REFRACTORY ALLOY FOIL ROLLING DEVELOPMENT PROGRAM

Interim Report No. 2
15 October 1962 - 15 January 1963

Manufacturing Technology Laboratory
Aeronautical Systems Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

ASD Project No. 7-987

The melting and testing of 6" ingots of each of the following alloys are described: D-43 (Cb-10%W-1%Zr-0.1%C), B-66 (Cb-5%Mo-5%V-1%Zr), Cb-752 (Cb-10%W-2-1/2%Zr), Ta-10%W and T-111 (Ta-8%W-2%Hf).

(Prepared under Contract AF33(657)-8912 by E. I. DuPont de Nemours & Company, Inc., Metals Center, Baltimore, Maryland.)

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FOREWORD

This Interim Technical Documentary Progress Report covers the work performed under Contract AF33(657)-8912 from 15 October 1962 to 15 January 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with E. I. DuPont de Nemours & Company, Inc., Baltimore, Maryland was initiated under Manufacturing Methods Project 7-987, "Refractory Alloy Foil Rolling Development Program". It is being accomplished under the technical direction of Mr. H. L. Black of the Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Mr. John Symonds, Development Engineer, Metals Center, Baltimore, is the engineer directly responsible for the work. Others who cooperated in the work were Dr. A. W. Dana, Jr., Technical Supervisor and Mr. W. F. Bumgarner, Production Supervisor.

The primary objective of the Air Force Manufacturing Methods Program is to develop on a timely basis manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. This program encompasses the following technical areas:

> Alloy Selection (Columbium, tantalum and tungsten alloys), Consolidation Techniques, Primary Breakdown, Rolling to Heavy Gauge Sheet, Foil Rolling.

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

PUBLICATION REVIEW

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ABSTRACT

The melting of three columbium-base alloy and two tantalum-base alloy ingots by arc-casting is described. The alloys were Cb-10%W-1%Zr-0.1%C (D-43), Cb-5%Mo-5%V-1%Zr (B-66), Cb-10%W-2-1/2%Zr (Cb-752), Ta-10%W, and Ta-8%W-2%Hf (T-111). The process consisted of hydrostatic compaction of elemental powders into electrodes, followed by double consumable arc melting to yield 6" diameter ingots. Analytical and metallurgical testing of the ingots is described.

TABLE OF CONTENTS

APPENDIX

FIGURES

TABLES

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I. INTRODUCTION

This report summarizes the work performed on Phase II of Contract No. AF33(657)-8912, entitled "Refractory Alloy Foil Rolling Development Program". The contract is sponsored by the Manufacturing Technology Laboratory, ASRCTB, AFSC Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

The program objective is to develop manufacturing processes for the production of tungsten, columbium and tantalum and/or their alloys in 24" wide foil, down to 0.001" thick. The program has been divided into five phases as follows:

1. The evaluation of the current state-of-the-art of refractory alloy foil rolling and the recommendation of the most promising alloys of tantalum, columbium, and tungsten for the remainder of the program.
2. The production and testing of ingots of columbium and tantalum alloys required for the manufacture of foil 12" wide by 100' coils and the investigation of the rolling of pure tungsten.
3. The production of coil blanks for each of the alloys selected and the evaluation of these coil blanks.

4. The rolling of foil in thicknesses from 0.001" - 0.005" and widths of 12" and the evaluation of the product.
5. The production of 24" wide coils of the approved alloys in the thickness range of 0.001" - 0.005".

At the conclusion of Phase I (State-of-the-Art Survey) the following compositions were selected for rolling to 12-inch wide foil:

1. Cb-10%W-1%Zr-0.1%C (D-43 alloy - alloy previously designated X-110)
2. Cb-10%W-2-1/2%Zr (Cb-752 alloy)
3. Cb-5%Mo-5%V-1%Zr (B-66 alloy)
4. Ta-10%W
5. Ta-8%W-2%Hf (T-111 alloy)
6. Pure tungsten

Processing of the columbium and tantalum base alloys under this program commences with consolidation and proceeds through the various processing steps to foil as outlined under the five phases of the contract. Tungsten sheet will be purchased from a number of sources for rolling to foil.

The Phase II program described in this report is concerned mainly with melting and testing of the 6" diameter columbium and tantalum base ingots required for rolling to 12" wide foil. The operations involved are: blending, compaction, and double consumable arc melting. Testing and evaluation procedures include: ultrasonic testing for soundness; chemical, spectrographic, and X-ray microprobe analyses; and metallographic examinations.

The tungsten portion of the program is at present concerned with design studies on various arrangements for continuous rolling of tungsten sheet with rapid heating at the roll-nip.

II. SUMMARY

The objective of Phase II was to develop casting techniques capable of producing 6" diameter ingots in the three columbium and two tantalum base alloys for subsequent processing to foil. An additional objective of the program was to carry out preliminary rolling evaluations of tungsten sheet from various sources.

Ingot Casting

Initial consolidation of the raw materials was accomplished by hydrostatic compaction. The compacts were tack-welded together to form first melt electrodes. First melt ingots (4-1/2" diameter) were inverted and used as electrodes for second melting. The second melt electrodes were joined by studding and welding.

Ingot soundness was determined by ultrasonic inspection. Shrinkage pipes detected by this means were cropped off.

A 1/4" thick test slice was taken from the top and bottom of each ingot. Chemical, spectrographic and microprobe analyses were carried out on these slices. The chemical and spectrographic analyses indicated the compositions of the five ingots to be satisfactory.

It was considered that compositional homogeneity was especially desirable in these ingots since they are being rolled to foil gauges. Microprobe analyses were carried out on specimens in the as-cast condition and after a high temperature homogenization treatment. Compositional variations were most marked in the columbium base alloys and were typically $\pm$ 25% of the average. Distances between adjacent maxima and minima in composition were of the order of 100 microns. The two tantalum alloys showed much less pronounced heterogeneity. The high temperature homogenizations did not result in more uniform distribution of the alloying elements in any of the alloys.

Metallographic examination and hardness measurements carried out before and after the high temperature treatments did not indicate significant changes in microstructure.

Tungsten Processing

Design studies are being carried out on two methods of rapidly and continuously heating tungsten which might be used in rolling of long lengths of tungsten strip or foil. The device would be installed on the entry side of a rolling mill, immediately adjacent to the rolls.

III. INGOT CASTING

Raw Materials

Information is given in Table 1 on purity and physical form of the elemental materials used in both the columbium and tantalum base alloys.

First Melt Electrode Preparation

Charges for each of the five ingots were blended in a twin-shell cone blender and loaded into rubber boots for compaction. The rubber boots, evacuated by a vacuum pump prior to compaction, were hydrostatically compacted at 60,000 psi.

First Melt Ingots

The compacted electrodes were melted to 4-1/2" diameter ingots using the consumable electrode process in the Heraeus vacuum arc furnace at the Du Pont Metals Center. Compacts were tack-welded to each other (and to the adapter) to give first melt electrodes typically 30" long. In the case of the two tantalum alloys, the tack-welds were reinforced by tacking 1/8" tantalum rod across the joints. Without this additional strengthening, the tack-welds were extremely susceptible to breakage in handling and while starting the arc.

Melting data for the first melt ingots are summarized in Table 2.

TABLE 1

RAW MATERIALS FOR COMPACTION OF COLUMBIUM AND TANTALUM BASE ALLOYS

Element	Supplier	Physical Form	Analyses Oxygen ppm	Nitrogen ppm	Carbon ppm	Hydrogen ppm	Others
Columbium	Du Pont	Granules, -20 +60 mesh	51-56	2	19-23	65-84	Ta <1000 ppm
Molybdenum	Universal Cyclops	Chips (*)	20	17	247	20>	
Vanadium	Union-Carbide	Particles, -1/4"	400	460	310	10>	
Zirconium	Carborundum Co.	Sponge, -1/8" +1/16"	850	58	500>	10>	
Tungsten	General Electric	Powder, -80 +150 mesh	64	16	7	20>	
Columbium Carbide	Fansteel	Powder, -325 mesh	1730	2760	(12.1%)	-	
Tantalum	National Research Corp.	Powder, -12 +100 mesh	279	38	44	17	
Hafnium	Carborundum Co.	Electrolytic crystal, -1/4"	414	41	7	7	Minimum purity 98.0%; principal impurity - Zr

*Obtained by machining an ingot.

TABLE 2

MELTING DATA FOR FIRST MELT 4-1/2" DIAMETER INGOTS

Alloy	Electrode dia., inches	Volts	Amps	Melt Rate lbs./min.	Furnace Pressure Microns
D-43	3	32	6000-6500	5.2	0.2-0.5
B-66	3	32	6500	8.5	0.1-0.3
Cb-752	3	32	7000	5.1	0.1-1.0
Ta-10%W	2	36-38	6500-7500	1.8-2.2	3.0-19.0
T-111	2	30-34	6500	2.0-3.0	3-10

Prior to melting, the furnace pressure was reduced to less than two microns, with a leak rate of less than one micron per minute (13 micron-liters per second). A magnetic stirring coil was used throughout the melts.

Melting of the T-111 alloy was started with 3" diameter compacts but arcing to the sidewall and a crucible burn-through occurred. The problem was eliminated by changing to 2" diameter compacts.

The high pressures obtained in the melting of the two tantalum base alloys are noteworthy. High pressures also obtained in second melting of these alloys. This is believed to be associated with the large volumes of gas removed as indicated by the high degree of interstitial upgrading which took place in these alloys. (See Table 5).

Second melt electrodes were prepared from the 4-1/2" diameter ingots. Two first melt ingots were required to obtain a second melt electrode. The 4-1/2" diameter ingots were inverted and the bottom of one was sawn square, drilled, tapped and joined to the adapter by a stud. The original top of this ingot and the bottom of the second ingot were likewise sawn flat, drilled, tapped and studded. This joint and the joint between the adapter and the ingot were further reinforced by a circumferential tack weld.

Second Melt Ingots

Final 6" diameter ingots were melted in the Heraeus furnace. Melting conditions are summarized in Table 3.

TABLE 3

MELTING DATA FOR SECOND MELT 6" DIAMETER INGOTS

Alloy	Volts	Amps.	Melt Rate lbs./min.	Furnace Pressure Microns
D-43	32	9000	11	0.1
B-66	32	7500-8000	15	0.1
Cb-752	36	9000	10	0.1
Ta-10%W	34-40	11,500-13,000	4	5.0-5.5
T-111	32-38	14,000-15,000	9	4.0-8.0

The crucible used was 6-3/8" diameter. Magnetic stirring was used for each melt. A hot topping procedure was carried out at the end of each melt to minimize the shrinkage cavity. Power was reduced incrementally over a period of 8-12 minutes. Currents were in the range 3000-5000 amps at arc-out.

Ingot Conditioning

Both ends of the as-cast ingots were cropped. The bottoms were cropped to remove the unmelted portion of the powder starting pads and the tops were cropped to remove any visible shrinkage pipe. The columbium alloy ingots were turned down to 5.57" diameter, the dimension required for steel canning prior to extrusion. The two tantalum ingots, to be extruded bare, were machined to a larger diameter - 5.72". At this diameter some surface porosity was still present in the bottom 2" of the ingots. These defects were ground out by spot conditioning.

Ultrasonic Testing

The machined ingots were ultrasonically tested for internal soundness. Test conditions are summarized in Table 4.

TABLE 4

ULTRASONIC TESTING CONDITIONS OF COLUMBIUM AND TANTALUM BASE ALLOY INGOTS*

Alloy	Crystal	Frequency
D-43	Branson 'Z' crystal	5 Mc.
B-66, Cb-752	Quartz	2.25 Mc.
Ta-10%W, T-111	Branson 'Z' crystal	2.25 Mc.

*Instrument: Curtiss Wright 'Immerscope' (immersion technique)

The portions of the ingot tops containing concealed shrinkage cavities (revealed by ultrasonic testing) were removed by sawing. The lengths of defect-free ingot obtained in each of the alloys was:

D-43 alloy	-	11-13/16"
B-66 alloy	-	11-1/4"
Cb-752 alloy	-	9-1/4"
Ta-10%W alloy	-	11-9/16"
T-111	-	10-1/2"

Ingot Evaluation

One-quarter inch thick slices were obtained from the top and bottom of each ingot. Each slice was subjected to the following series of evaluations:

1. Macro-etch on a 90° segment.
2. Microstructure.
3. Chemical analysis.
4. Spectrographic determinations of major alloying elements.
5. A high temperature homogenization treatment was given to a portion of each slice.
6. X-ray microprobe analytical scans before and after the homogenization heat treatment.
7. Hardness measurements before and after the homogenization treatments.

The macrostructures of the five ingots are illustrated in Figures 1-5.

Microstructures at a mid-radius location are illustrated in Figures 6-10. The B-66 alloy microstructure contained some very fine micro-porosity visible in Figure 7. The subsequent hot-breakdown operation is expected to eliminate this porosity completely.

D-43 alloy ingot.

Macro-structure, top

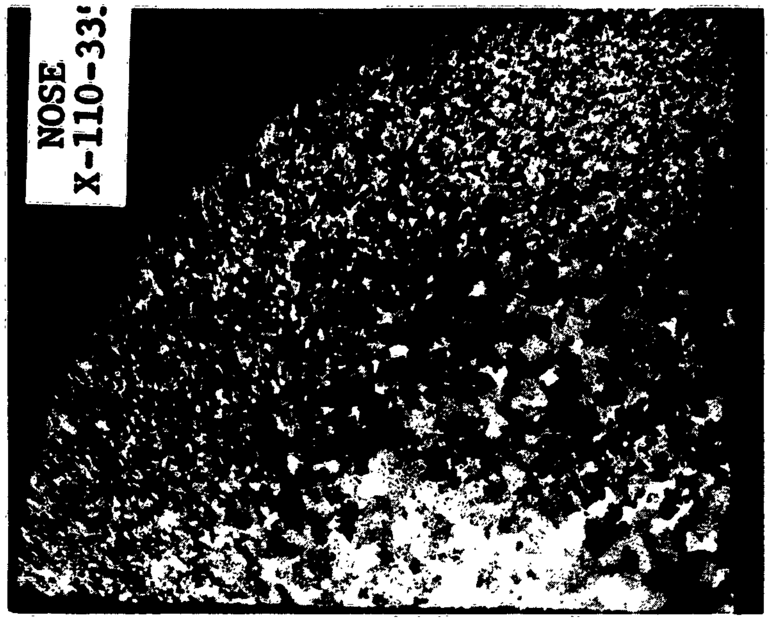
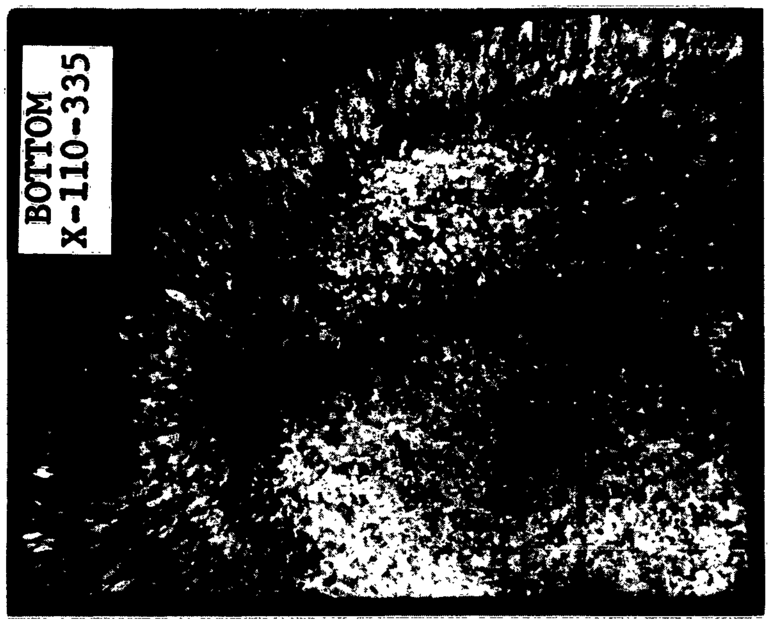


FIGURE 1

D-43 alloy ingot.

Macro-structure, bottom



B-66 alloy ingot.

Macro-structure, top

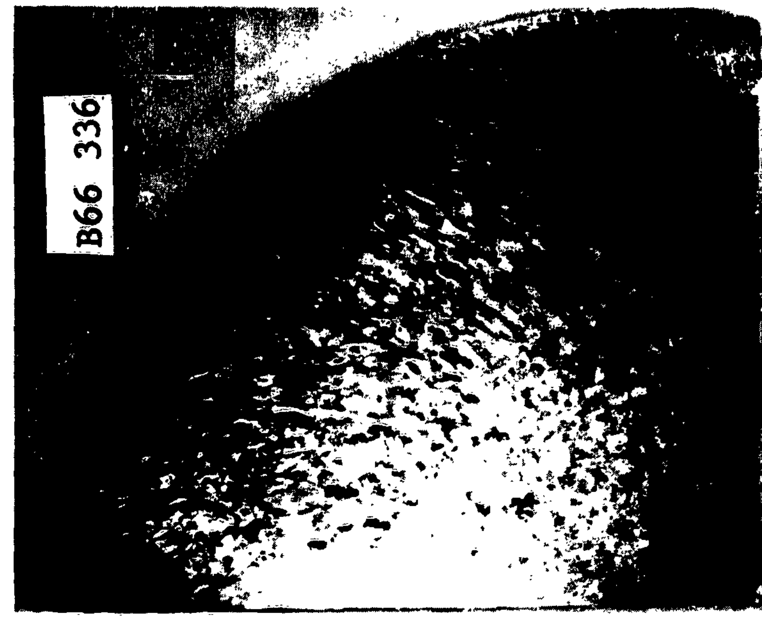


FIGURE 2

B-66 alloy ingot.

Macro-structure, bottom

Cb-752 alloy ingot.
Macro-structure,
top

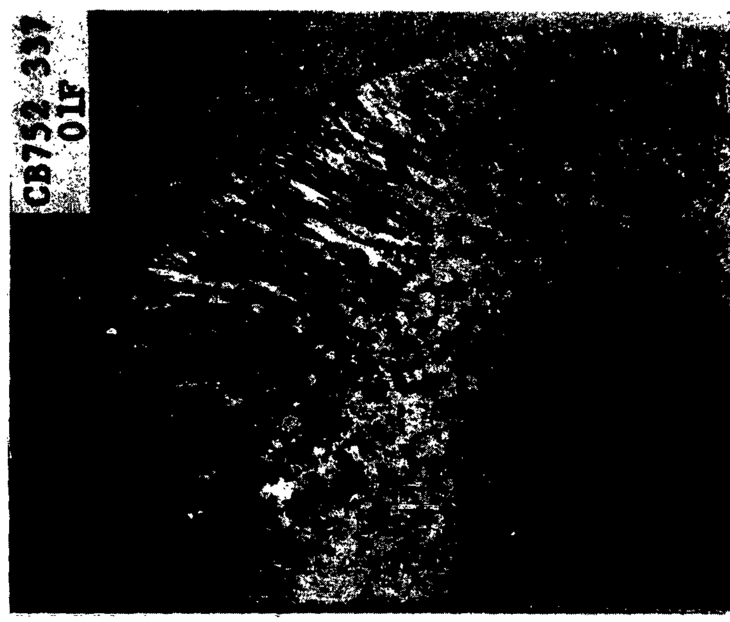
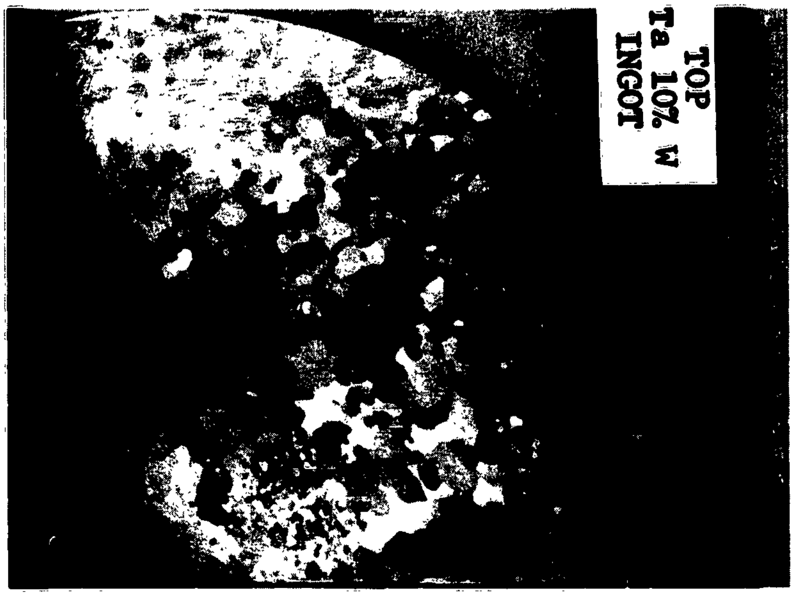


FIGURE 3



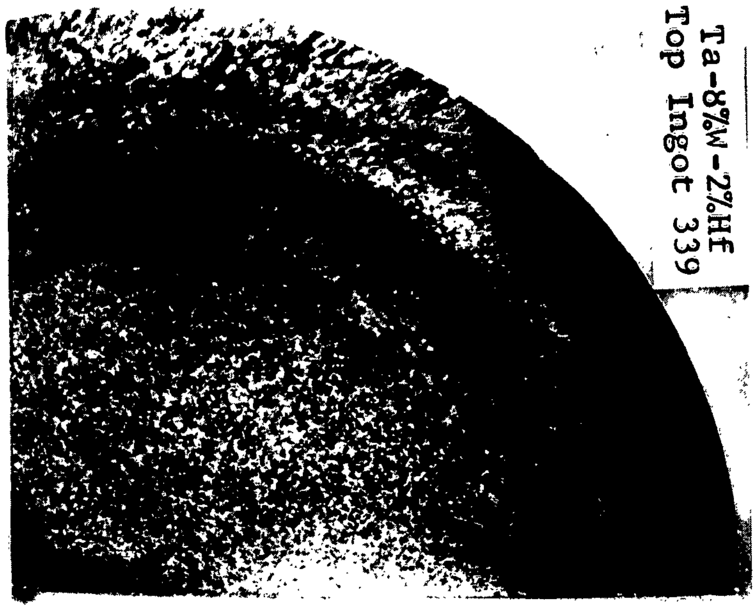
Ta-10%W alloy ingot.

Macro-structure, top

FIGURE 4

Ta-10%W alloy ingot.

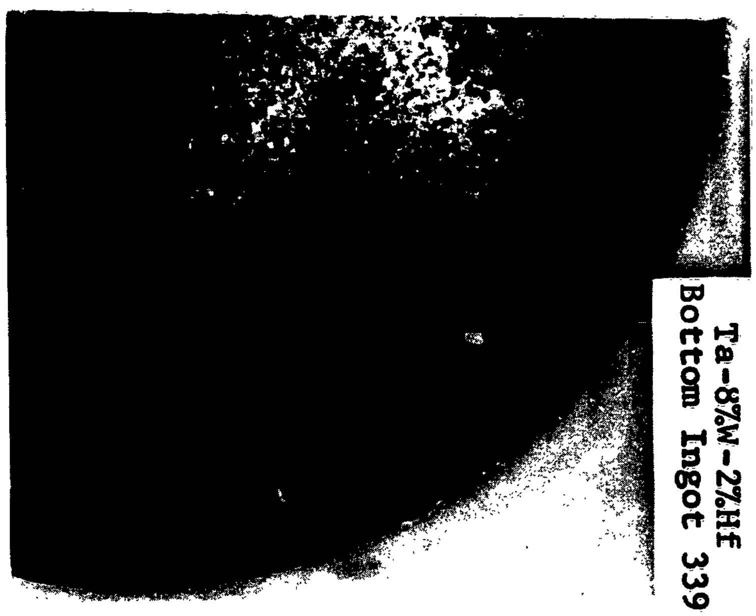
Macro-structure, bottom



T-111 alloy ingot.

Macro-structure,
top

FIGURE 5



T-111 alloy ingot.

Macro-structure,
bottom

As-cast micro-
structure.

D-43 alloy,
ingot top.

X250

Hardness R_A 54

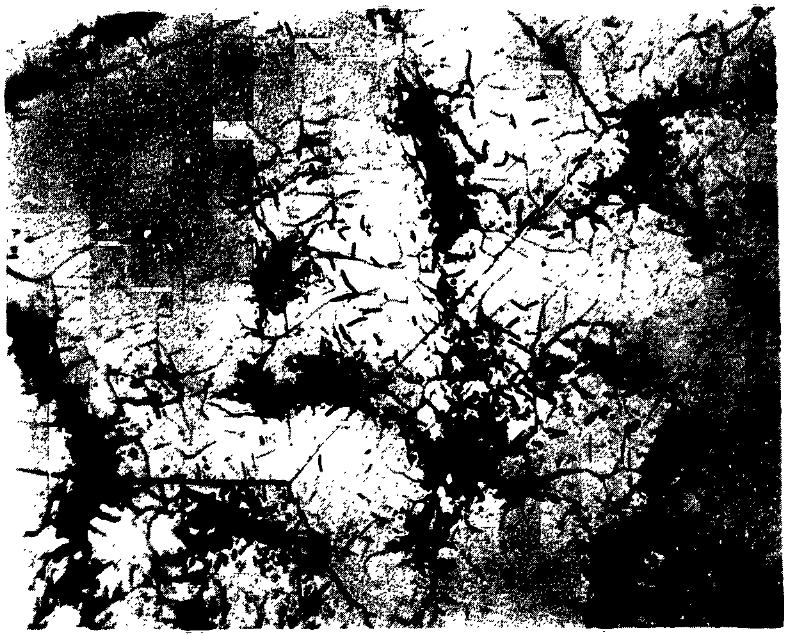


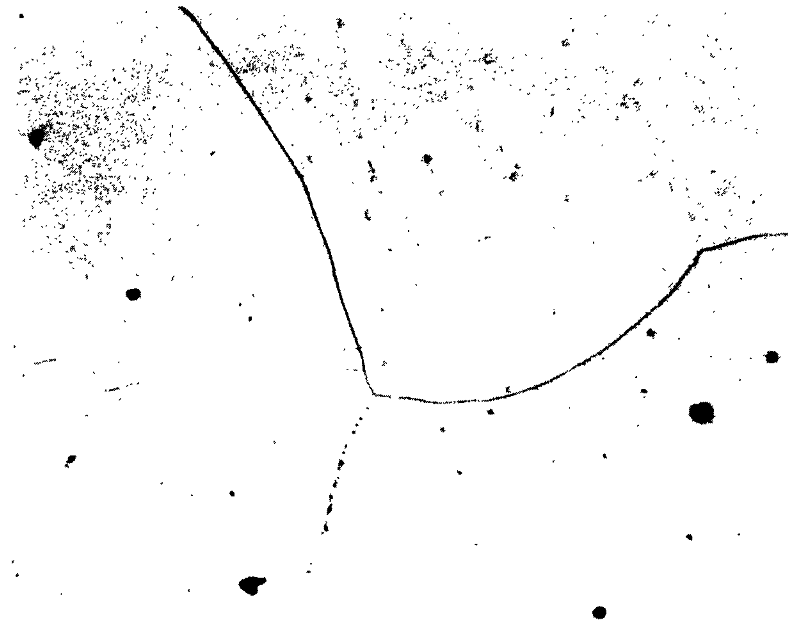
FIGURE 6

As-cast micro-
structure.

D-43 alloy,
ingot bottom.

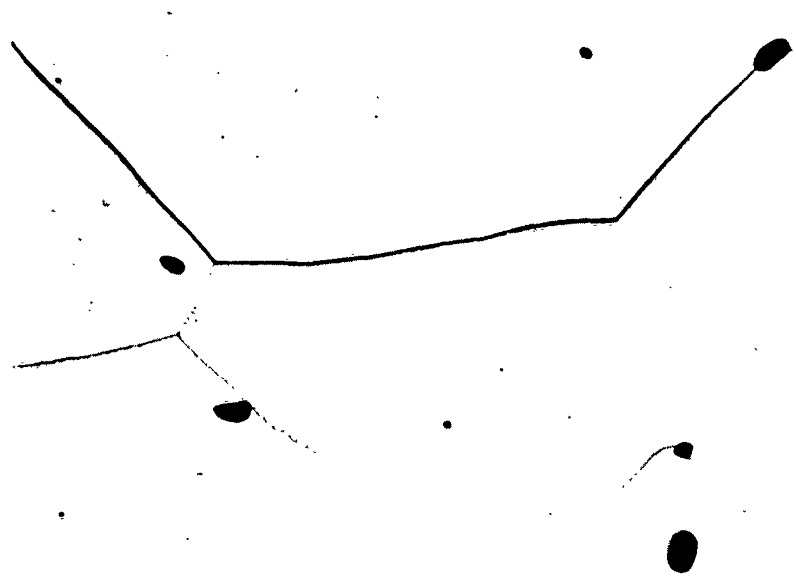
X250

Hardness R_A 54



As-cast micro-structure. B-66 alloy, ingot top.
X250 Hardness R_A 60

FIGURE 7



As-cast micro-structure. B-66 alloy, ingot bottom.
X250 Hardness R_A 59

As-cast micro-structure. Cb-752 alloy, ingot top.
X250 Hardness R_A 55

FIGURE 8

As-cast micro-structure. Cb-752 alloy, ingot bottom.
X250 Hardness R_A 56

As-cast micro-
structure.

Ta-10%W alloy,
ingot top.

X-100

Hardness R_B 89

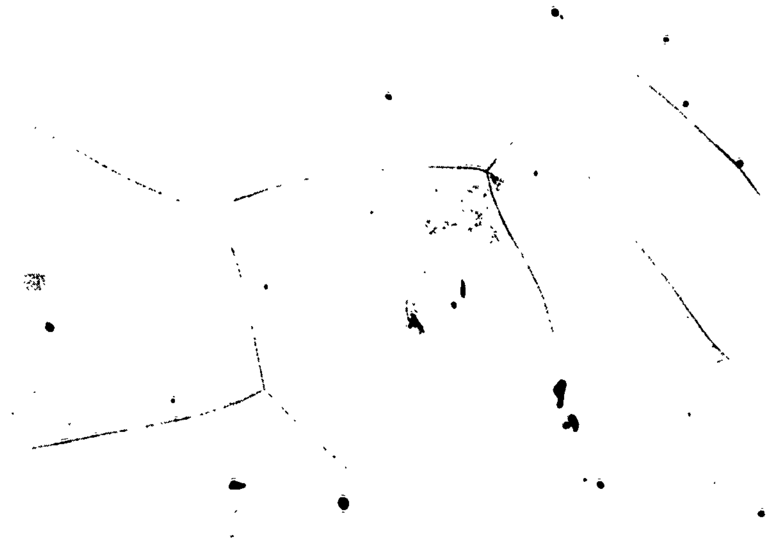
FIGURE 9

As-cast micro-
structure.

Ta-10%W alloy,
ingot bottom.

X-100

Hardness R_B 93



As-cast micro-
structure.

T-111 alloy,
ingot top.

X100

Hardness R_B 98

FIGURE 10

As-cast micro-
structure.

T-111 alloy,
ingot bottom.

X-100

Hardness R_B 96

Chemical analyses at mid-radius locations are given in Table 5. Also included in Table 5 are the calculated interstitial contents of the compacted raw materials and compositional specifications, where available.

Comparison of the calculated interstitial levels of the B-66 and Cb-752 alloy ingots with the ingot analyses indicates that a small amount of oxygen contamination occurred in melting. The source of this contamination has not been established.

The analytical data shown in Table 5 are considered to indicate satisfactory compositions for the five ingots except for the two following values which are of marginal adequacy:

1. Tungsten content at the top of the D-43 alloy ingot is low compared with the nominal 10% value. (Low tungsten values have been observed previously in the top of D-43 ingots. Generally, the low tungsten content is confined to the hot-topped portion of the ingot).
2. Oxygen content of the Cb-752 alloy ingot is marginal in view of published data* on interstitial content requirements for this alloy (although considerably lower than the North American Specification quoted). Compaction to 1-7/8"-2" diameter first melt electrodes (rather than 3" diameter) may have resulted in higher purity. (Gas removal would have been aided by the lower electrode/crucible ratio).

*Schussler, M. & Bewley, J. G., "Development of Processing Methods for Columbium Alloy Sheets". Contract No. AF33(657)-7210, Interim Report No. 1. July 1, 1962. Haynes Stellite Company.

TABLE 5

ANALYTICAL DATA ON Cb- AND Ta-BASE INGOTS

Alloy	%W	%Zr	%V	%Mo	%Hf	Interstitials, ppm O	N	C	H
D-43, top	8.7	1.1	-	-	-	35	41	865	2
bottom	9.8	1.1	-	-	-	68	48	970	3
Specification(1)	9-11	0.75-1.25	-	-	-	400 max.	100 max.	800-1200	20 max.
Calculated						76	48	1100	
B-66, top	-	0.97	5.4	4.9	-	129	37	56	3
bottom	-	0.87	5.0	4.9	-	135	39	59	2
Specification(2)	-	0.85-1.3	4.5-5.5	4.5-5.5	-	300 max.	200 max.	200 max.	-
Calculated						82	27	50	79
Cb-752, top	10.0	2.5	-	-	-	120	21	28	3
bottom	9.7	2.5	-	-	-	133	18	24	1
Specification(3)	9-11	2-3	-	-	-	400 max.	100 max.	100 max.	20 max.
Calculated						72	25	33	62
Ta-10%W, top	9.7	-	-	-	-	13	18	24	1
bottom	10.6	-	-	-	-	87	31	11	1
Specification(4)	9-11	-	-	-	-	100 max.	50 max.	50 max.	10 max.
Calculated						257	54	41	17
T-111, top	7.9	-	-	-	1.9	58	38	25	3
bottom	8.4	-	-	-	1.9	48	30	13	3
Calculated						265	51	42	17

(1) Tentative Du Pont Specification

(2) Westinghouse Special Technical Data Sheet 52-364

(3) North American Aviation Inc. Material Specification LB0170-176

(4) Tentative National Research Corp. Specification

Semi-quantitative spectrographic analyses of the main alloying elements were obtained at six points across the diameter of each slice. The results are tabulated in Table 6.

TABLE 6

SEMI-QUANTITATIVE SPECTROGRAPHIC ANALYSES ON Cb- AND Ta-BASE INGOT SLICES

Alloy	Alloy Element	Alloy Content % Edge 1	2	Center 3	4	5	Edge 6
D-43, top	W	8.8	8.5	8.8	8.6	8.2	8.4
	Zr	1.0	1.1	1.1	1.0	1.1	1.1
bottom	W	10.3	8.7	8.4	9.0	9.2	9.0
	Zr	0.97	0.98	0.98	0.97	0.93	0.96
B-66, top	Mo	4.5	4.2	4.2	4.4	4.2	4.2
	V	4.7	5.2	5.8	5.0	5.4	5.6
	Zr	0.89	0.95	1.02	0.91	0.98	-
bottom	Mo	4.5	4.2	4.2	4.3	4.3	4.3
	V	4.5	4.9	4.9	4.9	4.9	4.7
	Zr	0.91	0.94	0.95	0.93	1.00	0.95
Cb-752, top	W	10.5	10.0	9.6	9.5	10.0	9.8
	Zr	2.1	2.3	2.5	2.4	2.3	2.2
Bottom	W	10.3	10.1	10.2	10.0	10.1	10.1
	Zr	2.3	2.4	2.3	2.4	2.3	2.4
Ta-10%W, top	W	9.1	9.5	9.0	8.5	9.1	9.7
bottom	W	8.9	9.1	9.3	9.2	9.4	8.1
T-111, top	W	7.1	7.1	7.3	8.1	8.1	6.8
	Hf	1.8	1.6	2.2	2.3	1.9	2.0
bottom	W	7.8	7.2	7.1	7.4	7.4	7.4
	Hf	2.2	1.9	1.9	2.0	2.0	2.2

Since spectrographic analyses have not been standardized for absolute values of the alloying elements in these alloys, Table 6 can only be used to illustrate the variation in alloy element analyses from point to point. Locations 2 and 5 were closest to the mid-radius.

Since the ultimate product from these ingots will be foil, it was considered desirable to obtain information on the degree and dimensions of coring in the cast structure. Pronounced coring could result in significant composition gradients across relatively large linear dimensions in the foil. Microprobe analyses have been carried out on the as-cast ingot slices to obtain information on heterogeneity of composition on the micro-scale.

Small portions of the ingot slices were subjected to a one hour high temperature homogenization cycle (at 3000°F. for the columbium alloys and 3200°F. for the tantalum alloys)* Microproble analyses were then carried out on these and as-cast slices.

The microprobe scans were carried out on an electron microprobe made by Applied Research Labs, Inc. and located at the Du Pont Experimental Station in Wilmington. Semi-quantitative continuous X-ray determinations over distances of 0.3-1.0 mm were made for each alloying element. These line scans show the typical distances over which composition gradients occur and also the departure from the average composition along a line scan. The average composition is assumed to be close to the value shown in Table 5. The inaccuracy involved in this assumption can be judged by referring to the spread in analyses indicated by the spectrographic analyses in Table 6. The electron beam in the A.R.L. microprobe is less than 2 microns diameter and the X-ray beam is emitted from a slightly smaller area. Successive scans

*Temperatures of the order of 3500°-4000°F. were not tried because of the possibility of atmospheric contamination in the argon atmosphere billet heater (which would have been used to homogenize the ingots). The billet heater had not been used for prolonged heating in the 3500°-4000°F. range and the purity of the argon atmosphere could not be guaranteed.

were made for each element. At least two complete scans were made in both the as-cast and homogenized condition on mid-radius specimens from the top and bottom of each ingot. The results of the microprobe work on each ingot are summarized below. The correlation of these results with microstructure will be described in the Phase III report. It must be pointed out that the effect of high temperature heat treatment on hetergeneity could not be rigorously evaluated since different specimens were examined for the as-cast and homogenized slices (the two specimens were taken from locations less than 1/2" apart in the original ingot). The variations in composition reported are the extreme values.

D-43 Alloy

In the as-cast condition tungsten varied by ± 20% (i.e., from 8%W to 12%W, assuming nominal composition). Zirconium varied ± 30%. After homogenization tungsten variation was similar. Zirconium content varied ± 22%. These variations occurred across distances of 70-200 microns.

Zirconium and tungsten varied inversely to each other, i.e., low tungsten areas showed high zirconium and vice versa.

Cb-752 Alloy

Tungsten varied ± 32% in the as-cast specimen and ± 30% in the homogenized specimen. Zirconium varied ± 22% as-cast and ± 50% in the homogenized specimen. A cored substructure was detected within the as-cast grains of this alloy which could be related to the results of the microprobe scan. Tungsten content is low and zirconium high at the boundaries of this sub-structure while the reverse occurs inside the grains. Segregation occurred over distances of 100-200 microns.

B-66 Alloy

Molybdenum varied ± 9% as-cast and ± 8% on the homogenized specimen. Zirconium varied ± 25% in both conditions. Vanadium varied ± 15% as-cast and ± 12% in the homogenized specimen. Segregation distances were 50-150 microns. Molybdenum segregated inversely to zirconium and vanadium.

Ta-10%W Alloy

Tungsten distribution was extremely uniform in this alloy. Variation was ± 4% on the as-cast specimen and ± 6% on the homogenized specimen.

T-111 Alloy

Tungsten varied ± 4% as-cast and ± 4-1/2% in the homogenized specimen. Hafnium varied ± 16% in both samples. Segregation distances were typically 70-100 microns.

No appreciable difference was observed in the pattern of segregation before and after the high temperature homogenization treatment of any of the alloys. Some degree of homogenization may have occurred but it could not be determined from the relatively few data obtained in this work.

Radial hardness traverses were carried out on the ingot slices before and after the high temperature homogenization treatment. The results are shown in Table 7.

TABLE 7

HARDNESS MEASUREMENTS OF Cb- AND Ta-BASE ALLOY INGOT SLICES

	Edge				Center	
D-43 Alloy						
Top, As-Cast	54	54	54	54	53	(R_A)
Homogenized	50	51	50	52	51	
Bottom, As-Cast	54	54	54	54	53	
Homogenized	51	54	54	52	51	
B-66 Alloy						
Top, As-Cast	60	60	60	59	58	
Homogenized	60	60	60	61	59	
Bottom, As-Cast	60	59	59	59	58	
Homogenized	58	59	60	59	60	
Cb-752 Alloy						
Top, As-Cast	53	54	55	54	53	
Homogenized	54	54	54	51	52	
Bottom, As-Cast	55	57	56	56	56	
Homogenized	55	56	55	55	54	
Ta-10%W Alloy						
Top, As-Cast	89	90	89	88	88	(R_B)
Homogenized	91	90	90	89	89	
Bottom, As-Cast	93	94	93	93	91	
Homogenized	93	94	93	93	94	
T-111 Alloy						
Top, As-Cast	89	97	98	97	95	
Homogenized	92	93	94	94	94	
Bottom, As-Cast	85	96	96	93	96	
Homogenized	93	94	94	94	94	

The three columbium base alloys and the Ta-10%W alloy show no significant change in hardness as a result of the high temperature homogenization. No changes in microstructure could be detected in these alloys either. The hardnesses do not show any pronounced variation from edge to center.

The T-111 alloy shows an appreciably lower hardness at the edge than center in the as-cast condition. Since the difference no longer exists after the high temperature heat treatment, it is believed to be associated with a cooling rate effect rather than a difference in composition. The microstructure (at the mid-radius location) of this alloy contained slightly more of an unidentified second phase (visible in Figure 10) after homogenization than in the as-cast condition.

APPENDIX

PHASE III WORK SCHEDULE

Phase III extends over a period of three months and consists of production and testing of coil blanks in each of the columbium and tantalum base alloys. These coil blanks are for subsequent rolling to 12" wide foil.

All ingots will be extruded to 1-1/2" x 4" sheet bar. The three columbium alloy ingots will be canned in mild steel and extruded through flat dies. The tantalum alloy ingots will be extruded bare through cone dies.

The sheet bars will be warm rolled to 1/4" - 3/8" thickness. In order to obtain plate of adequate flatness for belt conditioning, the warm rolled plates will first be roller leveled. After conditioning and heat treatment the plates will be rolled to 0.100" thickness and evaluated.

Small scale rolling trials on tungsten sheet will be carried out during Phase III in order to select the most suitable starting material for rolling to foil at the 12" width. Information obtained in this manner will also be used as a basis for design of continuous strip heating equipment for tungsten rolling.

E. I. du Pont de Nemours & Co., Inc.
Metal Products
Pigments Department
Wilmington, Delaware
REFRACTORY ALLOY FOIL ROLLING DEVELOPMENT PROGRAM
January, 1963

ASD Project No. 7-987
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(over)

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2. Processing and properties of Columbium, Tantalum and Tungsten Alloys
3. Testing and Inspection Procedures

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