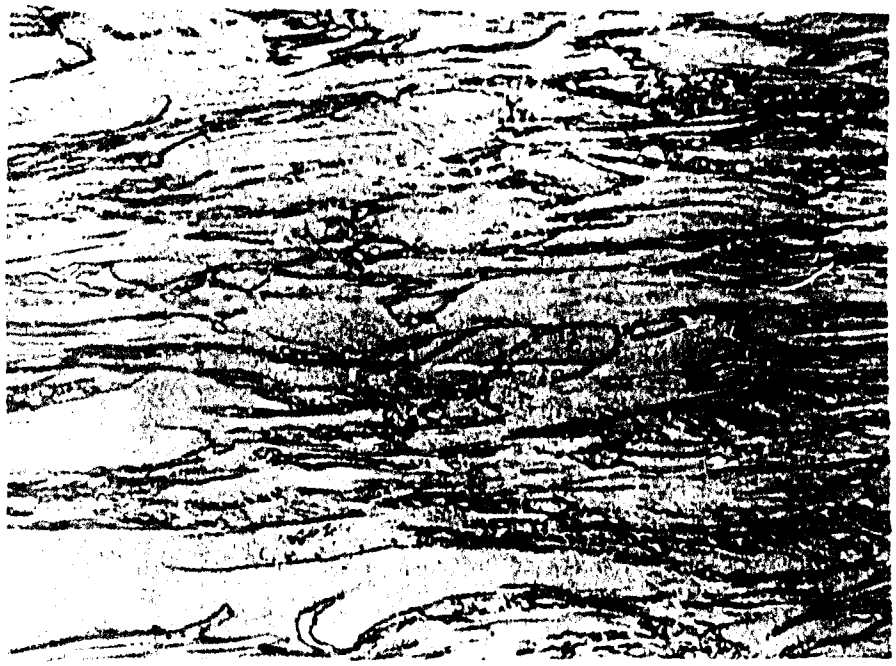
The microstructure of extrusion 9A was similar to that of 8A although more coarse as can be seen in Figure 27. Some fracturing on a micro scale can be observed as voids at grain boundary intersections (triple points). These voids at triple points were observed in the microstructures of most of the extrusions in the wrought areas. The beginning of recrystallization is also apparent in both sections.



Longitudinal

Transverse

Figure 22. Microstructures of 68W-20Ta-12Mo Extrusions Typical of Both 4A and 5A As-Extruded.



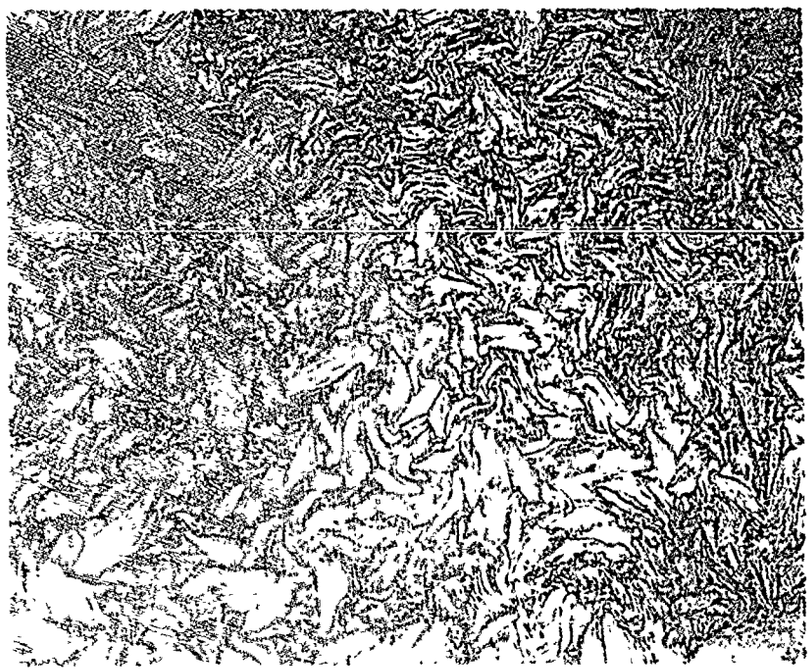
Longitudinal

Transverse

Figure 23. Microstructure of Extrusion 7A, 68W-20Ta-12Mo Alloy As-Extruded. 100X

Figure 24. View of Billets 8A and 9A From Both Sides Showing Ultrasonic Inspection Notations on One Side.

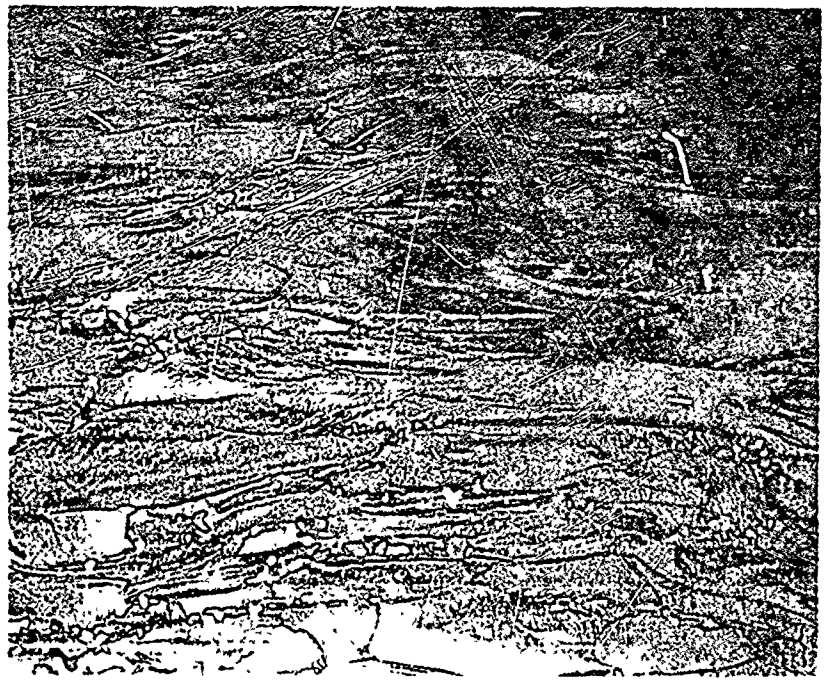
Longitudinal



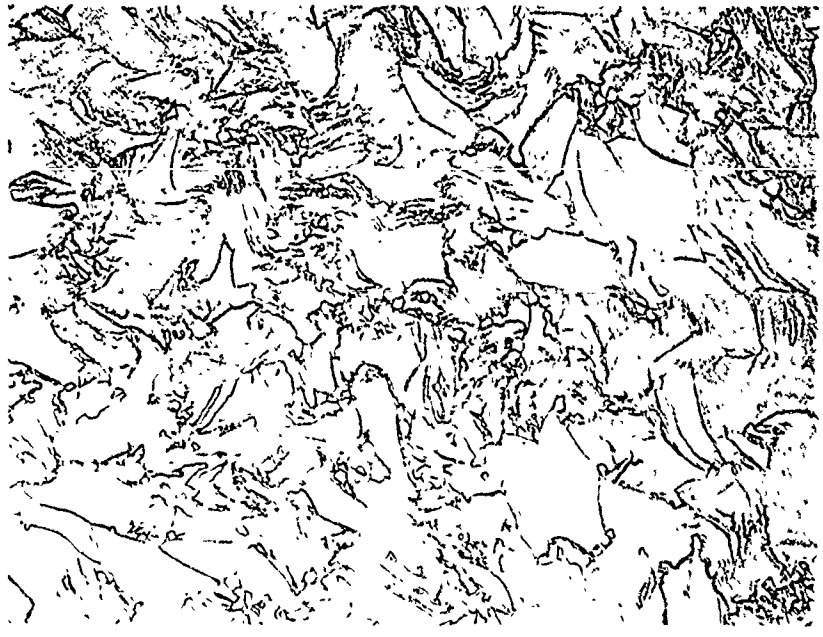
Transverse

Figure 25. Microstructure of Extrusion 8A, 68W-20Ta-12Mo Alloy, as Extruded. 100X

Figure 26. Extrusion 9A, 68W-20Ta-12Mo Alloy.



Longitudinal



Transverse

Figure 27. Microstructure of Extrusion 9A - 68W-20Ta-12Mo Alloy. 100X

Billets 10A and 11A were both extruded with success at 3900°F and 3870°F respectively. These are shown in Figure 28. The microstructures of both extrusions (Figure 29) were essentially wrought with only slight recrystallization apparent even though relatively higher temperatures were used. As shown in Figure 8 the oxygen and carbon levels were highest in this heat. The higher carbon content may have contributed to a higher recrystallization temperature although this effect is questioned by some investigators[5]. Differences in cooling rates inherent in the extrusion into a trough containing sand also result in variations in as-extruded microstructure.

Eight of the eleven billets of this alloy were extruded successfully, two with marginal success, and one stalled the press in attempting the rectangular extrusion. The primary breakdown by the extrusion process thus has been demonstrated to be a very satisfactory procedure for the 68W-20Ta-12Mo alloy.

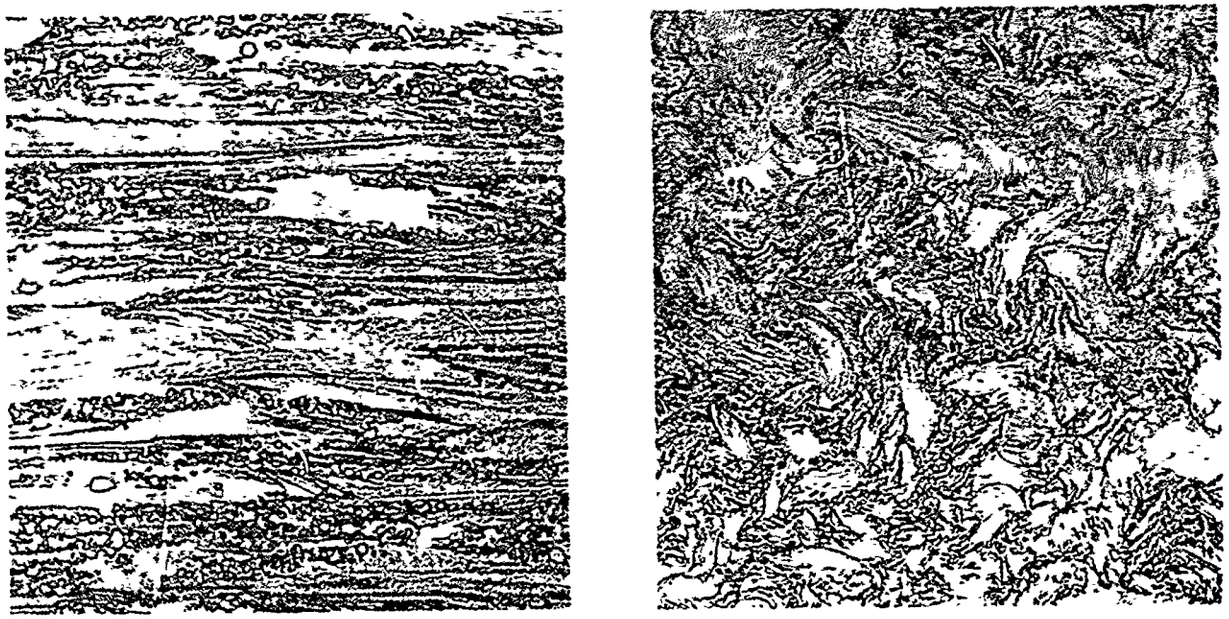
2. Forging, Rolling and Swaging

The secondary working studies of the 68W-20Ta-12Mo alloy were made to determine whether this alloy could be considered for potential sheet and bar applications. These studies were made with the Mo-0.5% Ti co-extruded envelope on the surfaces of the alloy. For example, the sheet bars in Figure 19 were 2" lengths of the 1.3" diameter extrusions. For protection of the ends, 1/4" thick Mo-0.5% Ti caps were TIG welded to the Mo-0.5% Ti envelope. The heating operations were accomplished with a 30KW induction furnace filled with flowing argon. Temperatures were measured with an optical pyrometer calibrated against a W-3% Re versus W-26% Re thermocouple. Transfer time from the furnace to the forging press or rolling mill was approximately 5 seconds. Working conditions and results for both flat forging and direct rolling are summarized in Table VI.

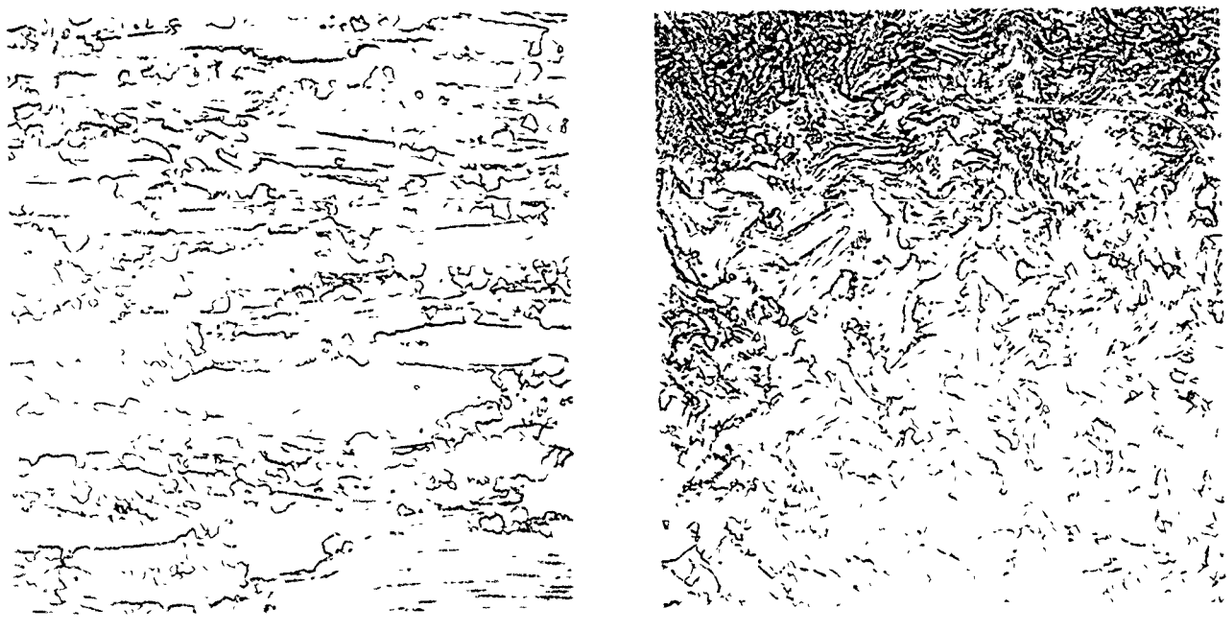
a. Forging

Fully recrystallized sheet bars from extrusion 1A were flat forged at 3350, 3800, and 4100°F on a fast acting hydraulic press with the results shown in Figure 30. Metallographic and visual evaluation of these flat forged samples indicated that 3800°F was the preferable hot working temperature for the extruded and recrystallized 68W-20Ta-12Mo alloy. In an effort to confirm this indication and provide rectangular stock for rolling, the initial sheet bar from extrusion 2A was side forged in the fully recrystallized condition with a reduction of 10% at 3800°F. This sample, unlike the previous one from extrusion 1A, cracked severely. After this failure, five additional sheet bar samples from 2A were flat forged in the fully recrystallized condition at temperatures both above and below 3800°F in an effort to establish a satisfactory working temperature. Within the range 3700 to 4000°F all samples forged exhibited severe cracking, Figure 31. Metallographic examination of the fractures within the samples revealed that all failures occurred in an intergranular manner. In subsequent efforts to evaluate the side forging characteristics of unrecrystallized stock, samples 10 through 14, prepared from extrusion 3A, were forged with varying degrees of deformation between 2800°F and 3800°F.

Figure 28. Extrusions 10A and 11A of 6Mn-2Mg-1 Mo Alloy.



10A



11A

Longitudinal

Transverse

Figure 29. Microstructures of Extrusions 10A and 11A 08W-20Ta-1.2Mo Alloy - as Extruded. 100X

TABLE VI

SUMMARY OF SECONDARY BREAKDOWN EFFORTS

68W-20Ta-12Mo

Sheet Bar No.	Extrusion No.	Bar Condition	Type of Deformation	Deformation Attempted (%)	Temp. (°F)	Results
1	1A	RX	Side Forged	30	4100	Transverse fracture
2.	1A	RX	" "	30	3800	Fractured in defect
3	1A	RX	" "	30	3350	Shattered
4	2A	RX	" "	10	3800	Single large crack
5	2A	RX	" "	10	3850	Single large crack
6	2A	RX	" "	50	3800	Shattered
7	2A	RX	" "	10	3900	Cracked
8	2A	RX	" "	10	4000	Cracked
9	2A	RX	" "	10	3700	Single Crack
10	3A	Wrought	" "	20	3800	Shattered
11	3A	Wrought	" "	20	3500	Single crack
12	3A	Wrought	" "	10	3250	Single crack
13	3A	Wrought	" "	20	3000	Shattered
14	3A	Wrought	" "	50	2800	Shattered
15	A	RX	Direct Rolled	30	3800	Refused to enter mill
	Second Attempt			20	3800	Shattered
16	2A	RX	Direct Rolled (in Mo can)	20	3750	Can deformed
	Second Pass			20	3600	Single crack
	Third Pass			20	3600	Shattered
17	2A	RX	" "	20	3700	Can deformed
	Second Pass			20	3700	Single crack

TABLE VI (continued)

Sheet Bar No.	Extrusion No.	Bar* Condition	Type of Deformation	Deformation Attempted (%)	Temp. (°F)	Results
18	5A	Wrought	Side Forged	30	3800	Cracked
19	5A	"	" "	30	4000	Cracked
20	(5A	"	" "	10	3000	Successful first reduction
	(anneal 1/2hr.at 3200)		" "	10	3000	Cracked on second reduction
21	5A	Wrought	" "	10	3000	Cracked
22	7A	RX	" "	10	2500	Cracked
23	7A	Wrought	" "	10	2500	Cracked
24	7A	Wrought	Upset Forged	15	2500	Successful
25	7A	"	" "	40	2500	Minor radial cracks
26	7A	"	" "	50	2500	Successful, checking and minor radial cracks
27	7A	"	" "	50	2500	Successful
26	Further reduction		Rolled	20	2500	Cracked
27	" " after 1/2hr.at 2500		"	30	2500	Cracked
28	11A	Wrought	Swaged	20	3550	Radial cracks
29	11A	RX	"	20	3530	End broken off
30	10A	RX	Side Forged	10	3500	Successful
	Further reduction		" "	10	3500	Radial cracks
31	11A	RX	Upset Forged	40	3050	
32	11A	RX	" "	45	3250	

(*Samples labeled RX were given a 1.5 hours - 3400°F heat treatment to impart complete recrystallization.)

4100°F 3800°F 3350°F

Figure 30. Recrystallized Sheet Bars from Extrusion 1A after Being Canned in Mo-.5Ti and after a 30% Reduction by Side Forging at the Indicated Temperatures.

These samples also failed to deform satisfactorily without cracking. Metallographic examination of the fractures in these samples indicated that the fracture mode was intergranular at 3800°F, Figure 32, and was both intergranular and transgranular from 2800 to 3500°F, Figure 33. In all cases the flat forged samples fractured in the plane of maximum tensile stress. These results indicate that the 60W-20Ta-12Mo extrusions from the third centrifugal casting attempt (ingots 2A and 3A) were low in ductility at temperatures as high as 4000°F.

The only promising side forging result was the one bar, #3 from extrusion 1A, which was reduced 30 % at 3800°F in the recrystallized condition. A crack was apparent in that sample but microscopic examination had indicated that it originated in an area showing aligned microporosity. The disappointing deformation results from ingots 2A and 3A were inconclusive because of the relatively higher oxygen content of that casting. Therefore, when ingot 5A was prepared with a lower oxygen level, the 3800°F side forging test was repeated with a wrought sample.

The extrusion 5A sample, #18, cracked when side forged 30% at 3800°F. The temperature was raised to 4000°F and sample #19 was forged 30%. This sample also cracked, Figure 34. After examining these samples, the temperature was reduced to 3000°F and sample #20 was successfully forged 10% without cracking. The sample was annealed for 1/2 hour at 3200°F to provide for stress relief and some recovery without recrystallization and re-forged an additional 10% at 3000°F. This reduction resulted in the cracks in the sample shown in Figure 35. Sample #21 was forged 10% at 3000°F in an effort to reproduce the results obtained with sample #20. It was planned to reduce the amount of reduction in the second operation, but inspection of the sample after forging revealed a crack.

Metallographic examination of the sheet bars from extrusion 5A disclosed that the samples forged at 3000°F failed in an intergranular manner. This fracture mode is believed due to grain boundary weakness resulting from interstitials. This type of failure, coupled with the change in the mode of deformation observed between 2500° and 3000°F in the tensile tests discussed in the following section indicated that future deformation studies should be conducted in the range between 2500°F and 3000°F with lower interstitial content material.

With the extrusion of billet 7A, bar stock became available for deformation studies on material having lower interstitial content. In view of the tensile elongation (25% at 2500°F) obtained from 7A and the type of fracture mode (intergranular) observed in forging samples #20 and #21 from extrusion 5A, 2500°F was selected as the forging temperature for 7A. Forging trials were conducted by side forging 2 samples 10% at 2500°F; one sample was fully recrystallized and the other wrought. Both samples fractured on forging in the same general manner as observed previously, i.e., by the formation of a longitudinal crack along the axis of the bar whose plane was parallel to the direction of working, see bars 5 and 9 in Figure 31. The cracking was observed to be more severe

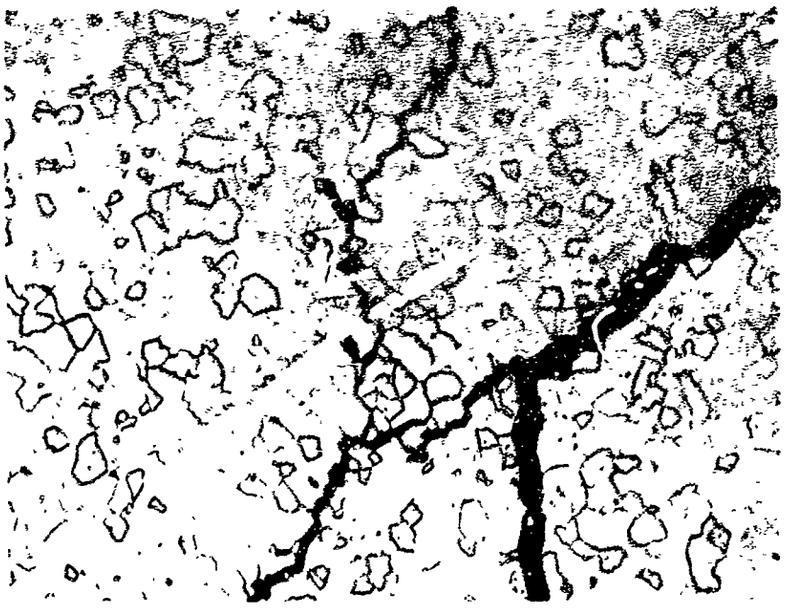
Bar #5 10% Reduction at 3800°F

Bar #6 50% Reduction at 3800°F

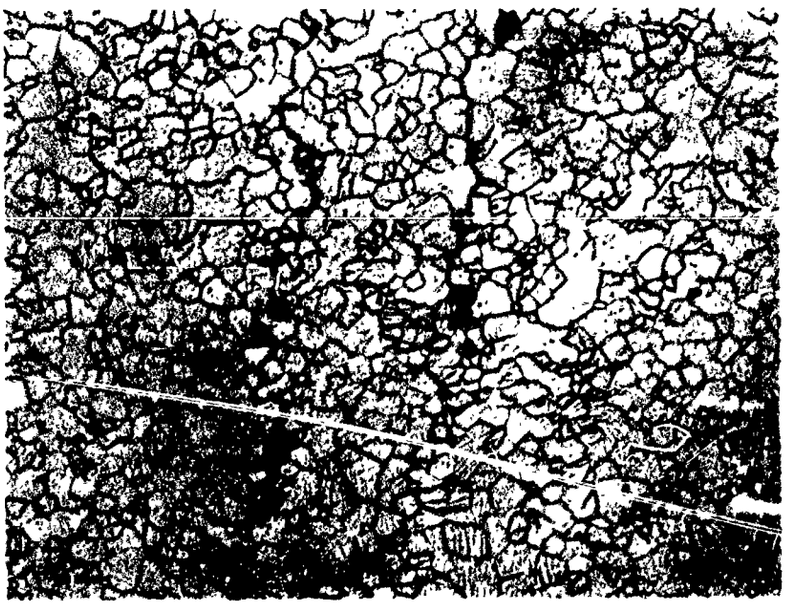
Bar #9 10% Reduction at 3700°F

Bar # 8 10% Reduction at 4000°F

Figure 31. Photographs of Side Forged Recrystallized 68W-20Ta-12Mo Sheet Bars from Extrusion 2A.

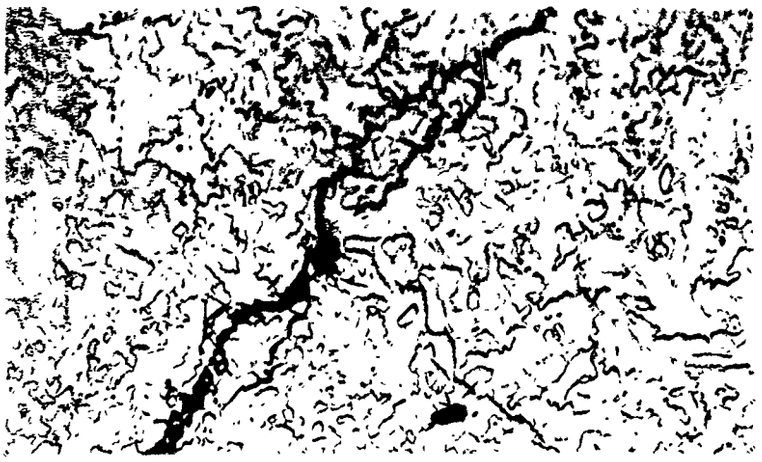


Bar #7 10% Reduction at 3900°F.



Bar #9 10% Reduction at 3700°F.

Figure 32. Photomicrographs of Typical Fracture Mode of Sheet Bars From Recrystallized Extrusion 2A Forged Between 3700°F and 4000°F. 100X

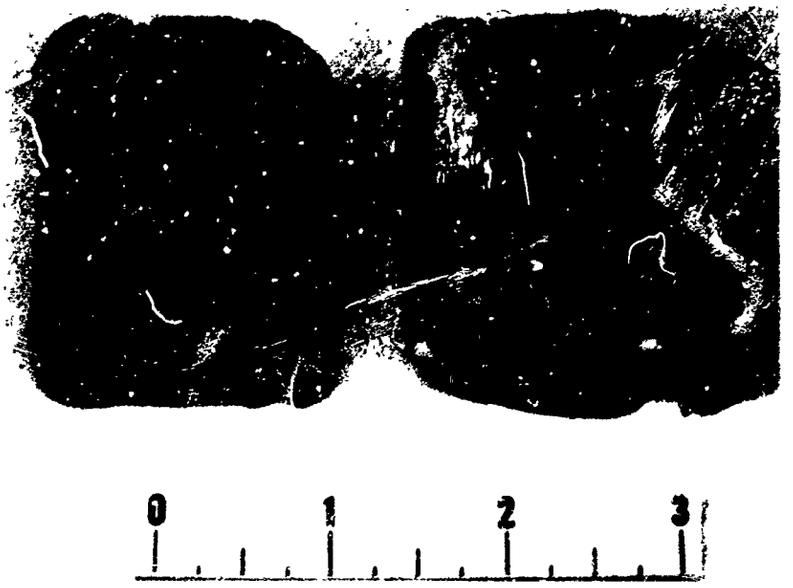


Bar #14 50% Reduction at 2800°F

Bar #12 10% Reduction at 3250°F

Bar #11 20% Reduction at 3500°F

Figure 33. Photomicrographs of Typical Fracture Mode of Sheet Bars Forged Between 2800°F and 3500°F. 100X



#18
30% - 3800°F

#19
30% - 4000°F

Figure 34. Sheet Bars #18 and #19 Side Forged 30% at 3800 and 4000°F Bars in the As-Extruded Wrought Condition from Extrusion 5A - 68W-20Ta-12Mo. Mo-0.5%Ti Container.

1st Reduction 10% - 3000°F

2nd Reduction 10% - 3000°F

Figure 35. Sheet Bar #20 After Being Successfully Side Forged 10% at 3000°F and After Fracture Upon Second 10% Reduction at 3000°F. Sheet Bar in As-Extruded Condition Taken from Extrusion 5A - 68W-20Ta-12Mo. Mo-0.5% Ti Container.

in the recrystallized than in the wrought sample. It should be noted that the fracture plane is a plane of high secondary tensile stress induced into the sample by the deformation process. This tensile stress acting on the weak grain boundaries resulted in intergranular fracture.

In an effort to reduce the tensile stresses in the sample during forging, 4 samples from extrusion 7A as-extruded were prepared for direct upsetting. The samples were deliberately made with low length to diameter ratios to induce a more uniform state of compression in the interior of the sample. The first sample was upset 15% at 2500°F. Other than minor end surface checks, presumed to have resulted from chilling by the die, the deformation was accomplished successfully. A second sample was upset 40% at 2500°F. To eliminate surface chilling the sample was placed on a 1/16" thick molybdenum sheet which was heated with the sample. This sample experienced minor radial cracks on the edges and checking on the end surface of the sample which was not protected by the molybdenum sheet. Two additional samples were upset 50% at 2500°F with molybdenum sheets on both top and bottom. Examination of the samples, shown before and after upsetting in Figure 36, revealed only minor radial cracking at the edges.

The final forging studies were made with material from extrusions 10A and 11A to evaluate the forging at temperatures above 3000°F with bar from a sound ingot having apparently more ductility as discussed in the section to follow. The first sample, #30 from 10 A, was recrystallized by annealing at 3400°F for 2 hours. It was then side forged at 3500°F for a 10% reduction successfully. The sample was then returned to a furnace and held for 5 minutes at 2500°F and slowly cooled to 1500°F, and air cooled to room temperature. The bar was reheated to 3500°F and forged to provide an additional 10% reduction which resulted in considerable cracking as shown in Figure 37.

Two samples of extrusion 11A were recrystallized by heating for 2 hours at 3400°F. Molybdenum sheet, .150" thick, was tack welded to the ends of the Mo-0.5% Ti cans and sample #31 was upset for a 40% reduction in height at 3050°F. The second sample was upset 45% at 3250°F. Both, as can be seen in Figure 38, experienced some rupture around the Mo-0.5% Ti periphery but not the catastrophic fracturing experienced in side forging and rolling deformations. Removal of the molybdenum showed no rupturing on the outer surfaces of the 68W-20Ta-12Mo alloy. The second sample showed two small internal ruptures which apparently originated with small defects in the bars.

b. Rolling

As the side forging results were disappointing, it was decided to evaluate the feasibility of rolling the recrystallized 68W-20Ta-12Mo alloy bar directly (without the intermediate side forging). With sheet bar #15 from extrusion 24 it was found that the molybdenum oxide from the

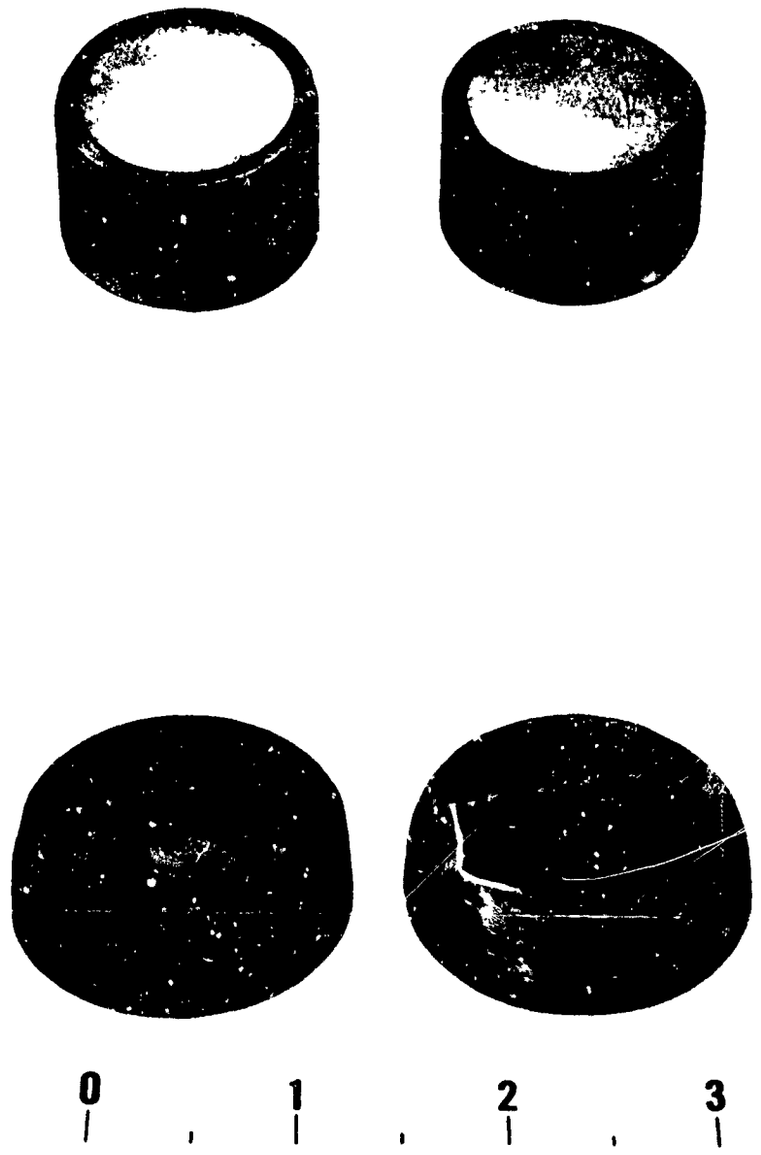
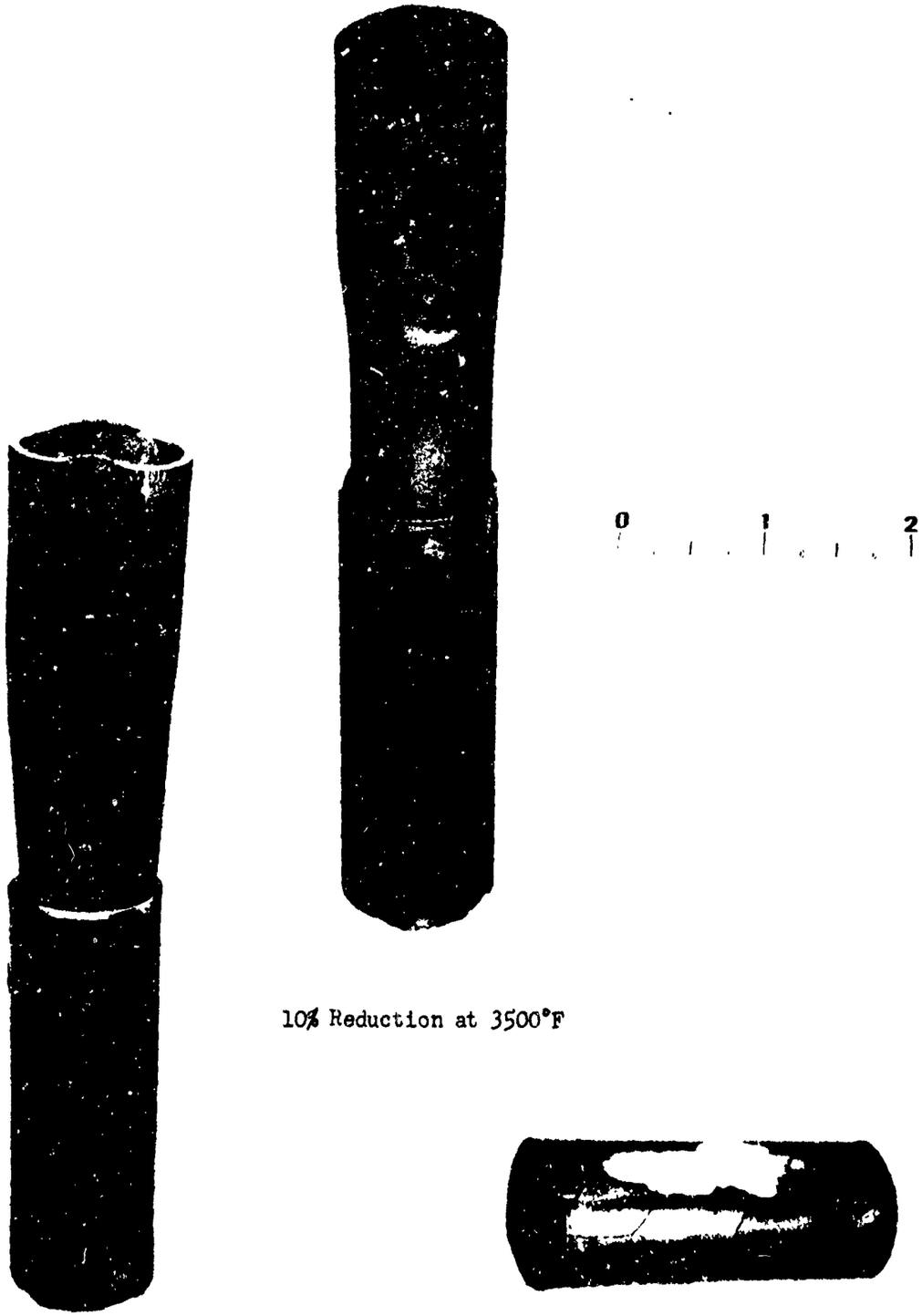


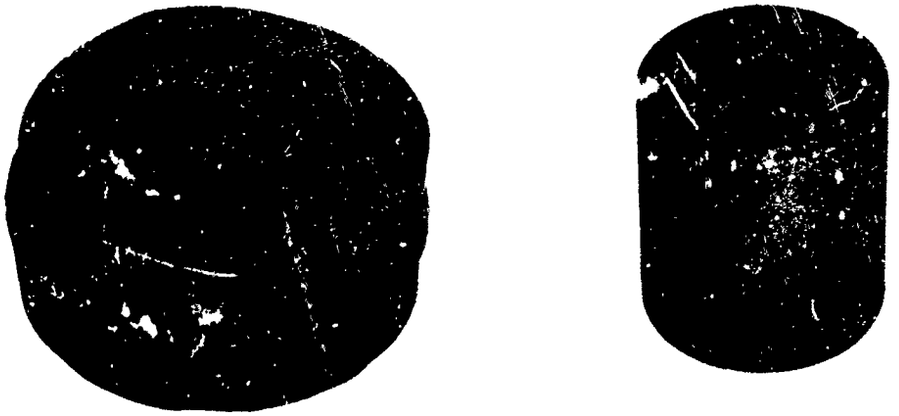
Figure 36. Upset Samples Both Before and After Being Reduced 50% at 2500°F, in the As-Extruded Condition. 68W-20Ta-12Mo Alloy, Extrusion 7A.



10% Reduction at 3500°F

20% Reduction in Two Steps at 3500°F

Figure 37. Side Forged Bars of 68W-20Ta-12Mo from Recrystallized Extrusion 104. Mo-0.5% Ti Cover on Outer Diameter.



#31 After and Before 40% Upset Reduction at 3050°F.

#31 With Mo Sheet End Caps Removed.

#32 After 45% Upset Reduction at 3250°F.

Figure 38. Upset Forged Samples of Recrystallized 68W-20Ta-12Mo Extrusion 11A. Mo-0.5% Ti Cans Cover the Alloy.

Mo-0.5% Ti cover reduced the friction so much that the bar would not enter the rolls for a 30% reduction. The sample was reheated to 3800°F and rolled to provide a 20% reduction. As can be seen in Figure 39, the results were equivalent to those obtained by hydraulic press forging.

Samples #16 and #17 from recrystallized extrusion 2A were canned in massive rectangular pressed and sintered molybdenum cans in an attempt to induce a more uniform stress pattern during rolling. These cans were intended to provide sufficient side restraint upon the sound bar to prevent fracture due to tensile stresses and to prevent chilling prior to rolling. The billets and the molybdenum cans are shown in Figure 40. Sample #16 was rolled in one pass with an initial 20% thickness reduction at 3750°F. Examination after rolling revealed that only the thick wall molybdenum can had deformed. This sample was then subjected to an additional 20% reduction pass at 3600°F. Examination revealed that it was cracked parallel to the rolling plane. After removing an end portion for metallographic examination, the sample was given a third 20% reduction pass at 3600°F. This operation resulted in severe fracturing of the sample as shown in Figure 41.

Sample #17 was rolled in an identical manner to #16, except that the temperature during the second pass was increased to 3700°F. This sample also fractured during the second pass in a plane parallel to the rolling direction. Thus it was apparent that this material had insufficient ductility even at 3700°F to permit deformation by side forging or rolling.

One additional attempt was made to work the 68W-20Ta-12Mo alloy by rolling when the lower oxygen content material from ingot 7A became available. The two samples which had been upset forged 50%, (#26 and #27 in Table VI), were stress relieved at 2500°F for 1/2 hour and were conditioned by hand grinding to eliminate the minor cracks. Molybdenum sheet was heated with the upset pieces to 2500°F and were held on the top and bottom surfaces of the alloy to prevent chilling by the rolls. The pieces were rolled to provide 20% and 30% reductions, respectively in one pass each. Both samples fractured severely as shown in Figure 42. The photomicrographs in Figure 43 show the transgranular shear fracture with only occasional grain boundary fracture propagation incurred in rolling the upset billets. The failure of the alloy to deform under these conditions resulted in the conclusion that no further rolling studies were warranted. The remaining deformation study was to determine whether swaging provided sufficiently lower tensile stresses to produce bar or rod products with the alloy.

c. Swaging

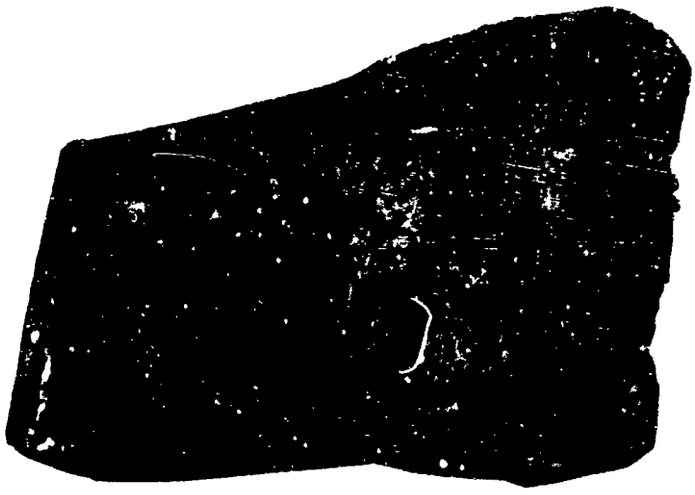
Eight samples were prepared from extrusions 10A and 11A for swaging. These were 0.79" diameter by 2.15" long. They were encased in Mo-0.5% Ti bars as shown in Figure 44 to reduce chilling and to provide tong hold material.

Bar #15 20% Reduction at 3800°F

Figure 39. Sample From Recrystallized Extrusion 2A Rolled Direct From Round Bar (above) and Photomicrograph Showing Fracture Mode (below). 100X

Bar #16 Bar #17

Figure 40. Rolling Bars #16 and #17 From Extrusion 2A with the Molybdenum Cans in Which They Were Sealed and Filled.



Bar #17

Bar #16

Figure 41. Sample #17 After the Second Roll Pass and Sample #16 After the Third Roll Pass.

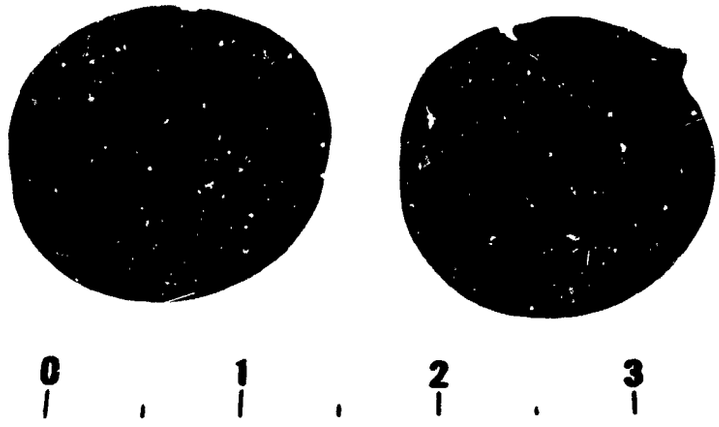


Figure 42. Upset Samples of Extrusion 7A After Being Reduced 20% and 30% by Rolling at 2500°F With Molybdenum Sheet on Surfaces of 68W-20Ta-12Mo Alloy.

#26 20% Reduction by Rolling at 2500°F

#27 30% Reduction by Rolling at 2500°F.

Figure 43. Photomicrographs Showing Fractures Produced by Rolling Bars from Extrusion 7A - 68W-20Ta-12Mo Alloy After Upset Forging 50%.

Figure 44. Swaging Samples of 68W-20Ta-12Mo Alloy Prepared From Extrusions 10A and 11A. Bars Were Encased in Mo-0.5%Ti Alloy Bars.

The first sample in the as-extruded condition was induction heated in an argon atmosphere to 3550°F and reduced 20% . After cooling in air, several radial cracks were apparent in the end of the swaged bar (No. 28 in Table VI). The swaging temperature was selected on the basis of ductility data from tensile tests of 11A which, as shown in the following section, indicated good ductility at 2500°F to 3500°F. The loss of temperature in swaging is severe so the higher temperature end of the range was used. Even with rapid transfer from the furnace and in swaging, the molybdenum tube was below red heat after completing the reduction.

The second sample was recrystallized by a 2-hour 3400°F anneal at a pressure of 4×10^{-6} torr prior to swaging. The sample was then heated in argon atmosphere to 3530°F and swaged in dies providing a 20% reduction. In addition to the radial cracks similar to those observed with the initial sample, the bar fractured about 1/2" from the end in a place approximately perpendicular to the axis.

The swaging efforts were discontinued since it was apparent that neither the wrought nor recrystallized specimens could be deformed satisfactorily with even this modified swaging procedure. The problem appeared to be that the temperature fell below 2000°F before the reduction was completed and below this temperature the alloy had insufficient ductility to permit the reduction desired.

C. Metallurgical Evaluation of 68W-20Ta-12Mo Alloy

Westgren and co-workers explored a series of solid solution refractory alloys[1] from which the 68W-20Ta-12Mo alloy was selected as one of the most promising. The original data represented, in most cases, one or two elevated temperature tensile tests of an as-extruded bar of each composition. One important contribution of the current program is the more thorough characterization of this alloy.

The microstructure of the castings and of the wrought products have been presented and discussed in the preceding sections. The consolidation and deformation studies were accompanied throughout the program with concurrent metallurgical evaluations consisting of recrystallization studies, metallographic examinations, and mechanical property evaluation including tensile and creep rupture tests.

1. Recrystallization Studies

The extrusion from ingot 1A was estimated to be 60% recrystallized. Small pie-shaped specimens of the bar (see Figure 18) were heated in vacuum for one hour at temperatures from 2500°F to 3500°F. At 3000°F the recrystallization was initiated in one hour although the hardness had not been substantially reduced. Since the bar was 60% recrystallized initially, a substantial decrease in hardness would not be expected. At 3300°F the structure appeared to be completely recrystallized in one hour. The microstructures and hardnesses are shown in Figure 45.

Extrusion 4A which was in an essentially wrought condition was treated in a like manner to determine recrystallization behavior. This bar also appeared to require one hour at 3300°F to complete the recrystallization. The microstructures and hardnesses are shown in Figure 46.

3. Mechanical Property and Metallographic Evaluation

The mechanical property tests of the 68W-20Ta-12Mo alloy were made with specimens prepared from extrusions and thus represent longitudinally oriented material. Mechanical properties of both wrought and recrystallized material have been determined over a considerable temperature range, i.e., tensile properties from room temperature to 4000°F and creep rupture properties from 3000°F to 4000°F.

a. Tensile Tests

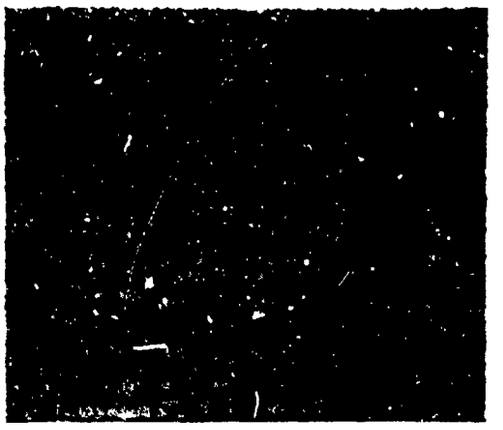
Photographs of fractured tensile specimens appear in Figure 47. Included are representative ductile and brittle failures. The data for all of the specimens tested are reported in Table VII.

Tensile testing was conducted in a resistance heated Brew vacuum furnace installed in an Instron tensile testing machine. The pressure during the tests as measured with an ionization gauge varied from 4×10^{-4} Torr to 4×10^{-5} Torr. Heating times varied from about 20 minutes to 40 minutes, depending upon the test temperature. Specimens were held at temperature for five minutes before testing. The temperature was measured during the test with a W-3% Re vs W-26% Re thermocouple. The crosshead speed was .020 in/in/minute with one exception (noted in the table) for which a 3000°F test was made at 10 times this rate to determine the effect on the properties.

A wide variation was apparent in the ductility of the various heats tested, particularly in the "intermediate temperature" range (from 2500°F to 3500°F for this alloy). After testing, the specimens were examined to provide additional information on the microstructure and the fracture mode. In a number of specimens, it was apparent that defects in the form of microporosity or cracks at grain boundary triple points existed prior to testing. These defects persisted from the casting or were generated by extrusion in such fine form that non-destructive testing was ineffective in identifying them. Examples of these are shown in Figure 48. Because of their location the cracks at the triple points are very detrimental to the mechanical properties of the wrought (or partially wrought) material. The recrystallization process to a large extent alleviates this effect because grain boundary motion leaves the voids at less detrimental locations. For this reason, if a specimen did not appear to be completely recrystallized as designated, the data were considered questionable. Those tests involving recrystallized specimens which were judged satisfactory are marked with an asterisk in Table VII. Ultimate tensile strengths, fracture stresses, yield stresses, elongations, and reductions in area for these specimens have been plotted as a function of temperature in Figures 49-53. The data show the significant strengthening resulting from cold work in this alloy.

Both elongation and reduction of area increase rapidly above 2000°F, the material exhibiting little ductility below 2000°F. After reaching a maximum at 2500°-3000°F, the ductility decreases somewhat at higher tempera-

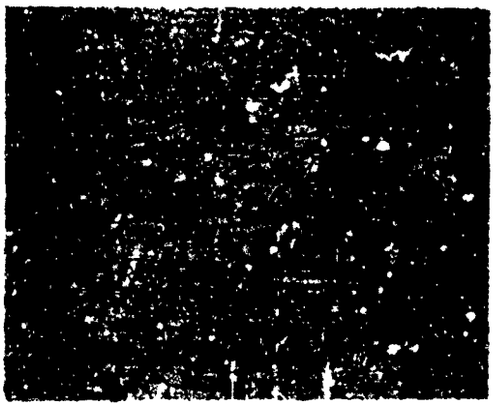
As Extruded (R_c38.4)



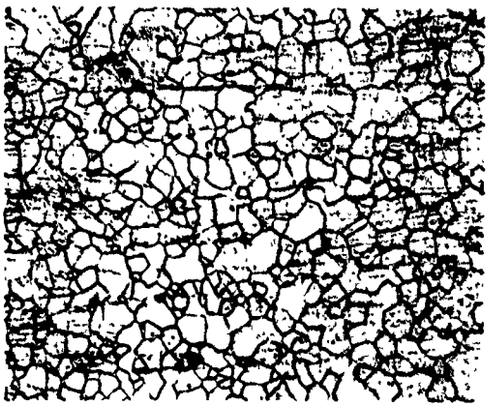
2500°F (R_c38.1)

3000°F (R_c 38.2)

3200°F (R_c 36.8)



3300°F (R_c 36.1)

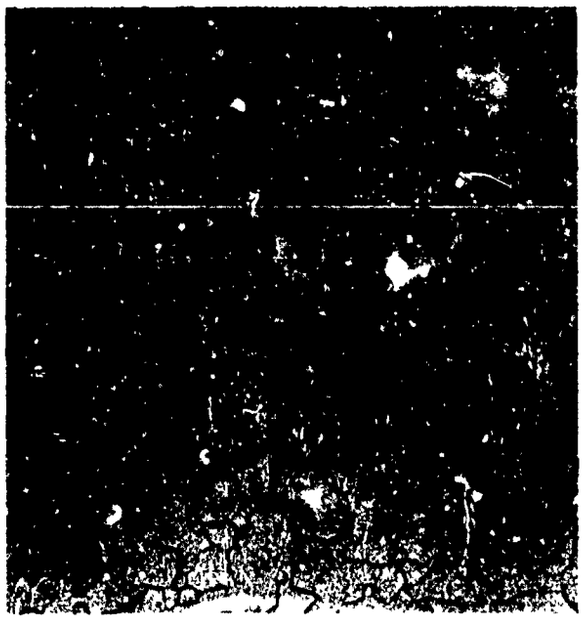


3500°F (R_c 35.2)

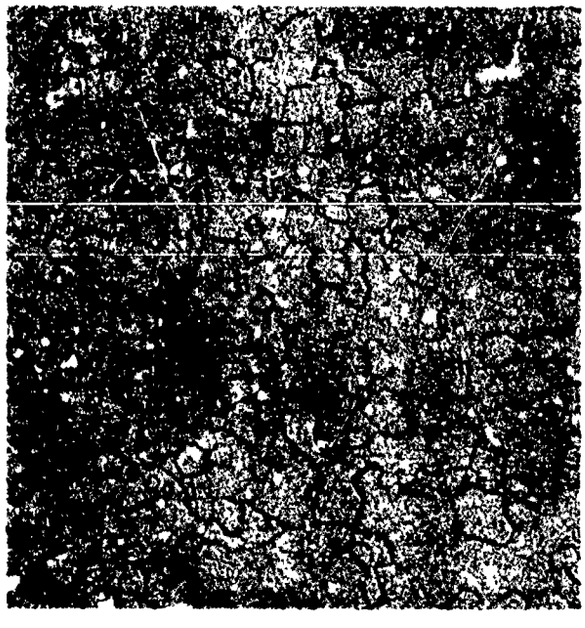
Figure 45. Microstructures of Extrusion 1A After One Hour at the Indicated Temperatures, 100X. Values in Parenthesis are Rockwell Hardness Numbers.

As Extruded (42l0 R_c)

3200°F (36.5 R_c)

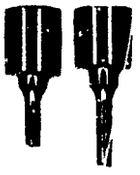


3300°F (36.0 R_c)

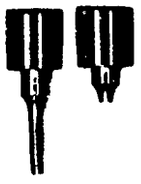


3400°F (35.0 R_c)

Figure 46. Microstructure of Extrusion 4A with Various One Hour Heat Treatments for Recrystallization Study. 100X

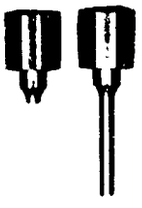


Extrusion 8A
4000°F



Extrusion 11A
4000°F

Extrusion 11A
3000°F



Extrusion 11A
1500°F

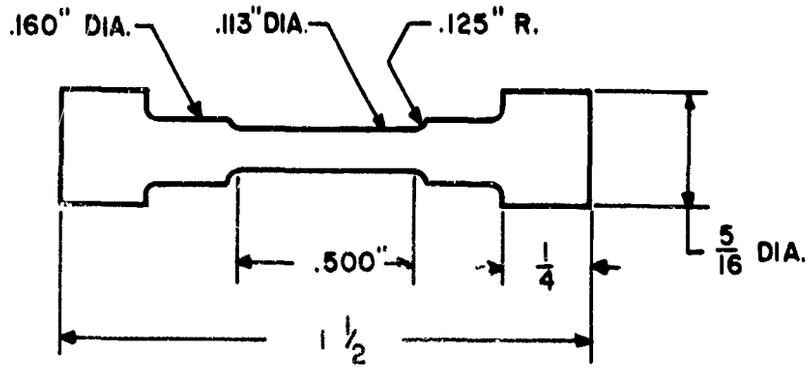


Figure 47. High Temperature Tensile Specimens Used to Determine the Tensile Strength of the 68W-20Ta-12Mo Alloy.

TABLE VII

RESULTS FROM TENSILE TESTS OF THE 68W-20Ta-12Mo ALLOY

Extrusion Number	Description of Nominal Structure	Test Temp (°F)	Stress (ksi) Yield	Ultimate	Fracture	R.A. (%)	Elong. (%)
1A	recrystallized	2000	104	104	129	10.6	8.2
1A	"	2500	54	98	76	17.6	20.5
1A	"	3000	43	52	48	10.4	8.2
1A	"	"	45	56	54	15.8	10.8
1A	"	"	43	49	45	11.2	10.4
1A	"	3500	18	34	28	38.3	25.5
1A *	"	"	20	22	+	38.1	39.3
1A	"	4000	16	17	17	45.1	54.7
1A *	"	"	15	15	+	+	+
3A	recrystallized	2500	48	74	76	2.5	3.3
3A	"	3000	41	50	50	5.0	3.0
3A	"	3500	23	24	22	1.5	1.5
3A	"	4000	16	16	13	4.0	5.4
4A	recrystallized	R.T.	92	92	92	0.0	0.0
4A	"	1500	70	70	72	1.2	0.3
4A	"	1750	74	108	150	15.3	17.2
4A	"	2000	68	108	183	49.1	19.4
4A	"	2500	68	92	50	8.8	14.1
4A	"	3000	55	58	57	5.3	3.7
4A	"	3500	32	34	31	3.6	2.2
4A *	"	4000	16	16	+	13.2	13.4
4A	wrought	R.T.	86	86	86	0.0	0.0
4A	"	1000	101	101	101	0.0	0.0
4A	"	1500	123	123	132	3.5	10.2
4A	"	2000	128	128	185	36.8	16.6
4A	"	3000	67	72	67	15.8	12.0
4A	"	3250	55	58	55	8.8	8.9
7A *	recrystallized	2000	70	81	83	2.3	4.2
7A *	"	2500	102	104	202	65.4	25.7
7A *	"	3000	44	64	112	73.2	60.0
7A *	"	3500	28	29	26	37.7	34.5
7A *	"	4000	20	20	18	30.2	34.0

* No defects in microstructure

** Crosshead speed 0.20 , All other tests 0.040 in/in/min.

Testing accomplished in vacuum.

TABLE VII (continued)

RESULTS FROM TENSILE TESTS OF THE 68W-20Ta-12Mo ALLOY

Extrusion Number	Description of Nominal Structure	Test Temp (°F)	Stress (ksi) Yield	Ultimate	Fracture	R.A. (%)	Elong. (%)
8A	recrystallized	2500	50	81	96	8.1	10.8
8A	"	3000	46	57	61	6.4	11.4
8A *	"	3500	27	27	27	9.4	14.0
8A *	"	4000	18	17	15	9.3	13.0
8A	wrought	2500	107	122	148	14.6	17.2
8A	"	2500	82	121	149	13.4	17.1
8A	"	3000	68	74	64	9.9	7.5
8A	"	3500	22	23	16	1.8	3.6
11A *	recrystallized	1500	71	71	71	0.0	0.6
11A *	"	2000	68	68	70	1.1	2.4
11A *	"	2000	60	60	60	0.6	1.8
11A	"	2000	56	50	56	0.6	1.2
11A	"	2000	66	66	66	2.0	1.2
11A *	"	2500	54	93	199	45.9	24.5
11A *	"	3000	43	56	112	50.5	43.2
11A *	"	3000	40	56	93	45.9	42.9
11A * **	"	3000	54	62	110	41.2	42.8
11A *	"	3500	25	26	27	30.5	58.0
11A *	"	4000	18	18	14	21.2	44.0
11A	wrought	2000	83	111	83	1.2	8.6
Cruc. (1)	As Extr.	3000	-	67	-	23	16

* No defects in microstructure

** Crosshead speed 0.20, All other tests 0.040 in/in/min.

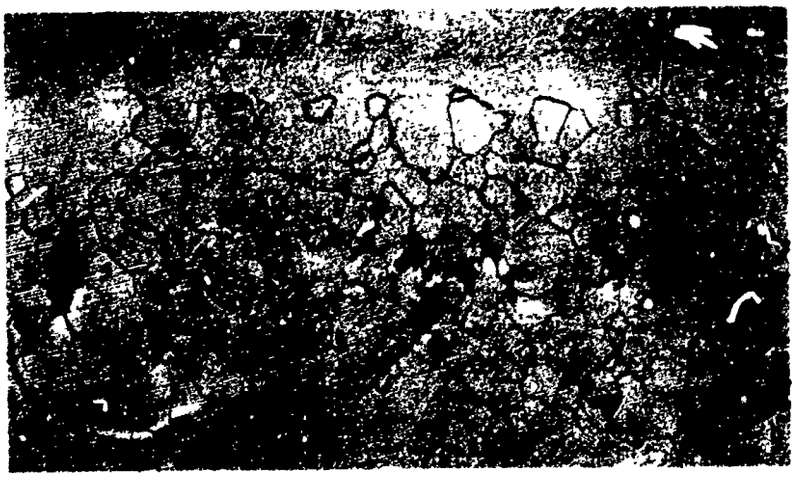
Testing accomplished in vacuum.

tures. Yield stresses decrease quite regularly with increasing temperatures, experiencing a rather rapid drop between 3000°F and 3500°F. Fracture stresses (load at fracture/area at fracture) increase quite abruptly to a maximum of 200,000 psi at 2500°F and then decrease rapidly to about 26,000 psi at 3500°F. This behavior is a result of localized necking prevalent in this temperature range. At higher temperatures the failure mechanism changes. Void formation in the range 3500°F to 4000°F results in failure at appreciably lower values of reduction in area, but at rather high elongations (approximately 30% at 4000°F). Voids formed during testing in this temperature range are typically located at triple points and are reminiscent of those associated with grain boundary shearing in other metals. If such is the case, then this material should exhibit an intermediate temperature ductility minimum. The drop in ductility above 3000°F may well be indicative of such a minimum. Presence of a minimum may explain the excessive scatter in ductility data above 3000°F. That is, the presence of a ductility minimum in any given material is dependent upon one or more as yet unknown and hence uncontrollable factors. One heat of a particular material may exhibit a minimum while another heat, to all intents and purposes, identical to the first, will not. Thus one can reasonably postulate that variations in unidentified factors amongst the various extrusions are responsible for the scatter in ductility data. A second rationalization can be based on the established fact that the minimum can be displaced along the temperature axis by small quantities of other elements. In the present alloy, perhaps variations in the oxygen and/or carbon contents are displacing the minimum.

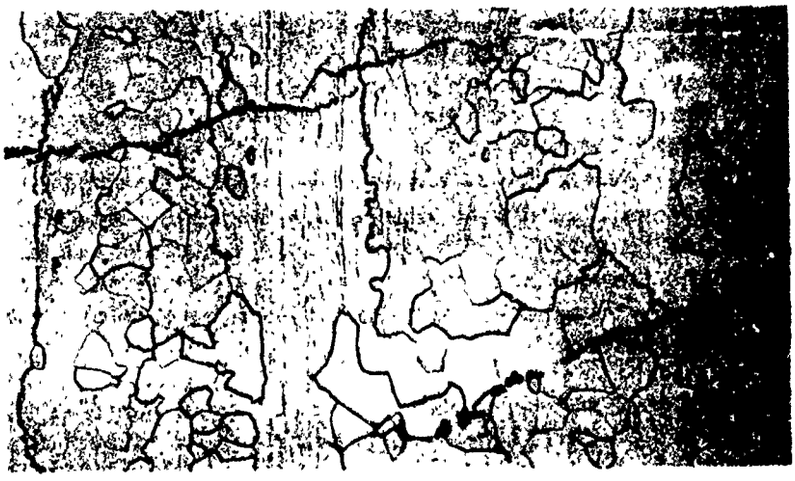
There is good reason to believe that oxygen contents above 45 ppm are very detrimental to ductility (e.g., results from extrusion 3A). On the other hand, carbon contents below 100 ppm appear to have little effect on the mechanical properties of this alloy, and if anything, improve the high temperature tensile properties. However, this is not the whole story. Tensile data indicate that other causes are responsible for some of the poor high temperature tensile results. For example, the best tensile results generally were from extrusions 1, 7 and 11 but these billets did not differ in chemistry from 4 and 8. Spectrographic analyses do not offer any obvious correlation with mechanical properties. Extrusions 2 and 3 were high in Si and Ni and had poor ductility properties; however, 8 and 9 were low in Si and Ni and also had poor ductility.

Metallographic examination of tensile fractures from the various tests revealed little unusual. If the structure was truly recrystallized, then the fractures can be described as follows:

1. For testing temperatures of 2000°F and below the material exhibits little ductility. Hence the individual grains show little departure from equiaxedness. The fracture is usually intercrystalline at 2000°F, but transcrystalline at 1750°F and below.



Porosity



Cracks

Voids at Grain Boundary Triple Points

Figure 48. Three Types of Defects Observed in Extrusions of 68W-20Ta-12Mo Alloy Persisting from the Casting or Generated in Extrusion. 100X

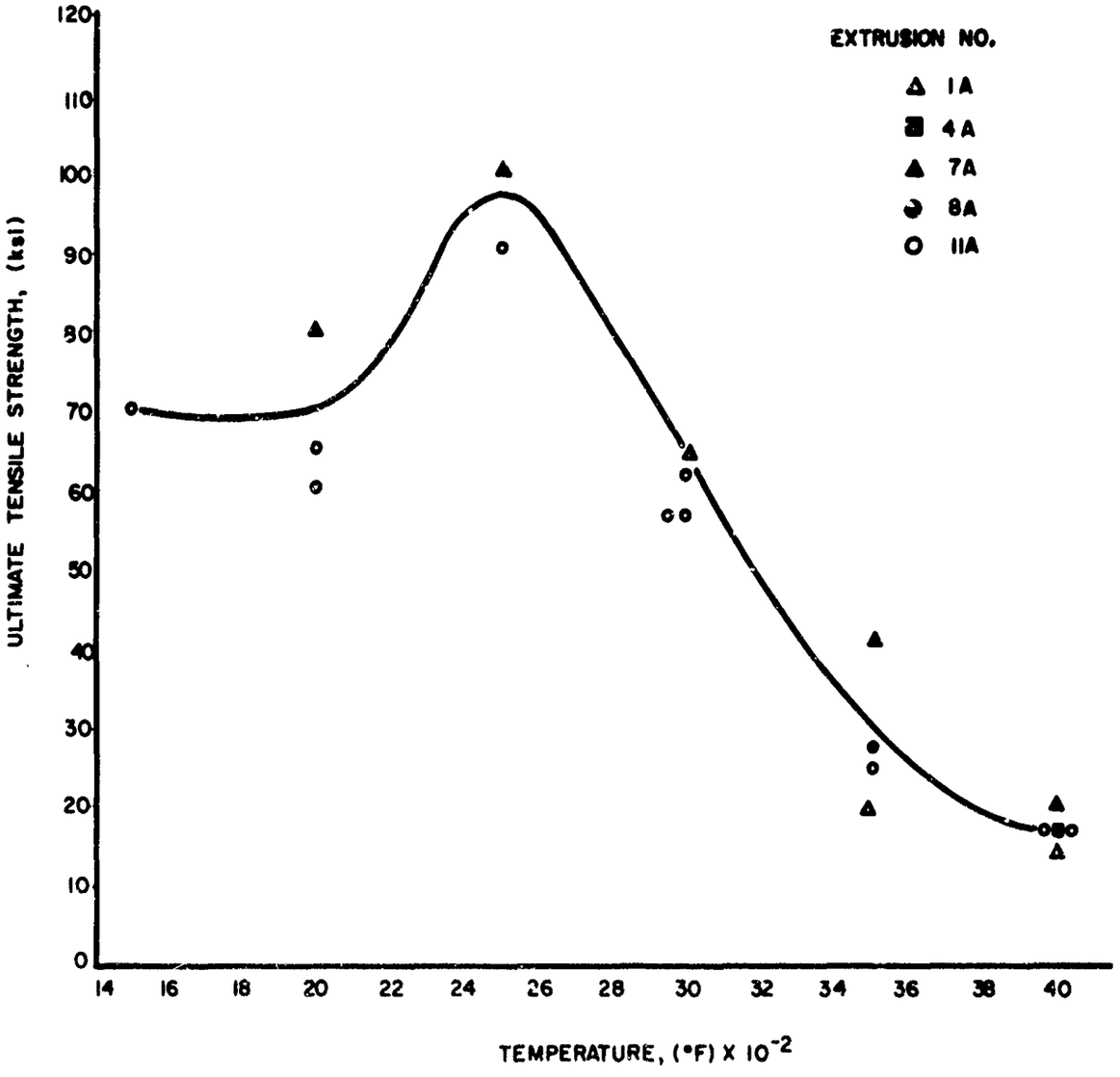


Figure 49. Ultimate Tensile Strength Versus Test Temperature for 68W-20Ta-12Mo Alloy.

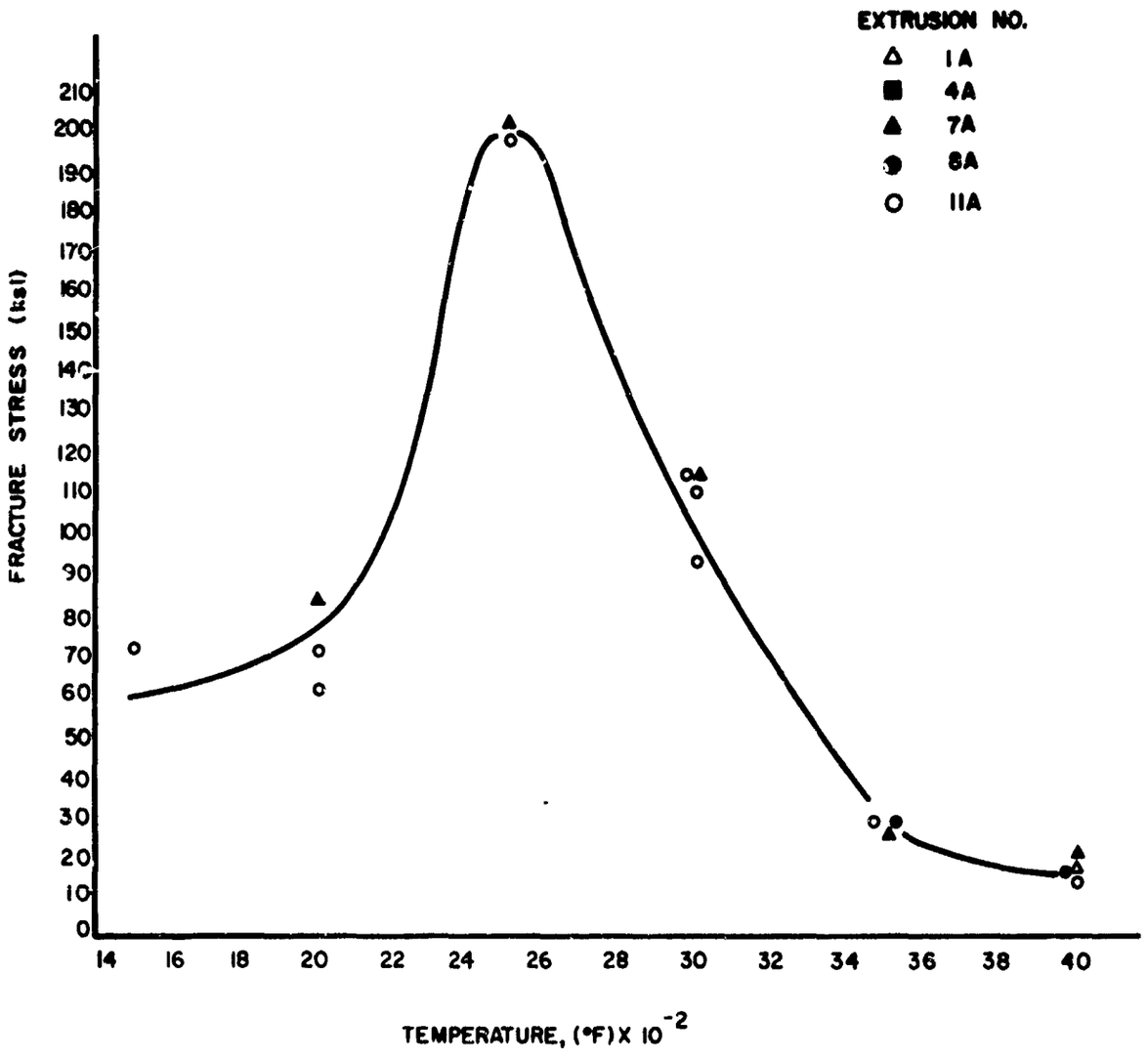


Figure 50. Fracture Stress Versus Test Temperature for Tensile Tests of 68W-20Ta-12Mo Alloy.

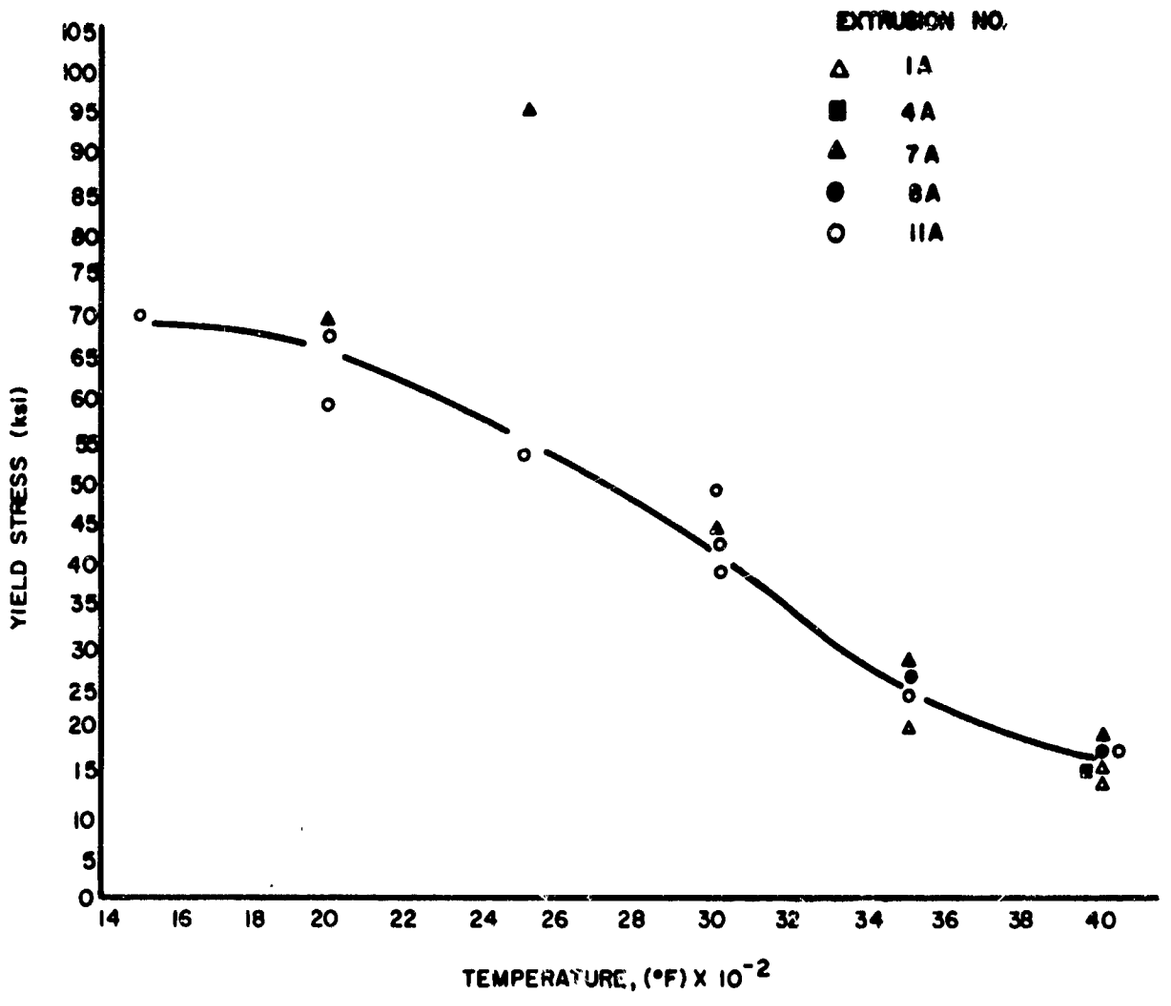


Figure 51. Tensile Yield Strength Versus Test Temperature for 68W-20Ta-12Mo Alloy.

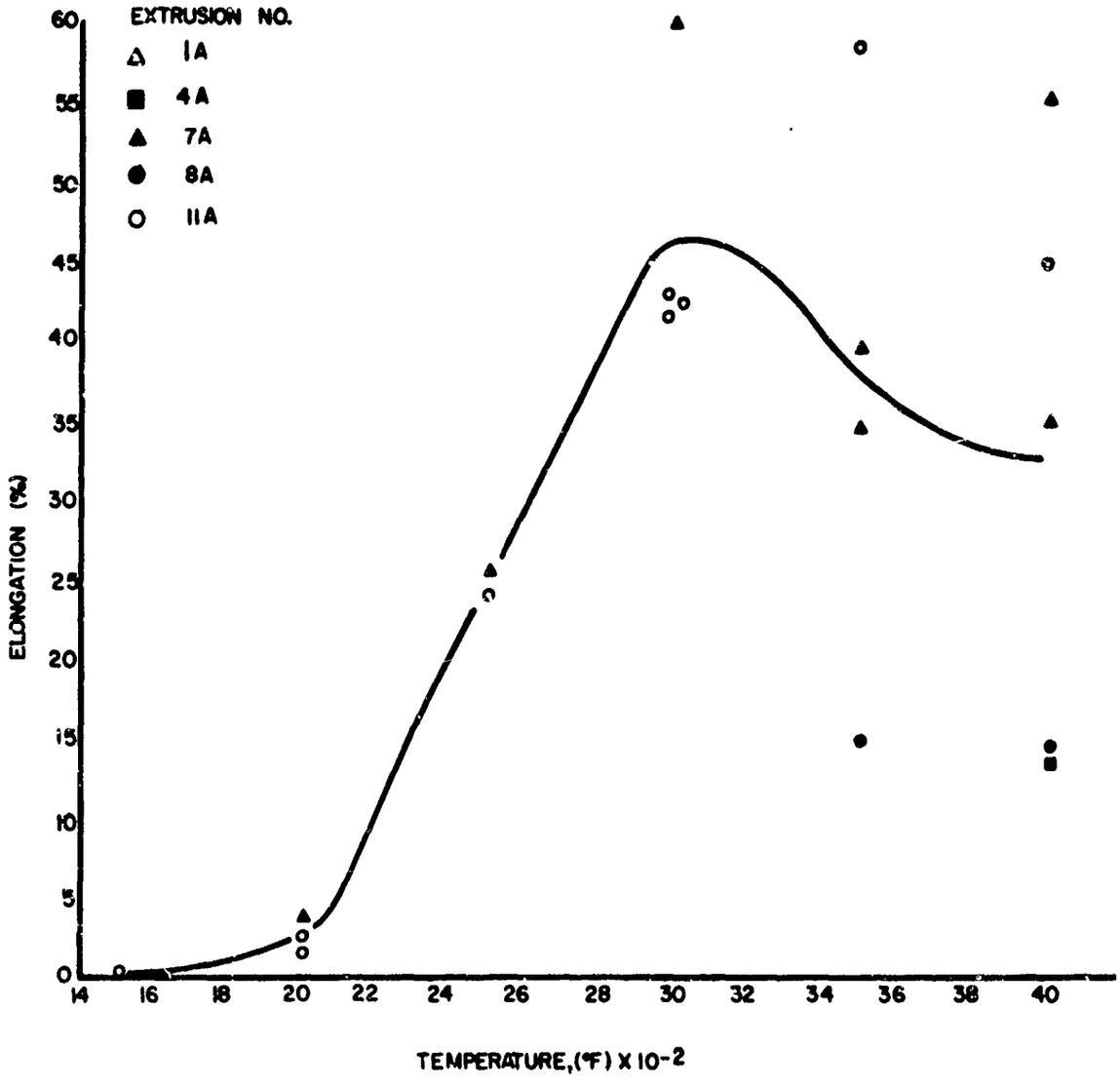


Figure 52. Elongation Versus Test Temperature for 68W-20Ta-12Mo Alloy.

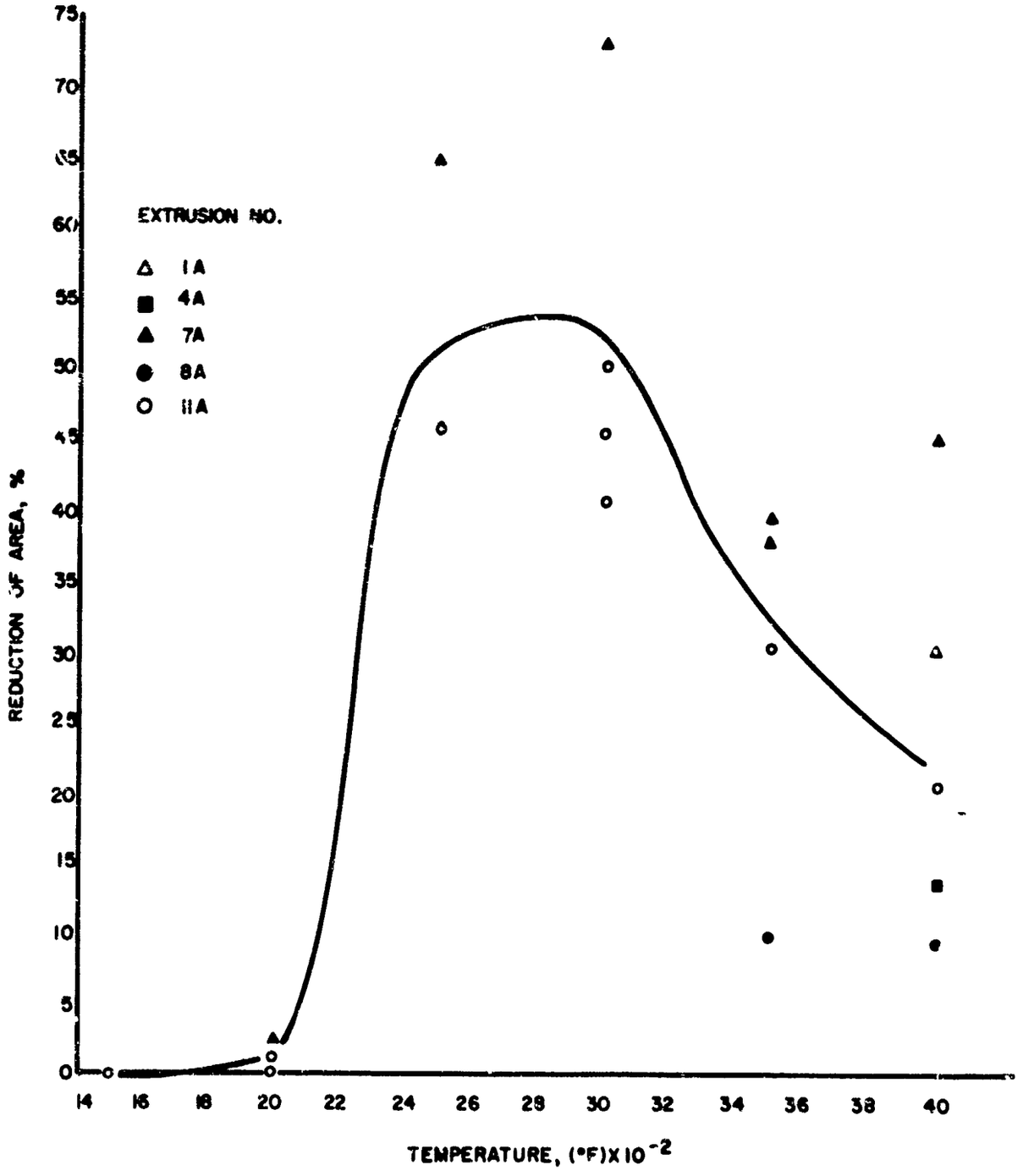


Figure 53. Reduction of Area Versus Test Temperature for 68W-20Ta-12Mo Alloy.

2. The increase in ductility in the region from 2500 to 3000°F is reflected in the marked elongation of the individual grains, especially near the fracture surface. Fractures are mainly transcrystalline and necking is marked.

3. Testing temperatures of 3500°F and 4000°F result in a radically different structure. This structure may be characterized by the following features:

i) rather equiaxed grains indicative, in view of the ductility, of considerable grain boundary motion during or after testing;

ii) serrated grain boundaries identical to those found in other hot-worked materials (Al, Ni, Nichrome, Zr, Mg, U, etc.) and believed to be a result of grain boundary motion due to the formation of bands of deformation;

iii) void formation near the fracture surface becoming less prominent as the distance from the fracture increases.

Initially wrought structures also exhibit a number of fracture types depending upon the testing temperature. At low temperatures (2500°F and below) fractures are typically transcrystalline. Increasing the test temperature to 3000°F and 3500°F results in more or less recrystallization. In this case, the fracture is often intercrystalline through the recrystallized material and transcrystalline in the wrought material. At the highest testing temperature (4000°F) the structure completely recrystallizes during heating and the structures are identical to those described above for fully recrystallized specimens tested at 3500°F and 4000°F.

The photomicrographs in Figure 54 and 55 illustrate the modes of failure in elevated temperature tensile tests of both wrought and recrystallized specimens of this alloy.

b. Creep Rupture Tests

Specimens machined from extrusion 3A were used to determine the 10-hour creep rupture properties of the 68W-20Ta-12Mo alloy in the recrystallized condition. Tests were performed at 3000, 3500, and 4000°F. The specimens are shown in Figure 56. Testing was conducted in a radiant heated Brew vacuum furnace mounted in an arcweld creep rupture rack. The results are summarized in Table VIII.

Upon observing the negligible elongation of specimen #1 and the immediate failure of specimen #2, testing procedures and specimen surfaces were re-evaluated. It may be noted that the loads applied for these first two tests were approximately 50% of the ultimate tensile strength. Upon confirmation that procedures were correct and since the first two samples tested had ground surfaces, the remaining samples were electropolished in the gage area to minimize any possible surface effects. The third specimen was then tested at 3500°F under reduced load. This sample also failed in a very short time. Comparison of these results with the tensile results

2500°F

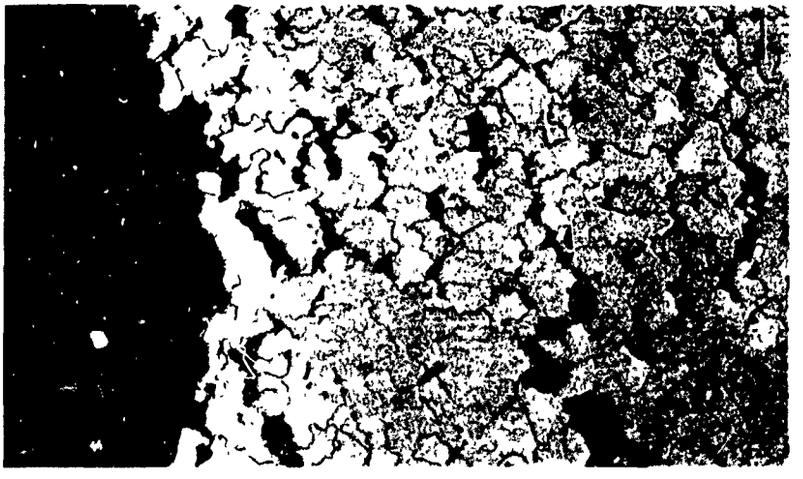
3000°F

3500°F

Figure 54. Photomicrographs of Tensile Specimens Tested at 2500°, 3000° and 3500°F for Extrusion 8A of 68W-20Ta-12Mo Alloy in the Wrought Condition. 100X

2500°F

3000°F



3500°F

Figure 55. Photomicrographs of Tensile Specimens Tested at 2500°, 3000° and 3500°F for Recrystallized 68W-20Ta-12Mo Alloy from Extrusion 11A. 100X

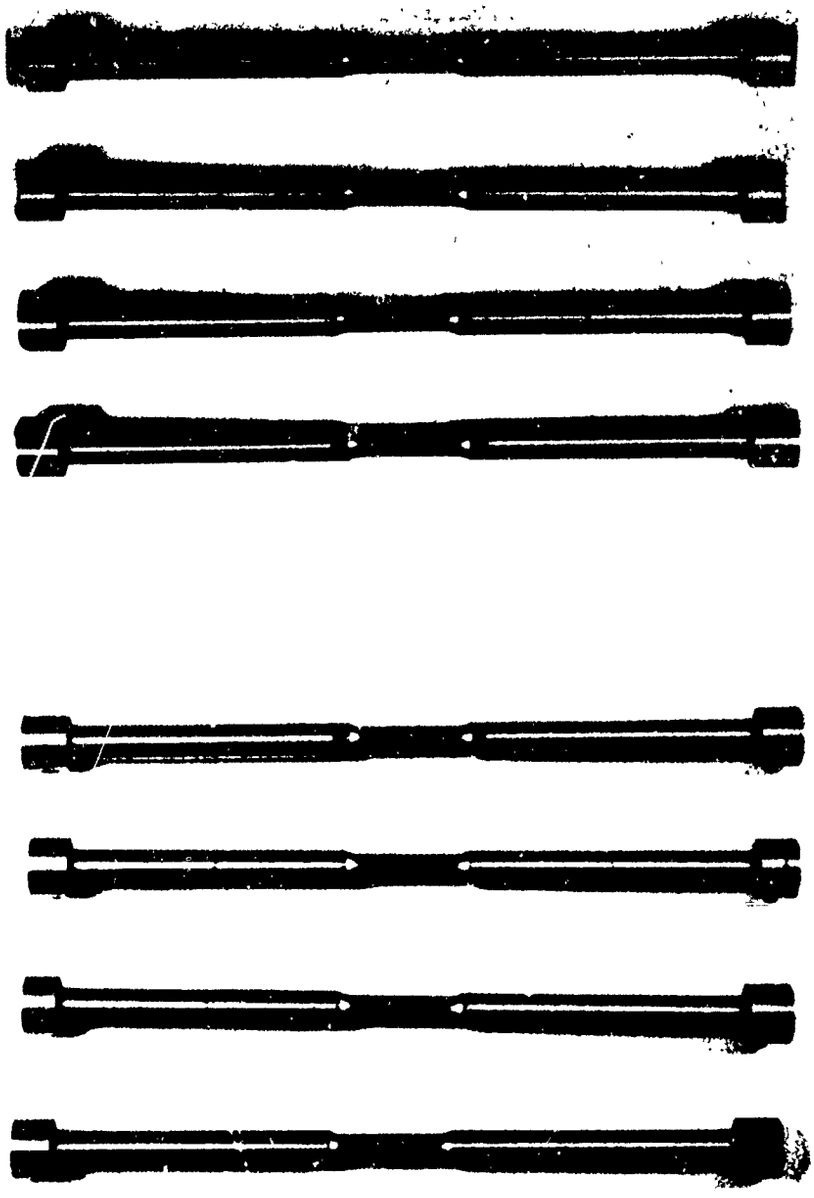


Figure 56. Photograph of the Eight Creep Rupture Specimens Machined from 68W-20Ta-12Mo Extrusion 3A.

TABLE VIII

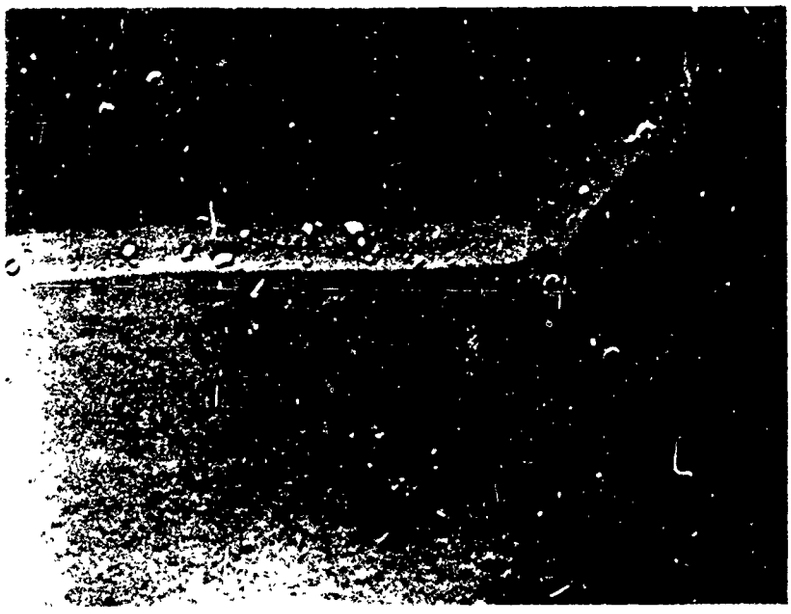
CREEP RUPTURE TESTS OF 68W-20Ta-12Mo ALLOY

Specimen No.	Temp. °F	Sample from Extrusion No.	Stress (psi)	Time to Rupture (hrs.)	Elongation (%)	Reduction in Area (%)
1	3000	3A	20,000	3.39	2.6	2.5
2	3500	3A	20,000	0.00	1.9	2.5
3	3500	3A	10,000	0.20	8.6	3.0
4	3000	1A	25,000	4.60	11.9	4.4
5	3500	3A	5,000	7.93	8.2	N.A.

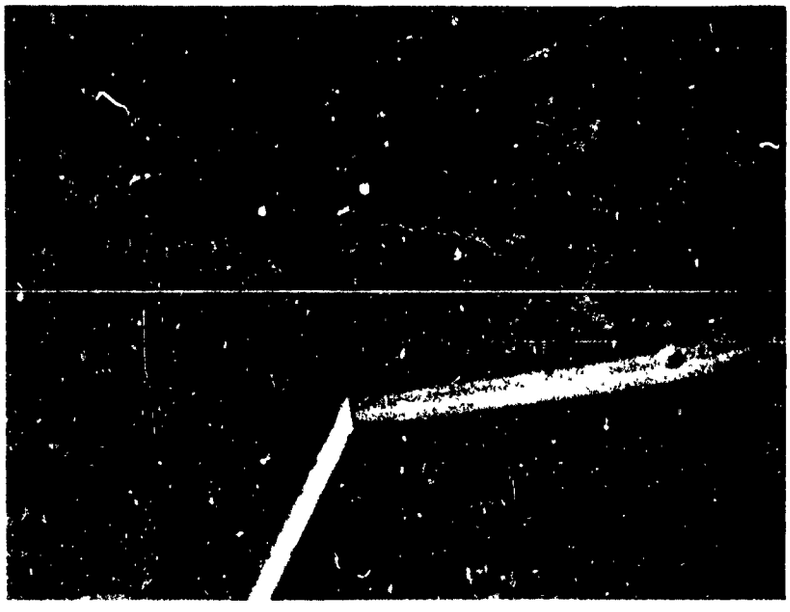
Samples tested in a vacuum Brew furnace mounted in an arcweld creep rupture rack. Samples were given a 1.5 hour - 3400°F heat treatment to impart complete recrystallization.

indicated that the elongation characteristics of extrusion 3A were different from those of the other extrusions. A specimen taken from extrusion 1A was therefore tested for creep rupture properties at 3000°F and 25,000 psi. The 4.60 hour rupture life plus the 10% elongation and 5.5% reduction in area values obtained confirmed the difference between the two extrusions. Metallographic examination of the fractured creep rupture specimens revealed that they failed by grain boundary separation; i.e., intergranular failure. Creep rupture specimen microstructures were examined using electron metallographic techniques. Specimens from 3A were found to contain large amounts of a precipitate at the grain boundaries; whereas material from extrusions 1A and 4A showed much less precipitate at the grain boundaries and these present were fine, discrete particles. The electron photomicrographs in Figures 57 and 58 illustrate the differences observed in extrusions 1A, 2A, 3A, and 4A. Although the precipitate was not identified, the oxygen analysis of the heat from which 2A and 3A were cast was appreciably higher than normal which suggests that the precipitate may be an oxide.

It was concluded that creep rupture testing should be suspended until the alloy had been developed to a more refined status. The effects of variations in interstitial levels on the creep rupture behavior definitely should be investigated when the melting and casting procedures have been established.

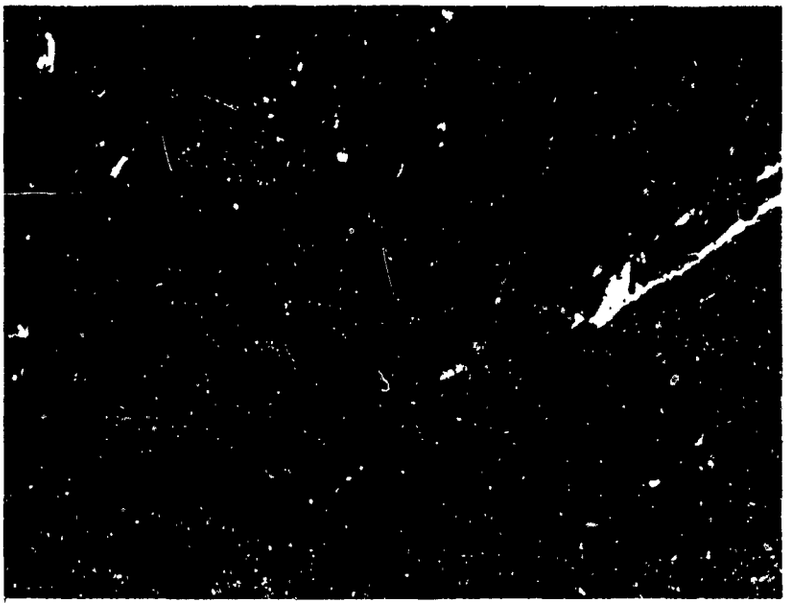


Extrusion 1A - 10,000X



Extrusion 4A - 10,000X

Figure 57. Electron Photomicrographs of Extrusions 1A, and 4A Showing the Relative Amounts of Precipitate in the Grain Boundaries.



Extrusion 2A - 10,000X

Extrusion 3A - 10,000X

Figure 58. Electron Photomicrographs of Extrusions 2A and 3A Showing the Relative Amounts of Precipitate in the Grain Boundaries.

IV Dispersed Phase Alloy - W-12Cb-(V,Zr,C)

A. Ingot Consolidation

1. Melting

The consolidation of the high strength W-12Cb-.29V-.12Zr-.07C alloy was considered an even greater challenge than the solid solution alloy. The centrifugal casting technique was selected as the most likely method for successful casting of the alloy. The material for the first melting was produced from elemental powders that were blended, pressed and sintered, and AC vacuum arc melted to produce a second melt casting electrode.

Severe difficulties were encountered at Oremet in attempting to melt this electrode in the skull furnace. In three attempts it was impossible to strike and maintain an arc between the arc melted electrode and the copper skull casting crucible. During the final attempt, a burn-through developed in the crucible. No further attempts were made to centrifugally cast this alloy. It is believed that the failure to sustain a stable arc was due to vanadium vapors.

To obtain material for preliminary extrusion trials and heat treatment studies, the 5 inch diameter AC arc melted electrode was examined for possible value as an extrusion billet. One section of suitable size was shipped to TRW for this purpose.

Using AC arc melting techniques the consolidation of the second melt electrode from the pre-alloyed pressed and sintered electrode was accomplished with no arc difficulties. For this reason, in view of the difficulties in casting, the decision was made to produce ingots by the AC arc melting technique.

Two ingots of the W-12Cb-0.29V-0.12Zr-0.07C alloy 3.5" diameter by 12" long were single AC arc melted at Oregon Metallurgical Corporation and have been processed into extrusion billets. The electrodes used to produce those two ingots were produced from powders blended to the nominal composition and double sintered in both hydrogen and vacuum. The melted ingots are shown in Figure 59. Each ingot was sectioned and ground to provide two extrusion billets. Examination during sectioning showed that both ingots were severely cracked.

In an effort to obtain sound material another ingot was melted with sections of W-15Mo on top and bottom sections. It was theorized that the more ductile W-15Mo alloy on the top and bottom of the ingot would reduce the cracking tendency in the starting pad and hot top areas during solidification and cooling. This ingot is shown in Figure 60 with the macroetched top and bottom sections which were removed from the ingot. It is apparent that the W-15Mo sections did not provide the desired prevention of cracking.

Figure 59. Photographs of the AC Arc Melted Ingots of the W-12Cb-0.29V-0.12Zr-0.07C Alloy in the As-Cast Condition.

Figure 60. Ingot No. 3 of the W-12Co-.29V-.12Zr-.07C Alloy as AC Arc Melted with W-15Mo Sections on Top and Bottom.

2. Chemical Analyses

The chemical analyses of the first two heats of the dispersed phase alloy from which Billets 1B, 2B and 3B were machined, are tabulated in Table IX. These analyses were obtained from samples removed from approximately the center of the ingots. The composition of the electrode was very well established to bring the major elements into good control immediately. The vanadium and to a lesser extent the zirconium content was high in Billet 1B. However, the billet was removed from the casting electrode and thus it would be anticipated that additional vanadium and zirconium losses would have occurred had Oremet been able to make the casting melt. The Billets 2B and 3B analyses represent an AC arc melted ingot for which excellent control was shown.

3. Microstructures

The inability to maintain an arc for the first casting melt at Oremet necessitated the use of the arc melted electrode for further processing studies. The microstructure of the AC arc melted electrode was more fine grained than might be expected with a refractory metal ingot as can be seen in Figure 61. Darker etching fields of the precipitated phase generally appear to follow the grain boundaries in the lower magnification view. Examination at higher magnifications revealed that the precipitate was generally dispersed throughout the grains as well as at the grain boundaries.

The following heats of this dispersed phase alloy were AC arc melted. Metallographic samples of the top, middle, and bottom ingot locations revealed uniform, fine grained microstructures similar to the electrode from which the first billet was prepared. Representative photomicrographs for the two ingots appear in Figure 62. (Two billets were machined from each of the ingots.) The dispersed phase was uniformly distributed throughout the matrix.

B. Deformation Studies

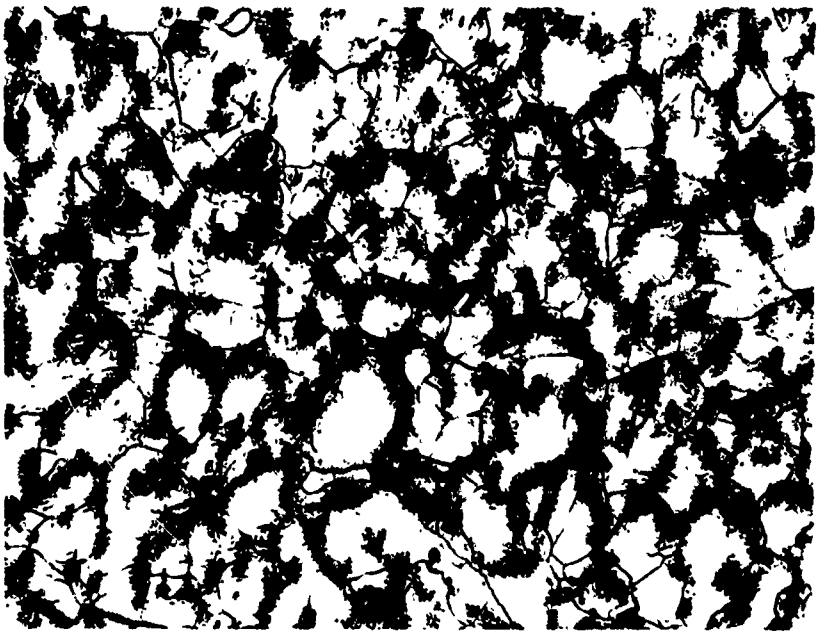
The initial billet (1B) of this alloy was prepared from a section of the AC arc melted electrode intended for remelting in the skull casting furnace. The billet machined from this section had an obvious fracture through the nose as well as local defects. These are apparent in the photographs in Figure 63.

The billet was "canned" in Mo-0.5% Ti as described for the solid solution alloy billets. This composite extrusion billet was induction heated to the highest temperature possible with the furnace - 4150°F. The attempt to make a round extrusion with a 5.5 to 1 area reduction stalled the press even at this temperature. The examination after this attempt showed that the billet had sustained more severe cracking. It was, however, remachined and encased in Mo-0.5% Ti for a second attempt at a lower extrusion ratio. This extrusion at a 4 to 1 ratio at 4150°F produced the bar shown in Figure 64. The extrusion data are recorded in Table X.

TABLE IX

CHEMICAL ANALYSES OF DISPERSED PHASE ALLOY NOMINAL W-12Cb-.29V-.12Zr-.07C

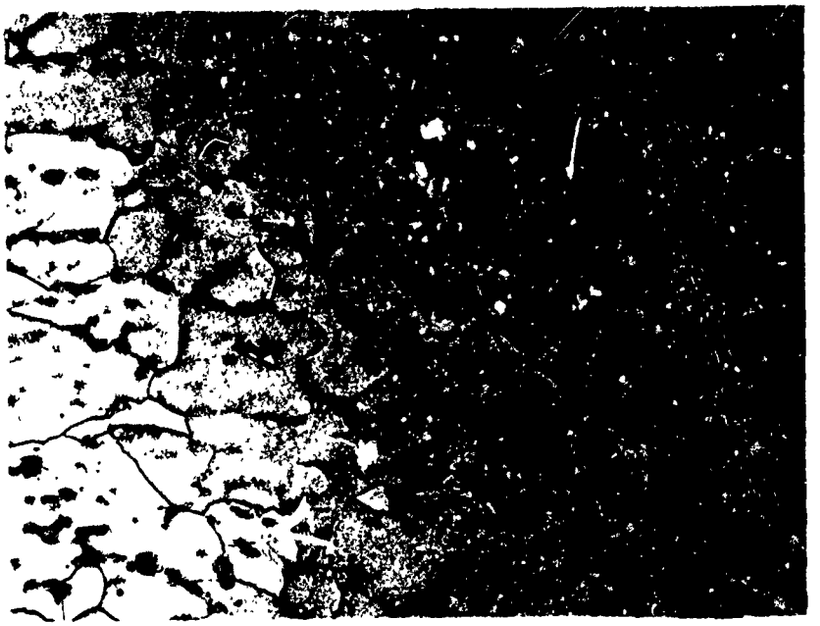
	Billet 1B	Billets 2B & 3B	Analytical Technique
Tungsten	86.15%	Balance	Wet
Columbium	13.0%	14.8%	Wet
Vanadium	.59%	.29%	Wet
Zirconium	.18%	.13%	Wet
Carbon	.076%	.062%	Leco Conductometric
Nitrogen	130 ppm	10 ppm	Micro Kjeldahl
Oxygen	70 ppm	20 ppm	Inert Gas Fusion
Hydrogen	-	1 ppm	Vacuum Fusion



100X

1000 x

Figure 61. As-Cast Microstructure of W-12%Cb-0.29%V-0.12%Zr-0.07%C Consumable Electrode Vacuum Arc Cast Ingot.



Ingot 1 Billets 2B and 3B

Ingot 2 Billets 4B and 5B

Figure 62. Microstructure from Center of Ingot 1 (Billet 2B-3B) and Ingot 2 (4B-5B) in the AC Arc Melted Condition, W-12Cb-0.29V-0.12Zr-0.07C Alloy. 100X

Figure 63. W-12Cb-0.29V-0.12Zr-0.07C Alloy Billet 1B Finish Ground and with Dye Penetrant Developer.

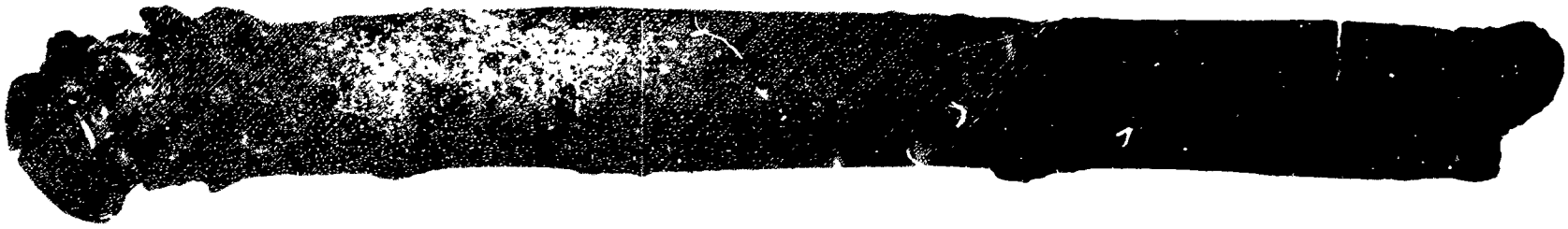


Figure 64. W-.2Cb-0.29V-0.12Zr-0.07C Alloy Extrusion 1B. Billet was Arc Cast, Canned in Mo-0.5%Ti and Extruded at 4150°F, 4 to 1 Area Reduction Ratio.

TABLE X

EXTRUSION CONDITIONS FOR W-12Cb (V,Zr,C) ALLOY

	1B	2B	3B	4B	5B
Temperature (°F)	4150	4050	4065	3950	3800
Extrusion Ratio	4:1	4.5:1	3:1	3.2:1	3.8:1
Ram Speed, ips	12	-	-	15	15
Breakthrough Pressure, psi	165,000	Stalled	Stalled	179,000	184,000
Min. Running Pressure, psi	142,000	-	-	179,000	184,000 (Stalled)

All Billets "Canned" in Molybdenum except 2B and 3B which had .035" Thick Molybdenum Spray Coating.

The extruded bar was cracked rather severely as the photograph indicates. The examination of bar sections revealed that the microstructure was approximately 50% very fine recrystallized grains, Figure 65. Thus, the high temperature necessary to deform this alloy in the extrusion operation also allowed the recrystallization process to proceed to an appreciable degree.

The second extrusion billet, 2B, was prepared similar to the first except that a molybdenum powder spray coating was applied in place of the "can". The billet was heated to the highest temperature attainable which was 4050°F. The extrusion attempt at a 4.5 to 1 reduction in a round die stalled the press. The third billet, 3B, was also molybdenum powder spray coated for extrusion. A lower reduction ratio, 3 to 1, was attempted at the highest temperature attainable, 4065°F. This extrusion also stalled the press before any significant amount of bar was produced.

The fourth and fifth dispersed phase alloy billets were prepared as the first one - that is they were "canned" in Mo-0.5% Ti. The temperatures attained in the induction furnace for these billets, 4B and 5B, were 3950° and 3800°F, respectively, as recorded in Table X. Although 4B was forced through the extrusion die for a 3.2 to 1 reduction at about the maximum pressure available (184,000 psi), it was very severely cracked, separating into the molybdenum nose section and four alloy segments as the photograph in Figure 66 illustrates. The photomicrographs representative of extrusion 4B show in Figure 67 large wrought grains and 10 to 20 percent fine recrystallized grains with an elongated precipitate phase in the unrecrystallized areas.

The fifth billet was only partially extruded as a result of the lower temperature (and higher reduction ratio) attained. The very short length of bar, the extrusion die and unextruded billet are also pictured in Figure 66. It was concluded that the combination of defective billets (resulting from ingot cracking) and the extremely high strength with low ductility prevented the successful deformation of these arc melted billets.

C. Metallurgical Evaluation of W-12Cb-(V, Zr, C) Alloy

Although the consolidation and deformation difficulties encountered in scaling up this alloy were formidable, some material was produced in bar form to permit a limited evaluation of metallographic and mechanical property characteristics.

1. Recrystallization Studies

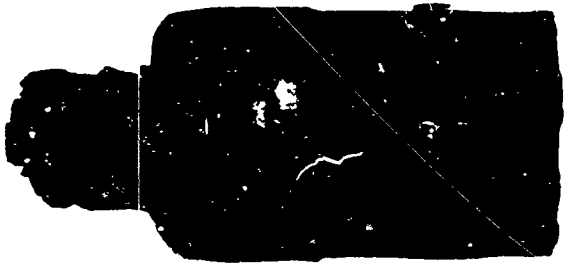
The material from extrusion 1B was of only limited value for recrystallization studies since the structure was approximately 50% recrystallized as extruded. However, small samples of this bar were treated in vacuum for one hour periods at temperatures ranging from 3700°F to 4100°F. This study revealed that the bar was not completely recrystallized with the highest temperature treatment - Figure 68. As shown in the following section, however, the structure was completely recrystallized after 4 hours at 4200°F.

100X

500X

Figure 65. Longitudinal Microstructure of Extrusion 1B As-Extruded W-12Cb-0.29V-0.12Zr-0.07C Alloy.

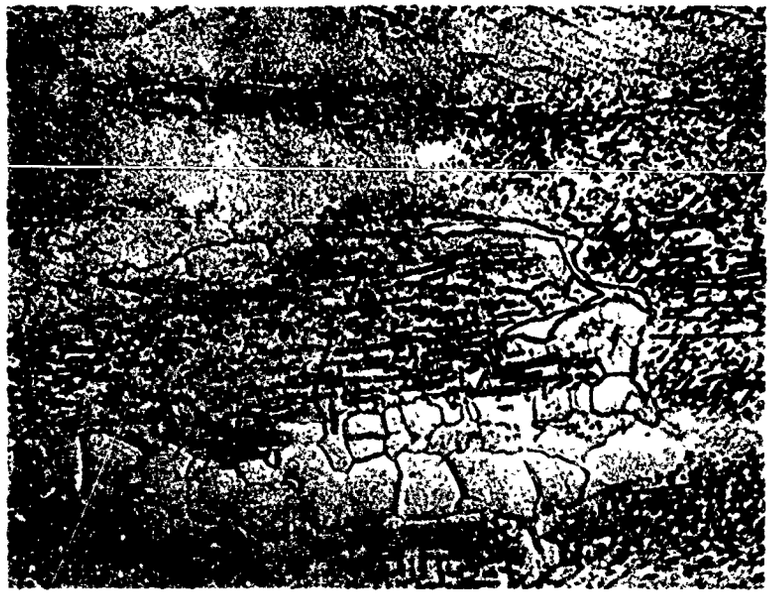
4B 3.2 to 1 Reduction at 3950°F



5B 3.8 to 1 Reduction at 00°F

Figure 66. Dispersed Phase Alloy (W-12Cb-(V,Zr,C) Alloy) Extrusion Results for Billets 4B and 5B in Mo-0.5%Ti "Cans".

100X



1000X

Figure 67. Photomicrographs of W-12Cb-(V,Zr,C) Alloy Extrusion 4B As Extruded.

3700°F

4000°F

4100°F

Figure 68. Longitudinal Microstructures of Extrusion 1B, W-12Cb-0.29V-0.12Zr-0.07C Alloy After 1 Hour Heat Treatment in Vacuum at the Indicated Temperatures. 100X

2. Mechanical Property and Metallographic Evaluation

The small amount of material available from extrusion 1B permitted some elevated temperature tensile testing of the dispersed phase alloy. The tests were made with the bar in the partially recrystallized as-extruded condition. The specimens were prepared and tested by the same techniques described for the solid solution alloy. The results for these six tests in the range from 2500°F to 4000°F are recorded in Table XI and plotted in Figure 69. These strength results are significantly higher than reported values for alloys in the 3000°F to 4000°F range[6] exceeding the 68W-20Ta-12Mo alloy by about 80% to 90% in this range. The 4000°F tensile strength is approximately six times that of unalloyed tungsten. These results are not considered optimum since they represent the as-extruded (50% recrystallized) condition. It is reasonable to expect to develop a dispersed phase structure by heat treatment which will provide better strength and ductility combinations than was obtained in the extruded condition.

Metallographic examination of the fractured tensile specimens revealed that elongation at 3500°F and higher temperatures was accompanied by void formation - Figure 70. The section of the specimens tested at 4000°F clearly shows the difference in deformation and fracture behavior of the recrystallized and wrought areas. Relatively few voids are apparent in the wrought structure. Thus, the elongation values in the 3500°F and higher temperature tests are increased through this mechanism.

The data obtained with the dispersed phase alloy have confirmed the extremely high temperature strengths available with this tungsten base alloy system. The consolidation and deformation problems prevented a more thorough study of the alloy.

TABLE XI

RESULTS FROM TENSILE TESTS OF THE W-12Cb-0.29V-0.12Zr-0.07C ALLOY

Specimen No.	Temp. (°F)	Ultimate Tensile Strength (psi)	.2% Offset Yield Strength (psi)	Elongation (%)	R.A. (%)
1	2500	107,000	71,800	.4	3.7
2	3000	86,000	64,200	18	18
3	3000	86,000	66,000	34.3	28
4	3500	45,200	34,800	30	36
5	3500	48,700	41,000	74	37
6	4000	30,000	24,650	36	27.5

Strain rate = .040 in/in/min.

Testing accomplished in vacuum

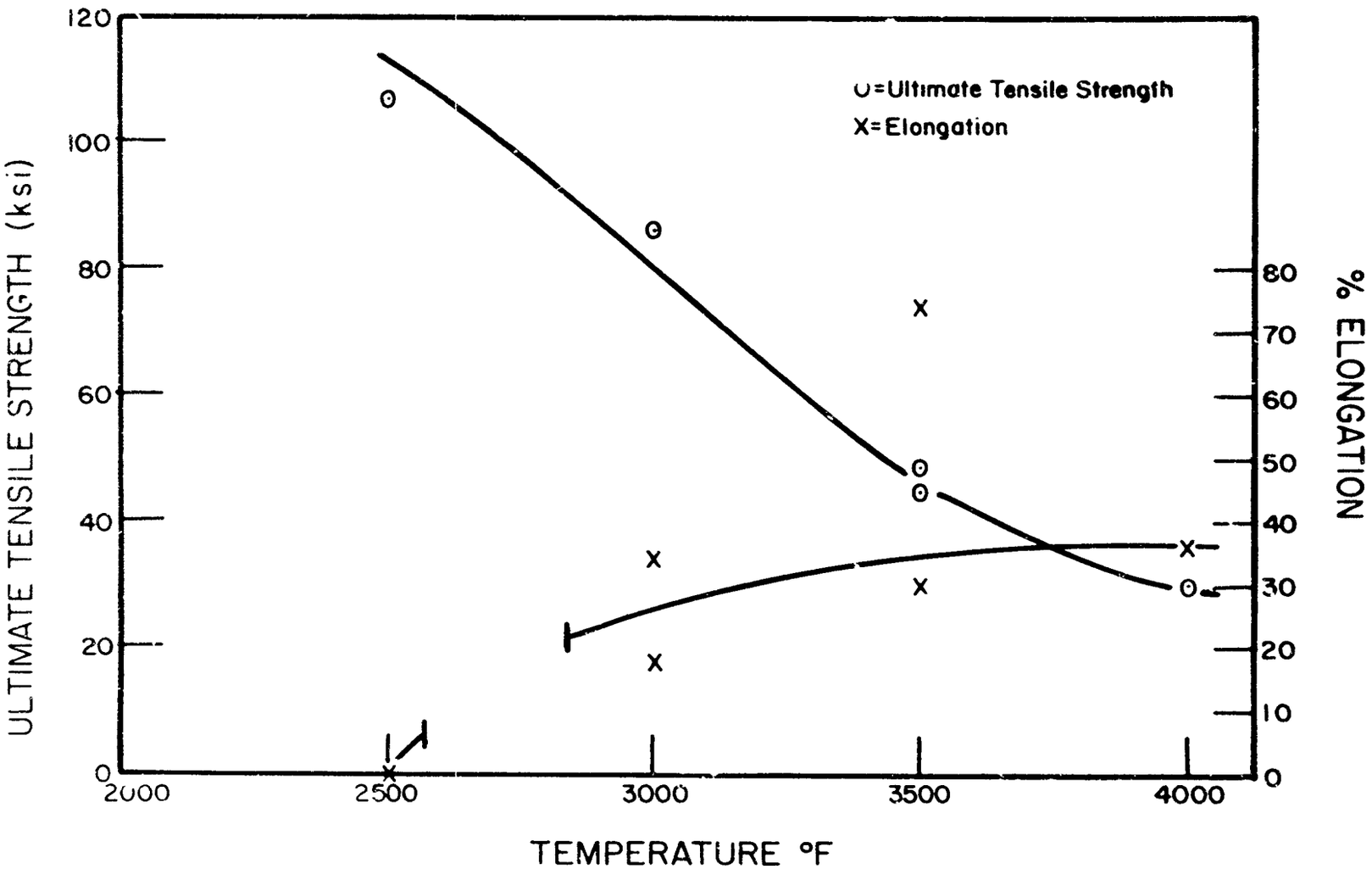


Figure 69. Tensile Properties vs. Test Temperature for the W-12Cb-0.29V-0.12Zr-0.07C Alloy. Specimens Taken from Extrusion 1B, Extruded at 4150°F at 4 to 1 Area Reduction Ratio. Specimens in As-Extruded Condition. Strain Rate 0.04 in/in/min.

2500°F

3000°F

3500°F

4000°F

Figure 70. Longitudinal Microstructures of the Fracture Surfaces of Tensile Specimens Taken from Extrusion 1B, W-12Cb-0.3W-0.12Zr-0.07C Alloy. Testing Temperatures as Indicated. 100X

V CONCLUSIONS

1. The consumable electrode vacuum arc melting technique employing a water cooled copper crucible is not a satisfactory procedure for melting three inch diameter (or larger) ingots of the highly alloyed tungsten and tantalum base alloys. The following compositions melted by this technique were severely cracked: 68W-20Ta-12Mo, 20W-68Ta-12Mo, 68W-20Ta-12Cb, and 44W-44Ta-12Cb.

2. The vacuum arc melting-centrifugal casting technique employing a graphite mold was modified and refined and resulted in a sound casting of the 68W-20Ta-12Mo alloy three inches in diameter by approximately six inches long. Recycling of skulls, sprues and turnings from the vacuum centrifugal castings was accomplished and interstitial contaminants generally decreased with recycling. Fine grained, clean microstructures were obtained in the centrifugal castings. Additional repetitive casting is necessary to establish the reliability of the method for casting this alloy.

3. An extrusion process was successfully applied for producing round bar of the 68W-20Ta-12Mo alloy and to a considerable degree welded the cracks present in the castings in the process. A minimum temperature of about 3700°F and a reduction ratio of about 5.5 to 1 are necessary to obtain a wrought structure. The only unsuccessful attempt to extrude this alloy was encountered when trying to effect a 5 to 1 reduction to a rectangular cross section at 3900°F.

4. Secondary working techniques including side forging, rolling and swaging were generally unsuccessful for 68W-20Ta-12Mo alloy material in either the wrought or recrystallized conditions. An exception was that a side forging reduction of 10% was accomplished with recrystallized material from the final heat. Upset forging of the alloy was successfully accomplished with wrought and recrystallized material to 50% and 45% reductions in height respectively.

5. Metallographic studies indicated that the one hour recrystallization temperature for the 68W-20Ta-12Mo extrusion was 3300°F.

6. Tensile tests of the extrusions representing wrought and recrystallized structures were made over the temperature range from ambient to 4000°F. The effects of strain hardening are evident at temperatures up to 3000°F. The yield strength in the recrystallized condition was approximately 43,000psi at 3000°F, 27,000psi at 3500°F and 18,000psi at 4000°F. The material exhibits little ductility at testing temperatures of 2000°F and below, reaches a maximum at 2500°-3000°F and decreases at higher temperatures.

7. The evidence from tensile tests and deformation studies of the 68W-20Ta-12Mo alloy indicated that oxygen in excess of 40 ppm has a very detrimental effect; carbon, if anything, improved the high temperature tensile properties. Spectrographic analysis do not offer any correlation with mechanical properties.

8. DC arc melting for centrifugal casting of the tungsten-12% columbium base dispersed phase alloy was prevented by arc instability. AC arc melting produced ingots which were severely cracked.

9. Extrusion of the tungsten-12% columbium base dispersed phase alloy required a temperature of 4150°F for a 4 to 1 area reduction.

10. Bar which was approximately 50% recrystallized as-extruded was not completely recrystallized after one hour at 4100°F.

11. Tensile tests of the dispersed phase alloy extrusion showed yield strengths of about 65,000psi at 3000°F, about 38,000psi at 3500°F and about 25,000psi at 4000°F. The ductility of the alloy was low at 2500°F, increasing with temperature to a maximum at 3500°F.

12. It is strongly indicated by this research that such ultra high-strength, high temperature alloys are not amenable to production into mill product forms within the present state-of-the-art. Further advancements are required in metallurgical knowledge of the alloys and in industrial facilities capability. The first effort, prerequisite to the development of such alloys into useful wrought product forms, will require an intense study of means for the initial successful consolidation into ingot or billet form.

VI RECOMMENDATIONS

1. The high strengths shown in the tensile tests and the successful upset forging of the 68W-20Ta-12Mo alloy indicate that further research should be performed on the W-Ta-Mo system.

2. Modifications and refinements in the centrifugal casting process resulted in the successful production of a 3-inch diameter ingot of the 68W-20Ta-12Mo alloy. Additional study of the melting of the high strength refractory metal compositions is a necessary prerequisite to the development of these systems.

3. The detrimental effect of oxygen and possible beneficial effect of carbon on the properties of the 68W-20Ta-12Mo alloy indicate that subsequent to the solution of the consolidation problem, an effort should be directed towards understanding the effects of the interstitial elements on the mechanical properties and structure of the material.

4. Variations in the alloy compositions including the addition of rhenium to both the solid solution and dispersed phase alloy systems studied in this program should be investigated.

5. Results from this program emphasize the importance of understanding the relationships between structure (as influenced by thermal-mechanical treatments) and high temperature properties in a solid-solution alloy. A research program to study the thermal-mechanical processing of solid solution alloys with emphasis on these relationships should be conducted with a convenient material.

VII REFERENCES

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VIII APPENDIX

VACUUM CENTRIFUGAL CASTING

WORK STATEMENT

TRW Processing and Material Work Statement No. 2323-4
Oregon Metallurgical Corp.
Centrifugal-Cast Tungsten-Tantalum-Molybdenum Alloy Ingots

March 3, 1964

The intent of this work statement is to insure quality, uniformity, and reproducibility of the centrifugal-cast tungsten-tantalum-molybdenum (68%W-20%Ta-12%Mo) alloy ingots procured by Thompson Ramo Wooldridge Inc. for an experimental alloy development program.

A. Starting Materials

1. The second melt electrodes shall be prepared from consumable electrode vacuum arc melted ingots melted with a clustered electrode of tungsten, tantalum, molybdenum, and/or reprocessed material from past melting attempts.

2. The tungsten electrode component shall be a pressed-sintered and swaged rod made from a single powder lot.

3. The tantalum electrode component shall be a rod made from a single electron beam melted ingot.

4. The molybdenum electrode component shall be strips cut from a single mill heat of 1/4" plate.

5. The reprocessed material shall be that salvaged from the initial melting attempt made for Thompson Ramo Wooldridge Inc.

B. Ingot Melting

1. All first-melt electrodes shall be fabricated using tungsten electrode inert gas welding techniques.

2. All first-melt electrodes shall be A.C. vacuum consumable electrode melted into a 4" diameter crucible.

3. First-melt ingots shall be machined to remove contaminated metal, vacuum arc melted into a tiltable 9" water-cooled crucible, and cast into a rotating graphite mold.

C. Mold Design

1. The mold shall be designed such that two billets will be obtained from each casting attempt.

2. One of the two molds shall be constructed with a thin (1/2") graphite liner backed with a vacuum gap and stainless steel reflector - the purpose being to obtain a slow and uniform cooling rate.

3. The second of the two molds shall be similar to the first, but with a 3/4" thick graphite liner.

D. Heat Treatment

1. No heat treatment shall be performed on ingots produced to this work statement without prior approval by TRW.

E. Chemistry

1. Target alloy composition is 68%W-20%Ta-12%Mo. Alloying additions of the three elements shall be made to maintain this composition.

2. Care shall be used to maintain the lowest interstitial composition possible.

3. All reprocessed material shall be etched to remove any contaminated surface layers.

4. Chemical samples will be taken from each ingot during machining at TRW and returned to Oremet for analysis. The elements to be determined and analytical methods required are listed below:

Element	Method
C	Leco Conductometric
O	Inert Gas Fusion
H	Pressure Equilibrium
N	Micro Kjeldahl
Al	Spectrographic
Co	"
Cr	"
Cu	"
Fe	"
Mg	"
Mn	"
Ni	"
Pb	"
Si	"

Element	Method
Ti	Spectrographic
V	"
Sn	"
Cb	"
Zr	"
W	X-Ray Spectrographic
Ta	" "
Mo	" "

F. Inspection

1. The ingots shall be of a diameter and length such that they will clean up after rough machining to finished billets 2.060 inch diameter by 4-1/4 inch long.

G. Ingot Shipment

1. The complete casting assembly sprue with ingots attached shall be suitably packed for shipment in a container with sawdust.

2. The ingots shall not be shipped without certification that they were manufactured and processed in exact accordance with this work statement.

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11 SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	Research and Technology Division Wright-Patterson Air Force Base, Ohio

13 ABSTRACT A deformation study and evaluation of high strength tungsten base alloys was accomplished. The two alloys studied included the solid solution strengthened 68% W-20% Ta-12% Mo alloy and the dispersed phase strengthened W-12% Cb-0.29% V-0.12% Zr-0.07% C alloy. The vacuum melting plus centrifugal casting technique was used for the consolidation of the solid solution alloy. A new type of ingot mold was developed to cast 3 inch diameter ingots. High temperature extrusion was successfully accomplished to provide 68% W-20% Ta-12% Mo bar for evaluation. Studies of upset and side forging, rolling and swaging of the solid solution alloy were made. Upset forging was the only successful technique for secondary working. The extruded bar was used to determine recrystallization behavior, tensile and creep rupture properties. The one-hour recrystallization temperature for the 68% W-20% Ta-12% Mo bar was 3300°F. The 3000°F, 3500°F and 4000°F tensile strengths of the recrystallized bar were approximately three times those of unalloyed tungsten.

The W-12% Cb-(V, Zr, C) alloy was consolidated by AC arc melting due to arc instability in DC melting. The three-inch diameter ingots couldnot be produced without cracking. High temperature extrusion produced sufficient sound bar to evaluate tensile properties. This dispersed phase strengthened alloy exhibited the highest strengths at elevated temperatures known to have been reported to date, for refractory metals.

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