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THE PREPARATION AND EVALUATION OF HIGH-PURITY, FINE-GRAINED TUNGSTEN AND TUNGSTEN ALLOY CASTINGS

by

S. G. Nelson and E. L. Foster, Jr.

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY REPORT

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THE PREPARATION AND EVALUATION OF HIGH-PURITY, FINE-GRAINED TUNGSTEN AND TUNGSTEN ALLOY CASTINGS

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ABSTRACT

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A technique was developed for the preparation of high-purity, cylindrical castings of high-melting-point materials. Relatively fine-grained, 3/4-inch-diameter, tungsten and tungsten-tantalum, -rhenium, -iridium and -osmium alloy castings were prepared by this technique. Most castings contained less than 50 ppm total impurities as determined by mass-spectrographic, gas, and conductometric analyses.

Author

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Battelle Memorial Institute

SUMMARY

The principal program objective was to fabricate, analyze, evaluate, and deliver to NASA ultrahigh-purity, fine-grained, cast specimens of tungsten, tungsten+5, +10, and +20 weight percent tantalum, tungsten+5, +15, and +23 weight percent rhenium, tungsten+2.5 and +5 weight percent osmium, and tungsten+1 and +2 weight percent iridium. Secondary objectives were to investigate the homogeneity of button castings of seven of the above compositions and to deliver to NASA small samples of the button castings. In general, the objectives of the program were achieved. A new technique was developed for the preparation of ultrahigh-purity, 3/4-inch-diameter, cylindrical castings of high-melting-point materials. This technique was utilized in the preparation of all the larger tungsten and tungsten alloy specimens.

INTRODUCTION

Tungsten and certain tungsten alloys display a number of properties which make them attractive as potential materials for ion engine applications. The use of tungsten or tungsten alloys as porous emitter plates in a cesium-fueled ion engine, for example, will permit operation of the engine at temperatures up to 2400 F for long periods of time. One particular property important in the long-term efficiency of ion engines is the work function of the emitter material. Tungsten and certain tungsten alloys when free from impurities are believed to possess a work function of 4.5 ev or greater, a very desirable property for use in ion engines.

This report describes a program that was undertaken at Battelle for NASA in which high-purity, fine-grained tungsten and tungsten alloys containing 5-, 10-, and 20-weight percent tantalum, 5-, 15-, and 23-weight percent rhenium, 2.5-, and 5-weight percent osmium, and 1-, and 2-weight percent iridium were prepared. Materials were first prepared as button castings in nonconsumable-electrode arc furnaces and then as drop castings in an electron-beam furnace. All materials were analyzed and evaluated and specimens taken from button castings and from drop castings were delivered to NASA for further study.

Battelle personnel who made valuable contributions to the program include W. J. Hildebrand, R. R. Walker and F. P. Holcomb, Jr. (in technique development and materials preparation); E. R. Blosser and W. M. Henry (in chemical analyses); and J. L. McCall and G. R. Strabel (in microprobe analyses).

STARTING MATERIALS

The elemental starting materials (tungsten, tantalum, rhenium, osmium, and iridium) were purchased from commercial vendors. Although a complete analysis of all purchased materials was requested, in most cases only an overall purity and a token analysis were supplied. All starting materials, however, were check-analyzed mass spectrographically at Battelle (see Table 1). The particular piece of equipment that was utilized for all mass-spectrographic analyses in the program was a high-resolution (MS-7) mass spectrograph. This English-made spectrograph is a relatively new instrument and has been available commercially only in the past few years.

Table 2 lists for each starting material, its form, its supplier, the supplier's stated purity, and the actual purity as found by mass-spectrographic analyses on each starting material. The analyses of materials received from the Allied Chemical Corporation (tungsten) and from the National Research Corporation (tantalum) agreed well with the supplier's indicated impurity levels. Materials obtained from the United Mineral and Chemical Company (osmium, iridium, and rhenium), on the other hand, did not. These latter materials were considerably less pure than was advertised or stated. Even though the impurity levels of these materials were high, however, these materials were considered usable since they would be employed as minor alloying additions and would not increase the impurity above those maximum values specified by NASA for the final alloys.

MELTING AND CASTING PROCEDURES

Preparation of Small Button Castings

Fifty-gram charges of pure starting materials were weighed out on an analytical balance and were melted and alloyed by arc melting under helium (1/3-atmosphere total pressure) in a water-cooled copper crucible utilizing a nonconsumable tungsten-tipped electrode. Each prepared button casting was turned over and was remelted 6 times to insure a high degree of homogenization. A direct current of 500 amperes at 30 volts was employed during melting. In melting, the hearth and charge were made the positive pole and the electrode acted as the negative pole. Figure 1 shows the arc-melting furnaces that were used.

No major problems were encountered in arc melting, although some spattering did occur in the preparation of tungsten-rhenium alloy buttons. Apparently the rhenium starting material contained a small amount of volatile impurity which was released during melting. Only very slight attack of the tungsten tips and little or no copper attack was observed in the preparation of the tungsten alloy button castings.

Electron-Beam Melting and Drop Casting

A new technique was developed for the preparation of cylindrical drop castings of ultrahigh-purity tungsten alloys. In this technique the features of drop casting as is

TABLE 1. MASS-SPECTROGRAPHIC ANALYSIS OF STARTING MATERIALS[a]

(PPM, Weight Basis)

Element	Sample W	Ir	Re	Os	Ta
Li	(a)	0.3	1.5	0.05	<0.02
Be	<0.02	<0.06	<0.02	≤0.01	≤0.03
B	<0.02	0.4	1	0.3	0.07
F	80.	0.08	<0.04	0.08	<0.01
Na	0.5	25.	8.	8.	<0.04
Mg	1.	25.	<5.	<5.	0.04
Al	0.3	3.	10.	1.2	0.3
Si	1.	5.	60.	3.	5.
P	0.3	0.7	1.5	0.15	≤1.
S	10.	15.	60.	10.	3.
Cl	8.	400.	7.	500.	0.1
K	0.4	8.	100.	5.	0.07
Ca	0.2	10.	20.	10.	0.07
Sc	<0.2	≤3.	<0.1	≤0.1	≤0.1
Ti	≤0.25	6.	0.4	2.	≤2.
V	<0.01	≤120.	0.03	<0.02	0.15
Cr	<0.05	6.	6.	0.15	0.4
Mn	<0.05	0.25	1.5	0.04	0.02
Fe	0.3	12.	40.	1.8	8.
Co	<0.05	0.3	0.6	0.04	3.
Ni	<0.08	5.	40.	0.06	3.
Cu	0.2	1.5	40.	0.15	1.
Zn	0.1	1.5	8.	0.15	<0.02
Ga	<0.2	<0.08	<0.05	≤0.05	<0.01
Ge	<0.3	0.15	0.12	≤0.05	<0.01
As	0.03	1.5	0.3	0.5	<0.01
Se	<0.03	0.07	25.	≤0.04	<0.02
Br	0.5	35.	<0.04	35.	0.03
Rb	<0.04	20.	0.08	<0.03	<0.02
Sr	<0.02	<0.1	0.06	<0.03	<0.02
Y	<0.02	<0.07	<0.02	<0.02	1.5
Zr	<0.1	20.	<0.04	<0.04	5.
Nb	Int.	≤0.06	1.	Int.	150.
Mo	<0.1	<0.15	1.	<0.1	40.
Ru	<0.05	8000.	<0.3	<0.15	<0.2
Rh	<0.02	0.6	<0.2	<0.1	<0.1
Pd	<0.05	10.	<0.1	<0.1	<0.3
Ag	<0.04	0.15	<0.05	<0.05	<0.06
Cd	<0.05	6.	<0.1	<0.1	<0.03
In	≤1.	≤10.	≤7.	≤1.2	≤0.4
Sn	<0.05	0.3	0.4	<0.08	<0.02
Sb	<0.04	≤0.3	≤0.4	≤0.3	<0.02
Te	<0.04	0.2	<0.1	<0.1	<0.02

TABLE 1. (Continued)

Element	Sample W	Sample Ir	Sample Re	Sample Os	Sample Ta
I	<0.01	<0.06	<0.03	<0.03	<0.01
Cs	<0.01	0.3	0.1	0.06	<0.01
Ba	≤0.08	0.4	0.4	0.12	<0.01
La	<0.02	<0.04	<0.04	<0.04	<0.01
Ce	<0.02	<0.04	<0.04	<0.04	<0.01
Pr	<0.02	<0.04	<0.04	<0.04	<0.01
Nd	<0.07	<0.15	<0.15	<0.15	<0.04
Sm	<0.08	<0.15	<0.15	<0.15	<0.04
Eu	<0.04	<0.08	<0.08	<0.08	<0.02
Gd	<0.08	<0.15	<0.15	<0.15	<0.04
Tb	<0.02	<0.04	<0.04	<0.04	<0.01
Dy	<0.08	<0.15	<0.15	<0.15	<0.04
Ho	<0.02	<0.04	<0.04	<0.04	<0.01
Er	<0.06	<0.15	<0.15	<0.15	<0.04
Tm	<0.02	<0.04	<0.04	<0.04	<0.01
Yb	<0.06	<0.15	<0.15	<0.15	<0.04
Lu	<0.02	<0.04	<0.04	<0.04	<0.01
Hf	<0.07	<0.15	<0.15	<0.15	0.2
Ta	<0.2	≤4.	≤8.	≤4.	--
W	--	≤0.3	7.	≤0.2	100.
Re	20.	<0.08	--	≤2.	<0.2
Os	<0.1	<0.6	<0.3	--	<0.3
Ir	<0.05	--	≤0.6	≤6.	<0.3
Pt	≤3.	≤0.6	≤1.5	≤0.5	5.
Au	≤1.	≤0.15	≤1.5	≤0.12	≤5.
Hg	≤5.	≤0.6	≤1.5	≤3.	<0.4
Tl	<0.2	≤7.	≤0.6	≤1.5	<0.2
Pb	≤1.	≤2.	25.	2.5	≤1.
Bi	≤0.3	≤0.5	≤0.1	≤0.2	≤0.2
Th	≤0.8	<0.05	<0.05	<0.05	<0.2
U	≤0.15	<0.05	<0.05	<0.05	0.2

(a) -- – Off end of plate;
Int. – interference;
≤ – presence of element in sample not certain;
< – not detected.

TABLE 2. STARTING MATERIALS

Element:	W	Ir	Re	Os	Ta
Form:	Coarse Powder	Fine Powder	Fine Powder	Fine Powder	EB-Melted Billet Slice
Source	Allied Chemical Corporation	United Mineral and Chemical Company	United Mineral and Chemical Company	United Mineral and Chemical Company	National Research Corporation
Purity Stated by Source	99.99	99.999	99.99	99.999	99.99
Actual Purity (Battelle Analyses)	99.99	99.1+	99.95+	99.95	99.99

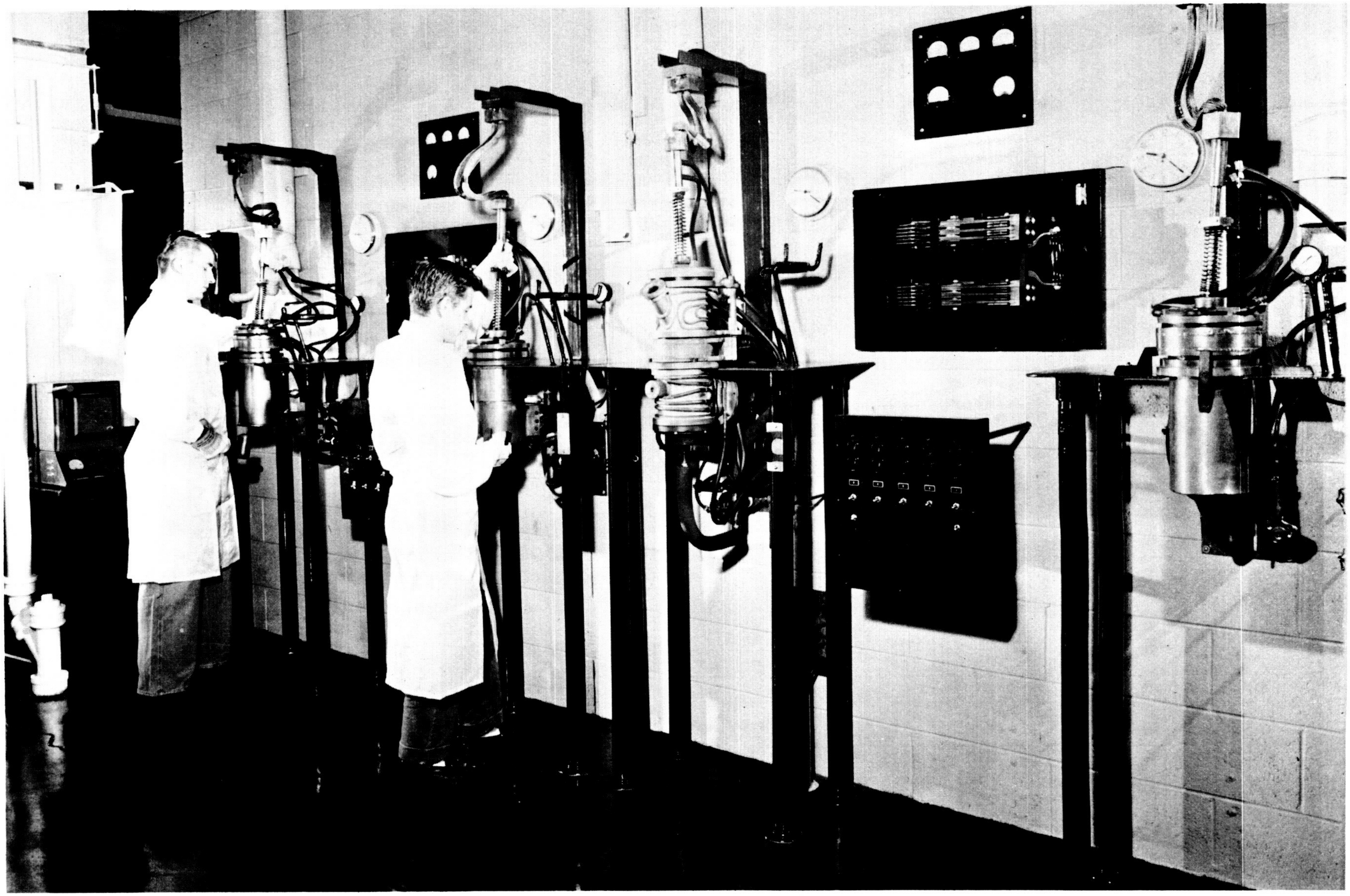

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FIGURE 1. NONCONSUMABLE-ARC FURNACES

commonly performed in the arc-melting furnace were combined with the features of melting in an electron-beam furnace.

In drop casting in an arc furnace, a button or slug of material is placed on a water-cooled copper hearth over a sprue which leads to a mold cavity below. Under an inert-gas atmosphere, an arc is struck between an electrode and the charge, and arcing is continued until the button or slug is molten and flows through the sprue and into the mold. This drop-casting principle was utilized in the electron-beam furnace.

Melting in an electron-beam furnace offers several advantages over other melting methods with respect to the melting of tungsten alloys. Melting in the electron-beam furnace is performed in vacuum which reduces the possibility of contamination and actually permits purification during melting. Low-melting-point impurities tend to "boil" off.

To melt and drop cast 200-gram quantities of tungsten and tungsten alloys in the laboratory properly, large amounts of energy are required and this energy must be controllable. Because much higher power levels are available and usable in the electron-beam furnace than are normally found in conventional laboratory arc-melting equipment today, the electron-beam furnace would appear most appropriate for such melting. Also, the use of the electron-beam furnace allows flexibility in mold design, an important consideration in the preparation of castings of materials with high thermal conductivities. Mold design plays an important part in that metal must be allowed to remain molten long enough to fill completely the mold cavity and thus to prevent the occurrence of cold shuts and other casting defects. The metal, on the other hand, must be permitted to freeze rapidly enough to prevent excessive grain growth, since the goal of the program is fine-grained material.

After considerable experimentation, the apparatus shown in Figure 2 and schematically in Figure 3 was designed and was constructed. This apparatus consisted principally of (1) a tungsten crucible or holder lined with the tungsten alloy to be drop cast; (2) tungsten supports; and (3) a water-cooled copper mold. Preparation of the tungsten crucible was performed in several steps as shown in Figure 4. Initially, tungsten billet slices, 3/4-inches thick and 3-1/8 inches in diameter, of 99.9+ purity were purchased from Sylvania Electric Products Inc. (A). A tapered hole 2-1/4 inches in diameter at the top and 1-1/4 inch in diameter at the bottom was drilled into each billet slice (B). Approximately seven small button castings of the desired alloy were prepared by arc melting and alloying ultrapure starting materials and these buttons were arranged on the inside of the tapered hole (C). The crucible containing the buttons was then placed inside an arc furnace and the buttons were remelted and were spread out over the entire tapered face of the crucible covering completely the hole in the crucible (D). The thickness of the alloy linings prepared in this manner was about 1/4 inch. An additional five or more button castings of the desired alloy were then placed on the lining over the hole in preparation for drop casting (E) A typical tungsten crucible and a liner of tungsten-5 weight percent osmium are shown in Figure 5.

The water-cooled copper mold was designed so that cooling would be rapid enough to eliminate any possibility of copper attack and to obtain material of relatively fine-grain size, but would be slow enough to permit metal to flow into the mold before freezing. These objectives were achieved in the mold design shown in Figure 3.

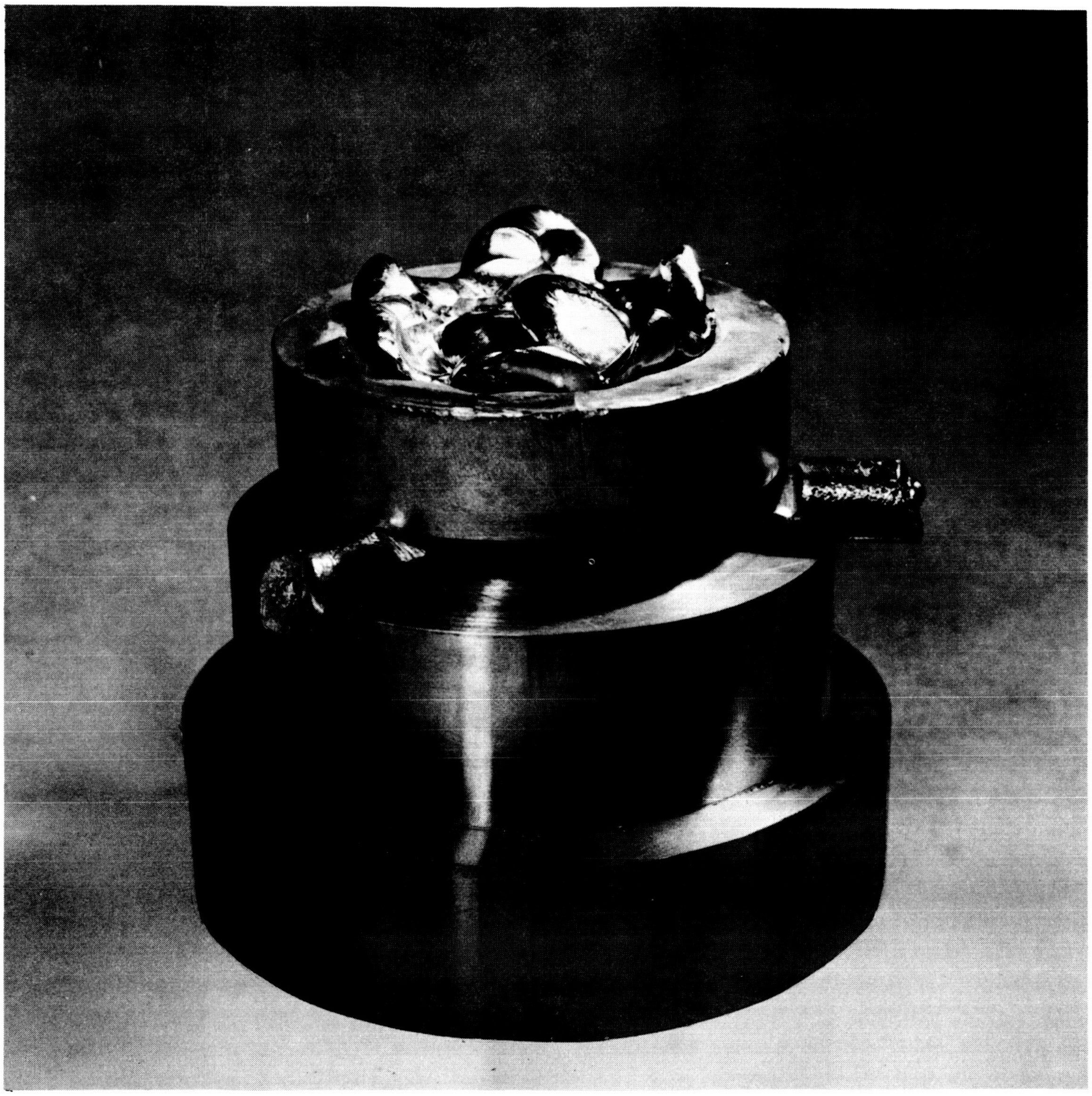

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FIGURE 2. APPARATUS FOR DROP CASTING IN THE ELECTRON-BEAM FURNACE

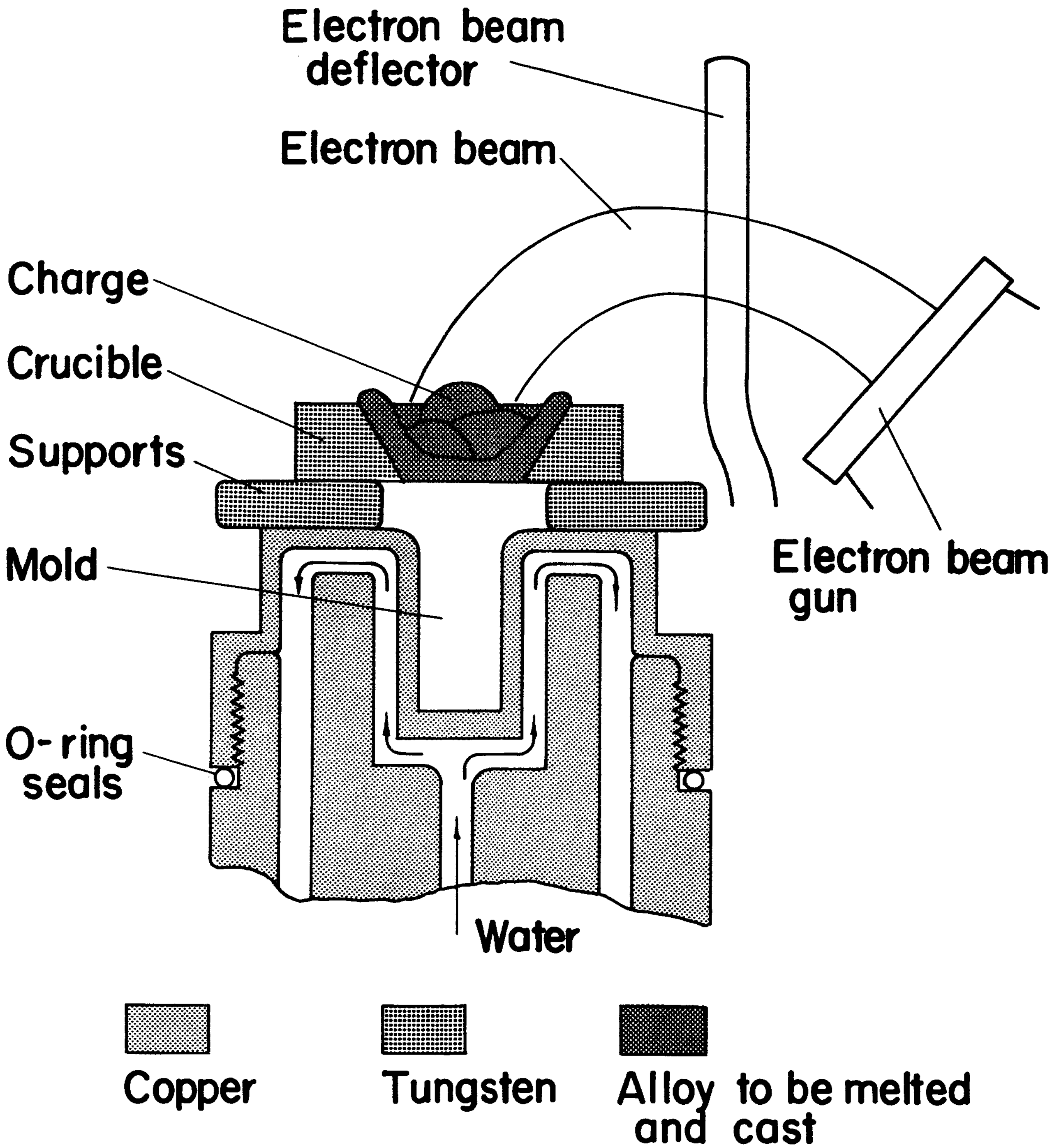

FIGURE 3. SCHEMATIC DRAWING OF DROP-CASTING APPARATUS IN ELECTRON-BEAM FURNACE

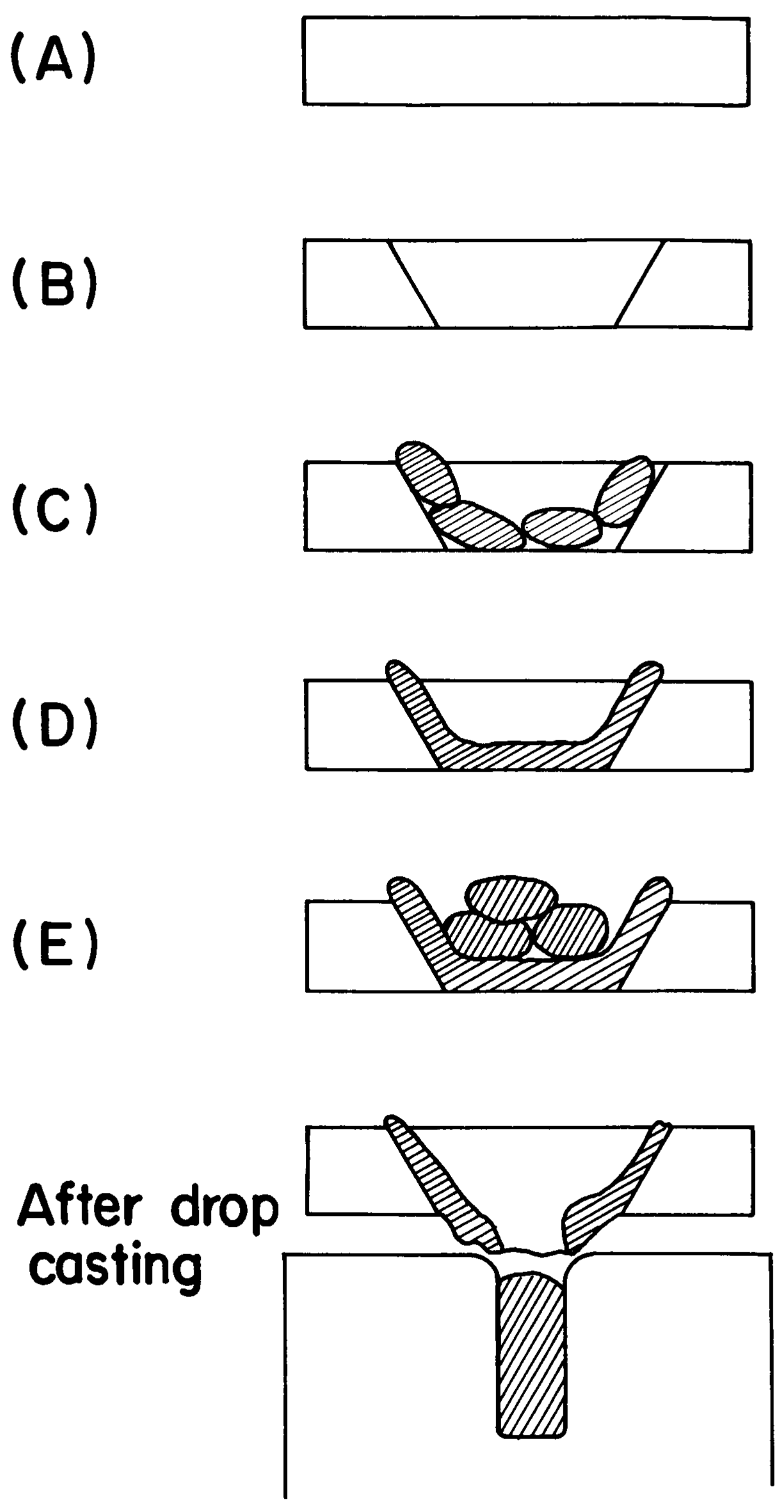

FIGURE 4. STEPS IN CRUCIBLE PREPARATION

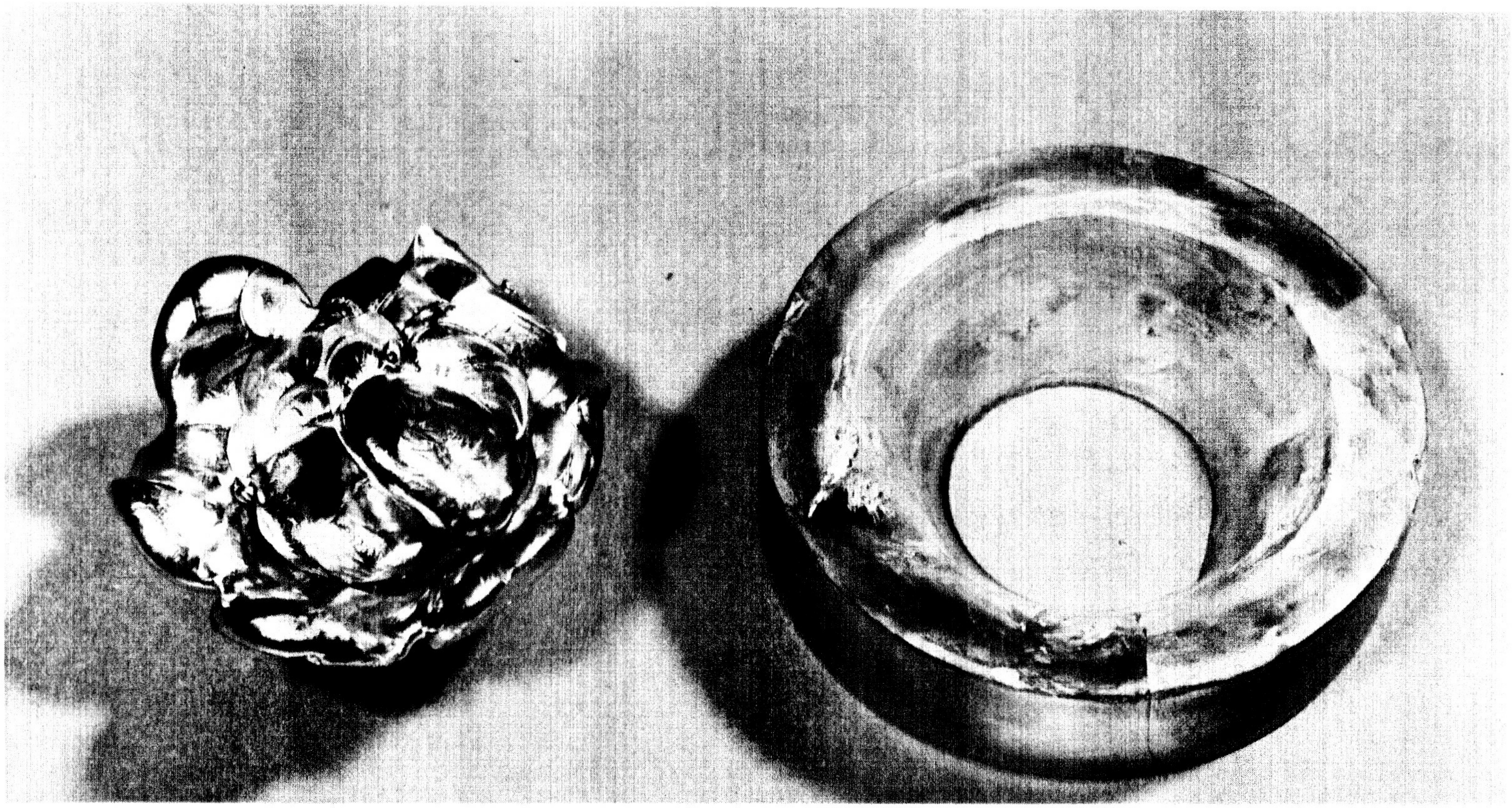

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FIGURE 5. TUNGSTEN-5 WEIGHT PERCENT OSMIUM CRUCIBLE LINER (LEFT) AND TUNGSTEN CRUCIBLE (RIGHT)

A transverse electron-beam gun was employed in melting. The beam was deflected and focused as shown in Figure 3. Before drop casting, the gun was operated at approximately 20 kw to outgas the apparatus' components and to achieve a satisfactory vacuum. A vacuum of 4×10^{-4} mm Hg or better was obtained in every run. Drop casting of the tungsten alloys was not achieved until the energy in the beam was increased to between 70 and 85 kw. In most cases, the alloys dropped into the molds satisfactorily. Figure 6 shows the electron-beam furnace during a regular run. In several early runs, the copper molds were lined with tungsten sheet to prevent copper pickup. This practice was abandoned, however, when it was found that little or no copper pickup occurred when the tungsten sheet was excluded. Four typical drop castings are shown in Figure 7. Specimen A in this figure was cast into a tungsten-lined mold.

EVALUATION OF PREPARED ALLOYS

Chemical Analyses

Complete mass-spectrographic, gas, and conductometric (for carbon) analyses were conducted on sections of button castings prepared by arc melting and on drop castings prepared in the electron-beam furnace. The results of these analyses are given in Tables 3 and 4.

Purity specifications established by NASA are given in Table 5. A comparison of the maximum allowable elemental levels with those levels observed for the button and drop castings showed that the purities of the alloys prepared were far better than were requested. The only impurity occurring in any noteworthy amount was ruthenium which appeared in the two tungsten-iridium alloys. The ruthenium was present in the iridium starting material. The levels of ruthenium in drop castings of the tungsten-1 and -2 weight percent iridium alloys were 40 and 90 ppm, respectively.

Metallographic Examination

Sections of button castings and of drop castings of each composition were mounted and examined metallographically, and representative photomicrographs of each material were taken. Figure 8 shows typical microstructures of drop castings of each material. Regions near the center of the drop castings and approximately 1/4 inch below and parallel to their top surfaces are shown in these photomicrographs. Three different etchants, 1 to 3 volume percent hydrogen peroxide in boiling water (dip), lactic acid-nitric acid-hydrofluoric acid (3:1:1 by volume-swab), and Murakami's reagent (dip), were used on the alloys. No one etchant was found suitable for all the alloys.

As might be expected from consideration of the equilibrium phase diagrams of the tungsten-metal systems, pure tungsten and the tungsten-tantalum, -rhenium, -osmium and -iridium alloys all appeared to be single phase. The drop castings of

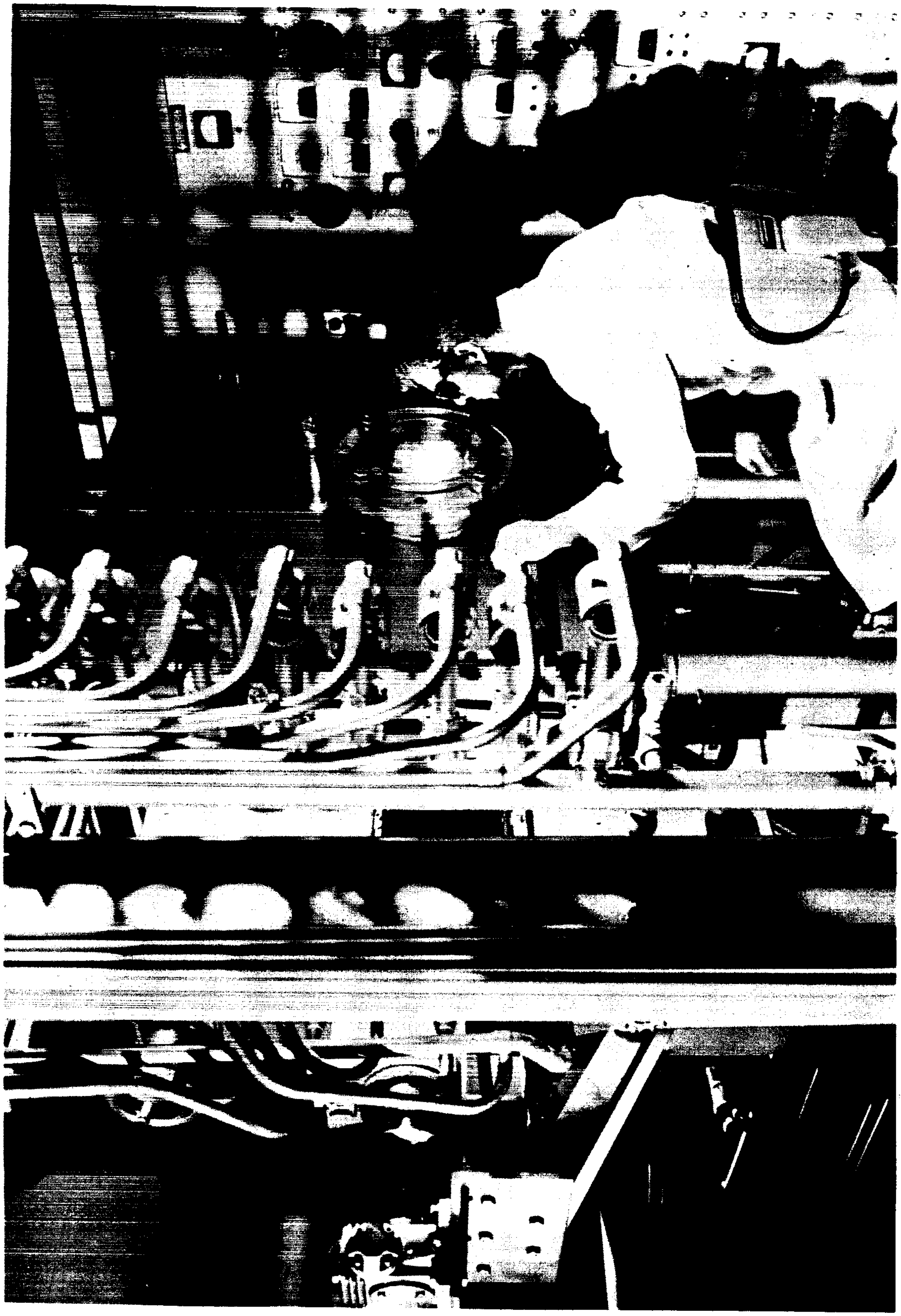

FIGURE 6. ELECTRON-BEAM FURNACE

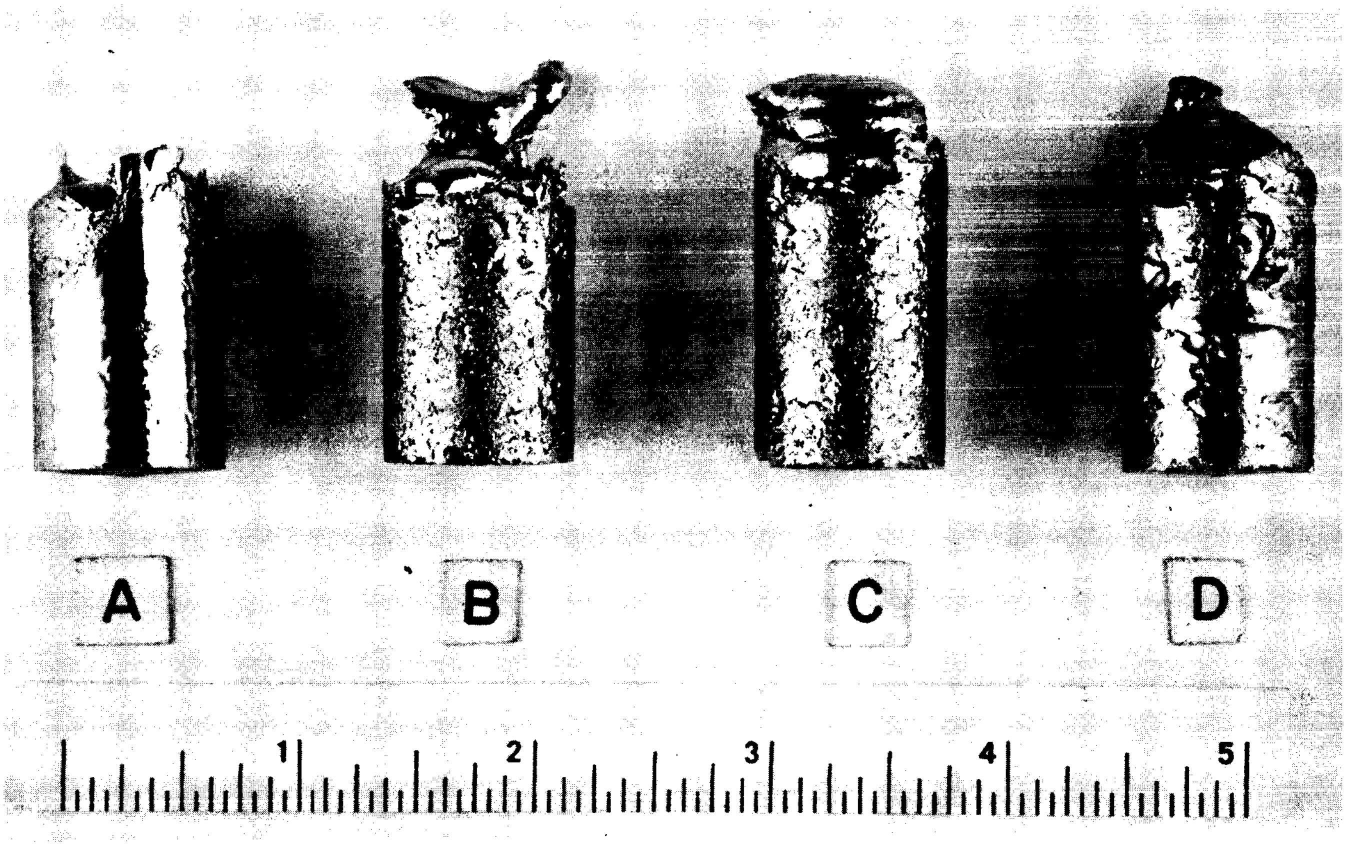

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FIGURE 7. DROP CASTINGS OF TUNGSTEN (A), (B), AND OF TUNGSTEN – 20 WEIGHT PERCENT TANTALUM (D)

TABLE 3. RESULTS OF CHEMICAL ANALYSES OF BUTTON CASTINGS

(PPM, Weight Basis)[a]

Element	Specimen										
	Pure W	W-5Re	W-15Re	W-23Re	W-2.5Os	W-5Os	W-5Ta	W-10Ta	W-20Ta	W-1Ir	W-2Ir
Li	<0.004	0.012	0.004	<0.004	<0.004	0.04	<0.004	<0.004	≤0.08	<0.005	≤0.2
Be	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.005	<0.002	<0.02	<0.006	≤0.006
B	0.001	0.004	<0.001	<0.001	<0.006	<0.002	0.3	0.03	0.03	0.003	0.07
F	0.1	<0.01	<0.01	<0.01	0.05	0.5	0.02	<0.01	<0.01	<0.01	0.02
Na	<0.1	<0.1	<0.1	<0.1	0.25	<0.1	<0.03	<0.15	<0.15	<0.15	0.1
Mg	<0.1	0.15	0.15	0.15	0.1	≤0.1	0.1	0.04	0.05	≤0.06	≤0.05
Al	0.02	3.[b]	0.05	0.05	0.4	0.2	0.01	0.05	0.03	0.2	0.04
Si	0.2	3.[b]	1.	<0.2	15.	3.	<0.05	1.	0.5	0.6	5.
P	0.1	<0.04	<0.04	<0.2	<0.04	≤0.5	<0.02	0.01	0.08	0.1	0.02
S	1.	3.	3.	1.5	1.5	1.	≤1.	0.3	1.2	0.4	1.
Cl	0.4	0.3	0.8	0.15	0.3	0.15	0.4	0.1	0.08	0.1	0.2
K	0.6	0.2	0.1	0.05	0.2	0.2	<0.04	≤0.2	0.2	0.2-1.*	0.2
Ca	0.02	0.1	0.05	0.03	0.01	0.05	0.02	0.04	0.06	0.1	0.2
Sc	<0.1	<0.05	<0.05	<0.05	<0.25	≤0.05	<0.01	<0.02	<0.02	≤0.02	<0.02
Ti	<0.04	0.1	0.1	0.1	≤0.5[c]	≤0.7[c]	<0.2	≤0.15	≤0.5	0.2	0.2
V	<0.002	<0.02	<0.002	<0.002	<0.002	0.002	<0.002	0.01	<0.003	≤0.5	≤0.5
Cr	0.1	0.08	0.01	0.12	0.01	0.4	0.04	0.2	0.1	0.6	0.15
Mn	<0.02	0.03	0.02	0.06	0.03	0.03	0.3	0.03	0.02	0.1	0.03
Fe	1.5	40.	25.	12.	30.	12.	0.7	0.3	0.7	1.-10.*	1.
Co	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002	<0.005	<0.01	<0.01	<0.003	0.02
Ni	≤0.2[d]	≤0.15	≤0.15	≤0.3	≤0.1	0.4	<0.05	0.2	0.1	0.15-1.5*	0.15
Cu	<0.03	1.5	0.3	0.15	<0.01	≤0.03	0.7	0.06	0.4	0.2	0.4
Zn	≤0.07[d]	0.012	<0.01	<0.03	<0.03	<0.06	<0.07	0.05	0.1	0.07	0.07
Ga	<0.1	<0.06	<0.005	<0.02	<0.02	<0.08	<0.04	0.02	<0.006	≤0.006	≤0.006
Ge	<0.2	<0.4	<0.4	<0.4	<0.4	<0.4	<0.03	<0.012	<0.012	<0.012	<0.012
As	<0.05	<0.003	<0.01	<0.008	<0.01	<0.04	<0.003	<0.02	<0.02	<0.004	<0.02
Se	<0.1	<0.006	<0.02	<0.006	<0.02	<0.02	<0.01	<0.008	<0.008	<0.008	<0.008
Br	<0.1	<0.006	<0.02	<0.006	<0.02	<0.02	<0.03	<0.007	0.1	<0.008	0.007
Rb	<0.02	<0.004	<0.08	<0.004	<0.02	<0.02	<0.02	<0.005	<0.005	<0.006	≤0.005
Sr	<0.02	<0.004	0.03	0.01	<0.02	<0.02	<0.02	<0.006	<0.006	<0.006	<0.006
Y	<0.01	0.05	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	≤0.01	<0.01
Zr	<0.3	<1.	<1.	<1.	≤100.[c]	≤100.[c]	≤1.	≤3.	≤8.	<0.3	<0.3
Nb	Int.	Int.	Int.	Int.	Int.	Int.	Int.	Int.	Int.	Int.	Int.
Mo	<0.3	1.	0.6	0.6	1.	<0.2	3.	6.	10.	<0.1	<0.1
Ru	<0.2	0.5	<0.15	<0.15	<0.15	<0.15	<0.2	0.4	<0.2	18.	50.
Rh	<0.07	<0.05	<0.05	<0.05	<0.05	<0.05	<0.07	<0.1	<0.07	<0.03	<0.03
Pd	<0.4	<0.15	<0.15	<0.15	<0.15	<0.15	<0.4	<0.4	<0.4	<0.07	<0.07
Ag	<0.15	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.2	<0.2	<0.06	<0.03
Cd	≤60.[d]	<0.3	<0.3	<0.3	<0.3	<0.3	≤20.[d]	<0.4	<0.7	≤0.3	≤0.3
In	≤0.4[d]	<0.04	<0.04	<0.015	<0.015	<0.05	≤4.[d]	<0.03	<0.1	<0.04	≤0.1

TABLE 3. (Continued)

Element	Specimen										
	Pure W	W-5Re	W-15Re	W-23Re	W-2.50s	W-50s	W-5Ta	W-10Ta	W-20Ta	W-1Ir	W-2Ir
Sn	<0.04	<0.02	<0.02	<0.05	<0.05	<0.05	<0.04	<0.03	<0.03	<0.03	<0.03
Sb	<0.04	<0.015	<0.015	<0.03	<0.03	<0.03	<0.04	<0.02	<0.02	<0.02	<0.02
Te	<0.2	<0.02	<0.02	<0.05	<0.05	<0.05	<0.2	<0.03	<0.03	<0.03	<0.03
I	<0.02	<0.008	<0.008	<0.015	<0.015	<0.015	<0.02	<0.01	<0.01	<0.01	<0.01
Cs	<0.02	<0.008	<0.008	<0.015	<0.015	<0.015	<0.02	<0.01	<0.01	<0.01	<0.01
Ba	<0.02	<0.01	<0.025	<0.025	<0.025	<0.025	<0.02	<0.01	0.03	<0.01	<0.01
La	<0.007	<0.008	<0.008	<0.008	<0.03	<0.03	<0.007	<0.01	<0.01	<0.01	<0.01
Ce	<0.007	<0.008	<0.008	<0.008	<0.03	<0.03	<0.007	<0.01	<0.01	<0.01	<0.01
Pr	<0.007	<0.008	<0.008	<0.008	<0.03	<0.03	<0.007	<0.01	<0.01	<0.01	<0.01
Nd	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1	<0.03	<0.04	<0.04	<0.04	<0.04
Sm	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1	<0.03	<0.04	<0.04	<0.04	<0.04
Eu	<0.02	<0.015	<0.015	<0.015	<0.05	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02
Gd	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1	<0.03	<0.04	<0.04	<0.04	<0.04
Tb	<0.007	<0.008	<0.008	<0.008	<0.03	<0.03	<0.007	<0.01	<0.01	<0.01	<0.01
Dy	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1	<0.03	<0.04	<0.04	<0.04	<0.04
Ho	<0.007	<0.008	<0.008	<0.008	<0.03	<0.03	<0.007	<0.01	<0.01	<0.01	<0.01
Er	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1	<0.03	<0.04	<0.04	<0.04	<0.04
Tm	<0.007	<0.008	<0.008	<0.008	<0.03	<0.03	<0.007	<0.03	<0.03	<0.01	<0.01
Yb	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1	<0.03	<0.1	<0.03	<0.03	<0.03
Lu	<0.007	<0.008	<0.008	<0.008	<0.03	<0.03	<0.007	<0.03	<0.01	<0.01	<0.01
Hf	<0.03	<0.03	<0.08	<0.08	<0.2	<0.2	<0.03	<0.1	<0.4	<0.1	<0.03
Ta	≤20. (e)	1.	1.	3.	10.	6.	--	--	--	≤10.	≤2.
Re	12.	--	--	--	≤40. (d)	10.	5.	8.	8.	8.	8.
Os	<0.3	<0.3	<0.3	<0.3	--	--	<0.3	<1.	<2.	<1.	<1.
Ir	<0.2	8.	<0.2	<0.2	<0.6	<1.	<1.	<0.5	<1.	--	--
Pt	<1.5	<4.	<3.	<4.	<3.	<4.	<6.	<1.	<1.2	<2.	<3.
Au	<1.5	<1.	<3.	<1.	<0.6	<1.	<2.	<5.	<4.	<1.	<1.
Hg	<8.	<10.	<10.	<10.	<10.	<20.	<6.	<8.	<8.	<10.	<10.
Tl	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.5	<0.2	<0.2
Pb	<0.7	<0.3	<0.3	<0.3	<0.3	≤0.7	<0.7	<0.7	<0.7	<0.2	<0.7
Bi	<0.4	<0.2	<0.2	<0.2	<0.2	<0.2	<0.4	<0.2	<0.4	<0.2	<0.4
Th	<0.15	<0.15	<0.15	<0.15	<0.15	<0.05	<0.15	<0.15	<0.07	<0.07	<0.07
U	<0.15	<0.15	<0.15	<0.15	<0.05	<0.05	<0.15	<0.15	<0.07	<0.07	<0.07
C	11.	11	11	8	7	18	3.	6.	17.	9.	13.
O	<2.	5.1	7.2	48.5	13.7	4.6	25.	25.	26.	60.	56.
H	<1.	<0.3	0.3	3.6	4.6	<0.3	1.	1.	1.1	<1.	<1.
N	<1.	5.	3.8	14.3	6.7	<0.4	<1.	<1.	<1.	2.	<1.

(a) < - Not detected, less than limit shown; ≤ - presence of element in sample not confirmed; * - appeared to be nonuniform in sample; Int. - interference from W.
(b) Appeared to be nonuniform in successive exposures.
(c) Interference from major elements.
(d) May be pickup from previously analyzed sample.
(e) May be pickup from source parts which are tantalum.

TABLE 4. RESULTS OF CHEMICAL ANALYSIS OF DROP-CASTING SPECIMENS

(PPM, Weight Basis)[a]

Element	Specimen										
	W	W-5Ta	W-10Ta	W-20Ta	W-5Re	W-15Re	W-23Re	W-1Ir	W-2Ir	W-2.5Os	W-5Os
Li	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004
Be	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
B	0.01	<0.001	<0.001	0.002	0.001	0.003	0.003	0.006	0.001	0.003	0.03
F	0.3	<0.005	0.02	0.02	0.01	0.02	0.02	0.005	0.005	0.005	0.1
Na	<0.05	<0.04	<0.04	<0.05	<0.04	<0.04	<0.04	0.08	<0.04	0.04	0.04
Mg	<0.1	0.03	<0.07	<0.15	0.1[c]	<0.07	0.1	0.05	<0.02	<0.08	0.1
Al	1.5	0.07	<0.02	0.04	0.07	0.3	0.1	0.05	0.02	0.2	0.1
Si	3.[b]	≤0.2	1.5	3.[b]	0.5[c]	0.7	0.7	0.5	0.3	1.	1.
P	0.03	≤0.03	0.03	≤0.15	0.02	0.2	0.02	0.01	0.02	<0.01	0.01
S	2.	0.2	0.7	1.5	0.3	0.5	0.4	1.5	≤1.	0.1	0.3
Cl	0.4	0.03	0.1	0.25	0.06	0.2	0.2	0.06	0.06	0.06	0.03
K	0.1	0.2	<0.06	0.06	<0.06	<0.06	<0.06	0.06[c]	0.06	<0.1	<0.1
Ca	0.05	0.02	0.01	0.01	0.02	0.06	0.06	0.04	0.02	0.02	0.01
Sc	<0.05	<0.02	<0.02	<0.05	<0.02	<0.02	<0.02	≤0.03	≤0.02	<0.02	<0.02
Ti	0.15	0.01	0.04	≤0.7	3.[c]	0.2	0.1	0.4	≤0.2	0.2	0.2
V[d]	<0.002	≤0.3	≤0.7	<0.002	≤0.3	≤0.3	≤0.8	≤0.8	≤0.8	≤0.2	≤0.2
Cr	0.08[b]	0.1	0.5[c]	0.04	0.5	3.	4.[c]	2.5[c]	0.1	0.2	0.4
Mn	0.02	0.006	0.03[c]	0.015	0.05	0.2	0.03[c]	0.1	0.03	0.03	0.03
Fe	<10.	0.15	1.[c]	15.	0.7	10.[c]	5.[c]	7.[c]	0.7[c]	0.2	5.
Co	<0.03	<0.003	0.004	<0.03	0.03	0.03	0.03[c]	0.02	0.01	0.01	0.03
Ni	1.[a]	0.5	0.6	<0.15	0.6	2.[c]	1.5	1.5	1.[c]	0.3	1.
Cu	0.05	0.05	0.1	0.5	1.	2.	0.5	0.5	0.5	<0.1	1.
Zn	<0.03	<0.006	0.02	<0.03	<0.02	<0.02	<0.02	<0.02	0.03	<0.02	<0.02
Ga	<0.015	<0.006	<0.006	<0.015	<0.006	≤0.008	≤0.02	≤0.02	≤0.02	<0.02	<0.02
Ge	<0.2	<0.01	<0.01	<0.2	<0.01	<0.01	<0.01	<0.01	<0.01	<0.03	<0.03
As[d]	<0.04	≤0.02	≤0.02	<0.04	<0.02	≤0.04	≤0.02	≤0.02	≤0.02	≤0.03	≤0.03
Se	<0.02	<0.01	<0.01	<0.05	<0.01	<0.01	<0.01	<0.01	<0.01	<0.03	<0.03
Br	<0.02	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.03	<0.03
Rb	<0.015	<0.008	<0.008	<0.015	<0.008	<0.008	<0.008	<0.008	<0.008	<0.03	<0.03
Sr	<0.015	<0.008	<0.008	<0.015	<0.008	<0.008	<0.008	<0.008	<0.008	<0.03	<0.03
Y	<0.05	0.02	<0.005	<0.01	0.005	1.[c]	1.[c]	0.005	<0.005	0.02	0.1
Zr[e]	<1.	≤2.	≤1.2	≤3.	≤1.	≤2.	≤1.	≤1.	<0.3	[≤80.]	[≤80.]
Mo	<1.	0.3	0.6	5.	0.5	1.	0.5	<0.3	<0.2	≤0.4	≤0.4
Ru	<0.15	<0.06	<0.06	<0.15	<0.06	<0.06	<0.06	40.	90.	<0.08	<0.08
Rh	<0.05	<0.02	<0.02	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
Pd	<0.2	<0.06	<0.06	<0.2	<0.06	<0.06	<0.06	<0.06	<0.06	<0.2	<0.2
Ag	<0.1	<0.06	<0.06	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Cd	<0.2	<0.08	<0.08	<0.2	<0.08	<0.08	<0.08	<0.08	<0.08	<0.1	<0.1
In[d]	<0.05	≤0.1	≤0.2	<0.05	≤0.1	≤0.1	≤0.1	≤0.4	≤0.4	≤0.3	≤0.3
Sn	<0.05	<0.02	<0.02	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
Sb	<0.03	<0.02	<0.02	<0.03	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
Te	<0.05	<0.02	<0.02	<0.05	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
Nb[f]	--	--	--	--	--	--	--	--	--	--	--

TABLE 4. (Continued)

Element	Specimen W	W-5Ta	W-10Ta	W-20Ta	W-5Re	W-15Re	W-23Re	W-1Ir	W-2Ir	W-2.5Os	W-5Os
I	<0.02	<0.007	<0.007	<0.02	<0.007	<0.007	<0.007	<0.007	<0.007	<0.02	<0.02
Cs	<0.02	<0.007	<0.007	<0.02	<0.007	<0.007	<0.007	<0.007	<0.007	<0.02	<0.02
Ba	<0.03	<0.02	<0.02	<0.03	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
La	<0.03	<0.02	<0.02	<0.03	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
Ce	<0.03	<0.007	<0.007	<0.03	<0.007	<0.007	<0.007	<0.007	<0.007	<0.02	<0.02
Pr	<0.03	<0.007	<0.007	<0.03	<0.007	<0.007	<0.007	<0.007	<0.007	<0.02	<0.02
Nd	<0.1	<0.02	<0.02	<0.1	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
Sm	<0.1	<0.02	<0.02	<0.1	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
Eu	<0.06	<0.02	<0.02	<0.06	<0.02	<0.02	<0.02	<0.02	<0.02	<0.06	<0.06
Gd	<0.1	<0.03	<0.03	<0.1	<0.03	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1
Tb	<0.03	<0.01	<0.01	<0.03	<0.01	<0.01	<0.01	<0.01	<0.01	<0.03	<0.03
Dy	<0.1	<0.04	<0.04	<0.1	<0.04	<0.04	<0.04	<0.04	<0.04	<0.1	<0.1
Ho	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1
Er	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.3	<0.3
Tm	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1
Yb	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.3	<0.3
Lu	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03	<0.1	<0.1
Hf	<0.1	<0.05	<0.05	<0.1	<0.05	<0.05	<0.05	<0.05	<0.05	<0.2	<0.2
Ta	2.	--	--	--	8.	8.	15.	10.	>10.	30.	5.
Re	15.	25.	5.	8.	--	--	--	2.	2.	12.	20.
Os	<0.3	<0.3	<1.	<0.3	<0.3	<1.	<1.	<1.	<1.	--	--
Ir	<0.3	0.6	2.	<0.3	0.7	0.5	1.5	--	--	≤1.	≤1.
Pt	<7.	<1.	<1.	<0.7	<1.	<1.	<1.	<1.	<1.	<2.	<2.
Au	<4.	<0.3	<0.3	<10.	<0.3	<0.3	<0.3	<0.3	<0.3	<0.6	<0.6
Hg	<15.	<1.	<1.	<10.	<1.	<1.	<1.	<1.	<1.	<6.	<6.
Tl	<0.2	<0.2	<0.6	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Pb	<0.3	<0.3	<0.8	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.2	<0.2
Bi	<0.15	<0.2	<0.2	<0.15	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Th	<0.15	<0.02	<0.02	<0.03	<0.2(g)	<0.2	<0.3	<0.02	<0.02	<0.2	<0.2
U	<0.15	<0.02	<0.02	<0.03	<0.04(g)	<0.2	<0.2(h)	<0.02	<0.02	<0.2	<0.2
C	7.	4.	4.	10.	13.	8.	5.	8.	13.	11.	8.
O	4.7	3.	9.	9.2	17	6.	3.	2.	3.	2.	5.
H	6.3	0.14	0.7	1.5	0.9	0.5	0.3	0.1	0.3	0.3	0.4
N	1.9	<0.5	<0.5	8.1	<1	<1	<0.5	<0.5	<0.5	<0.5	<0.5

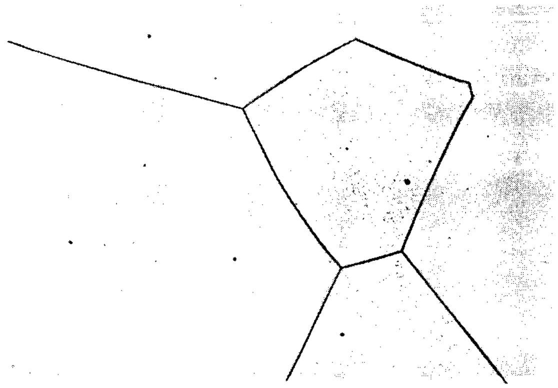
(a) < - Not detected, less than limit shown; ≤ - presence of element in sample not confirmed.
(b) Appeared to be nonuniform in successive exposures.
(c) Appeared to be nonuniform in successive exposures — value is estimated average.
(d) Possible source "memory" from previous samples.
(e) Varying degrees of interference from major elements.
(f) Extreme interference from W.
(g) One exposure showed 0.8 ppm Th and U.
(h) One exposure showed 0.6 ppm U.

TABLE 5. LEVELS OF PURITY DESIRED BY NASA IN TUNGSTEN AND TUNGSTEN ALLOY CASTINGS

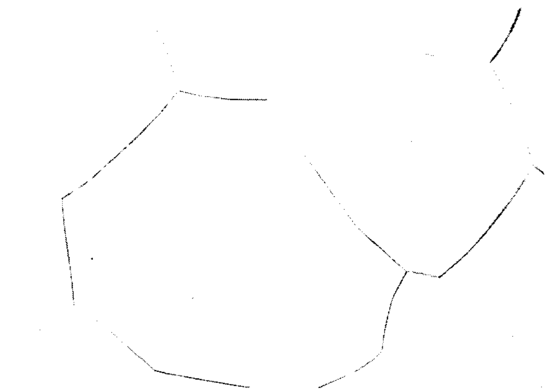
Element	Maximum Desired Content, PPM (Weight Basis)	Group	Element	Maximum Desired Content, PPM (Weight Basis)	Group
Li	10	I A	Hf	10	IV B
Na	10	I A	Th	10	IV B
K	10	I A	Sb	10	V A
Rb	10	I A	Bi	10	V A
Cu	15	I B	V	10	V B
Ag	10	I B	Nb	10	V B
Ca	10	II A	Ta[a]	50	V B
Sr	10	II A	Cr	15	VI B
Ba	10	II A	Mo	250	VI B
Be	10	II A	Mn	10	VII B
Mg	10	II A	Re[a]	50	VII B
Zn	10	II B	Fe	50	VIII
B	10	III A	Co	10	VIII
Al	10	III A	Ni	10	VIII
C	30	IV A	Rh	10	VIII
Si	20	IV A	Pd	10	VIII
Sn	10	IV A	Os[a]	10	VIII
Pb	10	IV A	Ir[a]	10	VIII
Ti	10	IV B	Pt	10	VIII
Zr	10	IV B			

(a) This limit only applied when element was not specifically added.

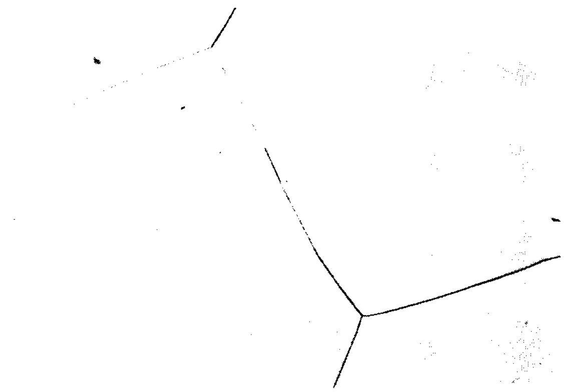
150X RM44337
Tungsten



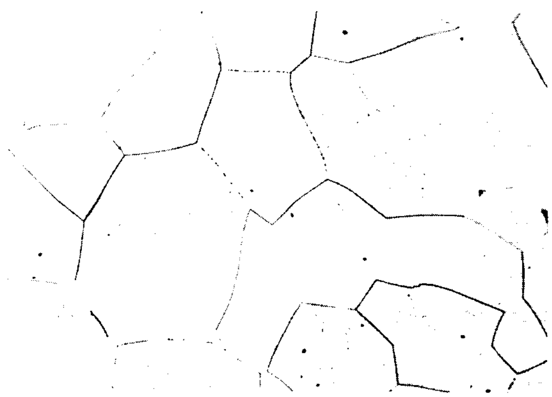
150X RM44353
Tungsten-5 Wt. % Tantalum



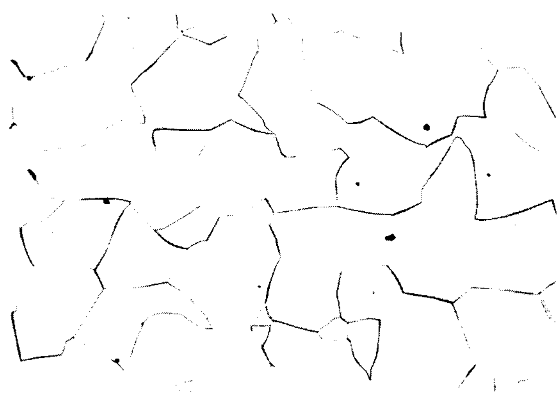
150X RM44355
Tungsten-10 Wt. % Tantalum



150X RM44358
Tungsten-20 Wt. % Tantalum

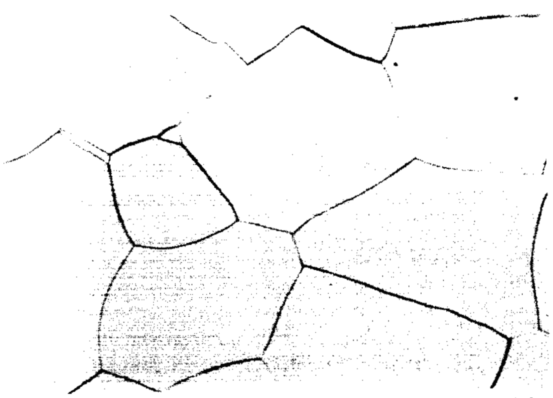


150X RM44362
Tungsten-5 Wt. % Rhenium



150X RM44366
Tungsten-15 Wt. % Rhenium

FIGURE 8. MICROSTRUCTURES OF TUNGSTEN AND TUNGSTEN ALLOY DROP CASTINGS

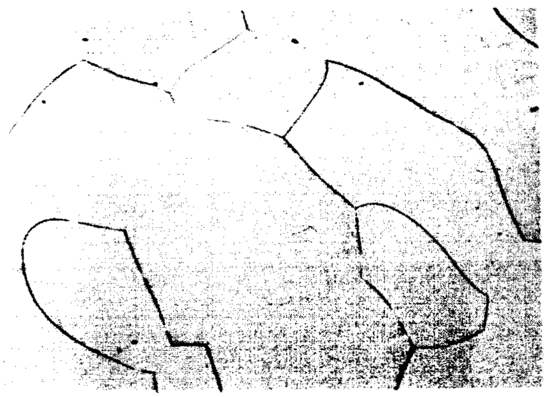


150X RM44368

Tungsten-23 Wt. % Rhenium

150X RM44343

Tungsten-2.5 Wt. % Osmium

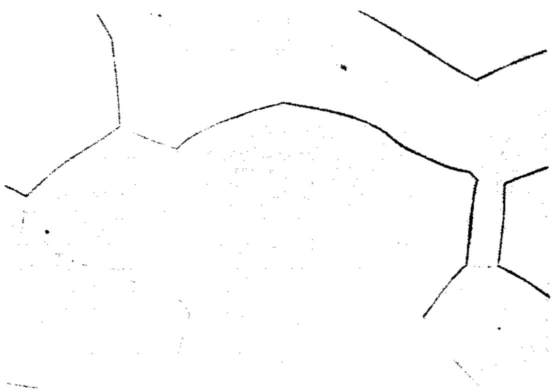


150X RM44347

Tungsten-5 Wt. % Osmium

150X RM44349

Tungsten-1 Wt. % Iridium



150X RM44351

Tungsten-2 Wt. % Iridium

FIGURE 8. (Continued)

tungsten-2.5 weight percent osmium alloy, however, appeared to possess a somewhat cored structure. Also, this alloy and pure tungsten exhibited a subgrain structure upon slightly over-etching.

The grain sizes of drop castings of the different alloys varied. The results of grain size measurements which were determined by using Federal Test Method Standard No. 151 are given below:

Material	ASTM Grain Size
Pure tungsten	No. 2 (1-1/2 to 3 grains/sq in.)[(a)]
	Subgrain size No. 6 (24 to 48 grains/sq in.)
Tungsten-5 tantalum	No. 2 (1-1/2 to 3 grains/sq in.)
Tungsten-10 tantalum	No. 2 (1-1/2 to 3 grains/sq in.)
Tungsten-20 tantalum	No. 1 (up to 1-1/2 grains/sq in.)
Tungsten-5 rhenium	No. 4 (6 to 12 grains/sq in.)
Tungsten-15 rhenium	No. 6 (24 to 48 grains/sq in.)
Tungsten-23 rhenium	No. 3 (3 to 6 grains/sq in.)
Tungsten-2.5 osmium	No. 5-No. 8 (mixed-12 or more grains/sq in.)
Tungsten-5 osmium	No. 2 (1-1/2 to 3 grains/sq in.)
Tungsten-1 iridium	No. 2 (1-1/2 to 3 grains/sq in.)
Tungsten-2 iridium	No. 2-No. 3 (1-1/2 to 6 grains/sq in.).

(a) At 100X.

From the above data, it is apparent that only several alloys possessed grain sizes within the ASTM No. 4 to No. 7 range desired by NASA.

Button castings, in general, possessed grains which varied greatly in size and in shape. Also, considerably more coring appeared to be present in button castings than in drop castings.

Electron-Probe Microanalyses

Electron-microprobe studies were conducted on seven samples of arc-melted button castings of the following compositions:

Tungsten-1 weight percent iridium

Tungsten-2 weight percent iridium

Tungsten-5 weight percent rhenium

Tungsten-15 weight percent rhenium

Tungsten-23 weight percent rhenium

Tungsten-2.5 weight percent osmium

Tungsten-5 weight percent osmium.

After electron-microprobe analysis, all samples were sent to NASA for further study.

The objectives of the microprobe study were to determine quantitatively the area of each sample that was most homogeneous and to determine quantitatively the degree of microhomogeneity in these areas. Profile-type electron-microprobe traverses were made at seven positions on each sample to determine the degree of homogeneity. The positions were the approximate center of the samples and six positions separated by 60 degrees approximately halfway between the center and the edge. The position determined by these traverses to be most homogeneous on a microscale was then analyzed by slow-traversing profile-type analyses to record the concentration variations along a line in the sample.

The areas of the samples found to be most homogeneous on a microscale were identified on the samples. In many of the samples, the degree of homogeneity was approximately the same at all seven positions analyzed. When this occurred, the center position was chosen for the slow-traverse analyses.

The results of the slow-traverse analyses are shown in Figure 9. The graphs shown in this figure are reductions of tracings of the actual readout values of X-ray intensity from the microprobe. Accuracies of the analyses calculated in percent concentration are as follows:

For osmium	± 0.09 percent
For iridium	± 0.15 percent
For rhenium	± 0.15 percent.

All samples, in general, were homogeneous on a macroscale and the samples of tungsten-2 weight percent iridium and tungsten-2.5 weight percent osmium were found to be essentially homogeneous on a microscale at all seven positions analyzed. The sample of tungsten-1 weight percent iridium was more or less homogeneous on a microscale, except for a few widely separated areas rich in iridium. Typical areas are shown on the graph for this alloy at about 20 and 120 microns and contain approximately 3 percent iridium. The tungsten-5 weight percent osmium sample was inhomogeneous at all positions analyzed with variations in concentration from 4 to 6 percent. In addition to the gradual changes in concentration in this sample, abrupt changes occurred indicating the presence of second-phase particles rich in osmium. Examination of the microstructure of this alloy supported this observation. One of these particles was intersected by the traverse at 170 microns as shown on the graph for the tungsten-5 weight percent osmium sample in Figure 9.

All tungsten-rhenium samples were found to be microinhomogeneous at all the positions analyzed. The graph of the tungsten-5 weight percent rhenium sample showed a minimum concentration of about 3.5 weight percent at 48 microns and a maximum concentration of about 6 weight percent at 8 microns. The tungsten-15 weight percent rhenium sample was very microinhomogeneous with large changes in concentration over small distances. The highest peaks on graphs indicated a concentration of 22 weight percent rhenium while the valleys indicated only 12 weight percent rhenium. The tungsten-23 weight percent rhenium sample was fairly homogeneous except for occasional peaks which contained up to 31 weight percent rhenium.

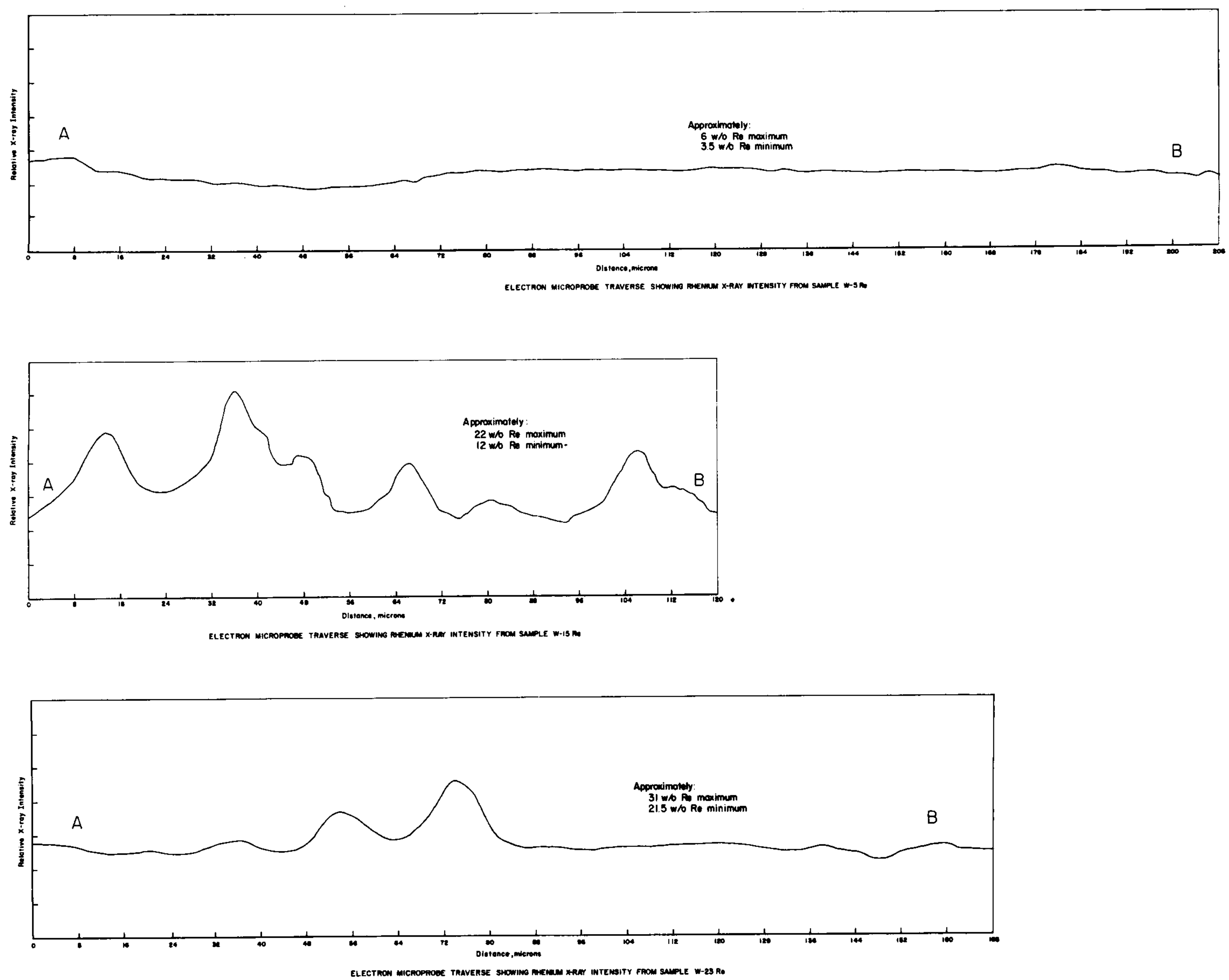
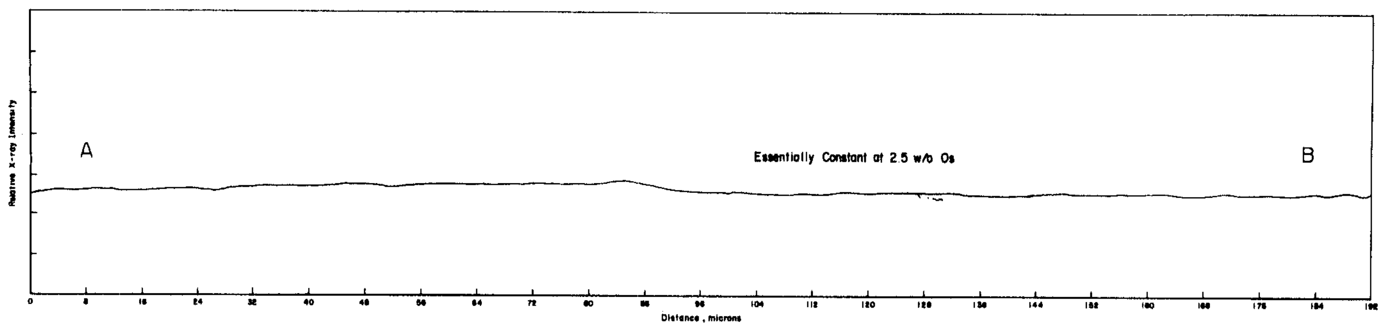
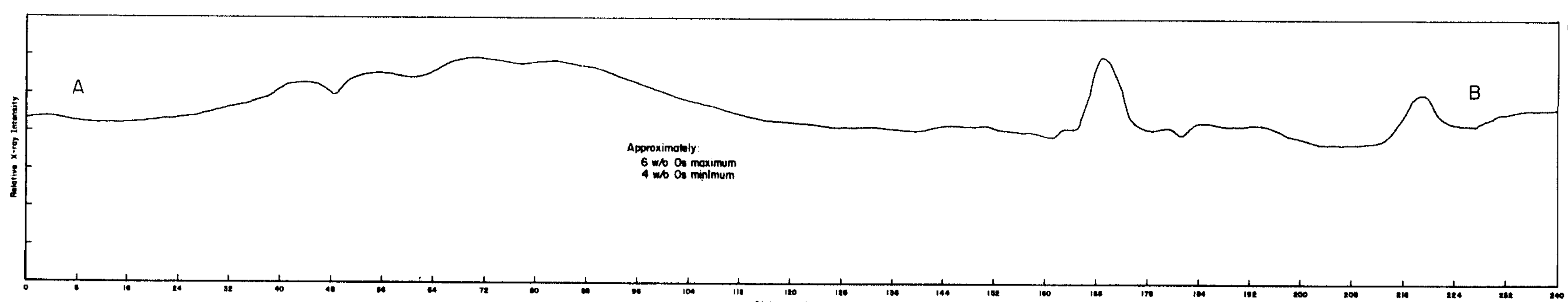


FIGURE 9. ELECTRON-MICROPROBE TRAVERSES

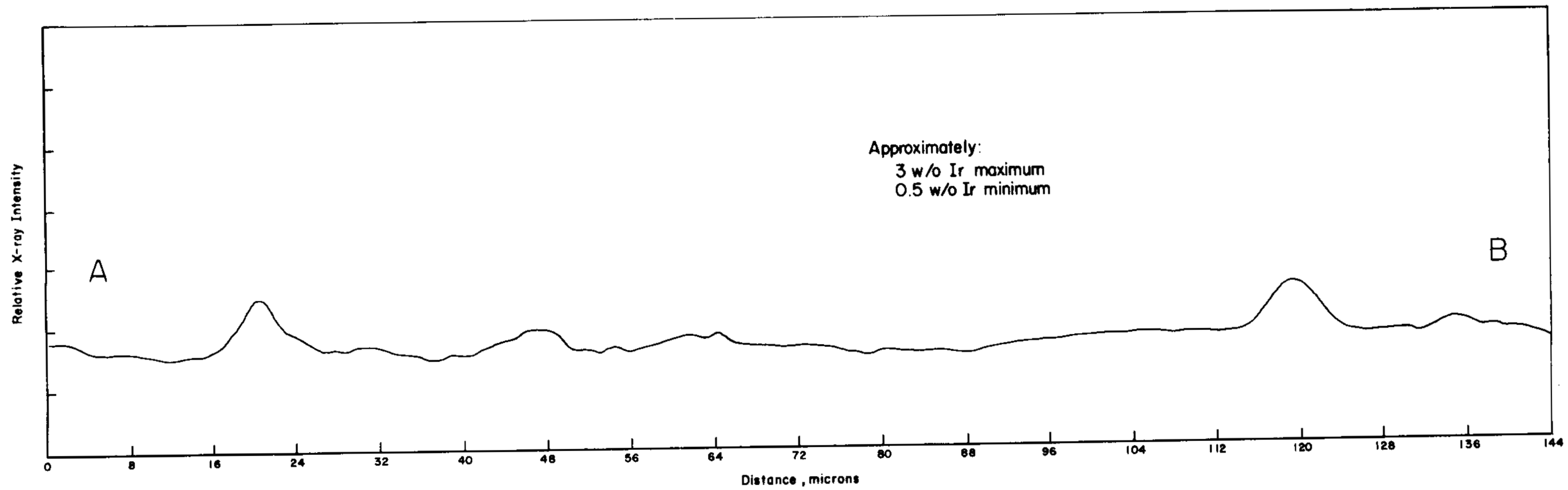


ELECTRON MICROPROBE TRAVERSE SHOWING OSMIUM X-RAY INTENSITY FROM SAMPLE W-2.5 Os

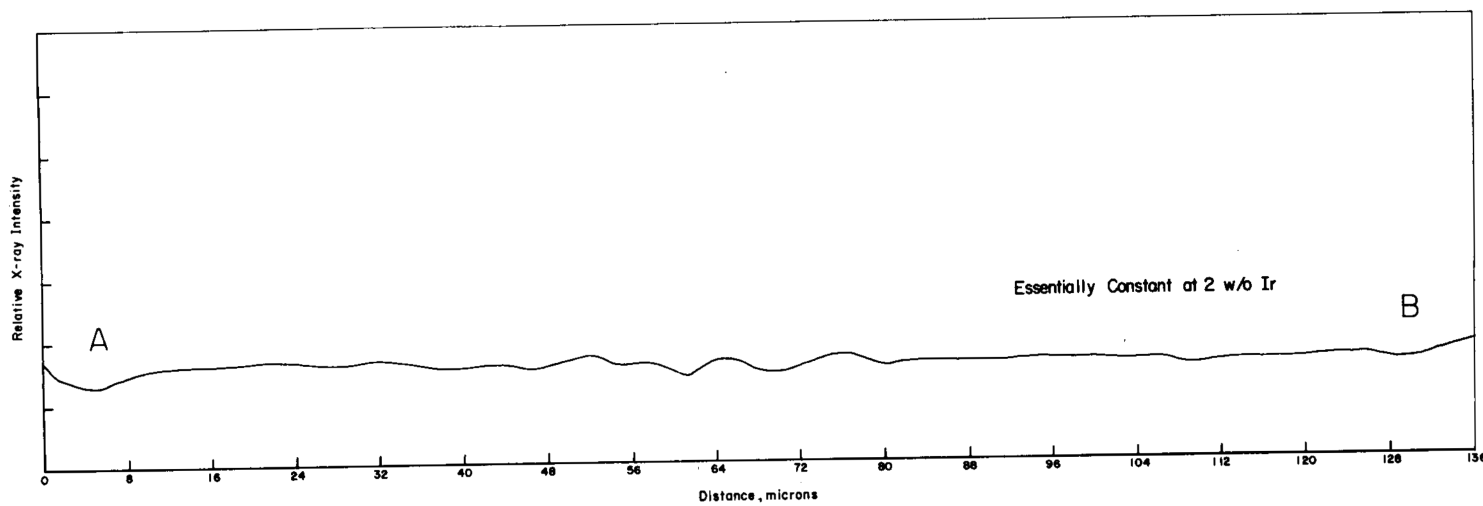


ELECTRON MICROPROBE TRAVERSE SHOWING OSMIUM X-RAY INTENSITY FROM SAMPLE W-5 Os

FIGURE 9. (CONTINUED)



ELECTRON MICROPROBE TRAVERSE SHOWING IRIDIUM X-RAY INTENSITY FROM SAMPLE W-1Ir



ELECTRON MICROPROBE TRAVERSE SHOWING IRIDIUM X-RAY INTENSITY FROM SAMPLE W-2Ir

FIGURE 9. (CONTINUED)

Radiographic and Dye-Penetrant Inspection

Cylindrical specimens, 5/8 in. in diameter and 1/2 or more inches long, were ground for each drop casting. In most cases, two or more drop castings of each alloy composition were prepared. All specimens were examined by standard X-ray radiographic and dye-penetrant techniques. The results of these examinations are given in Table 6. After the examinations, all specimens were delivered to the NASA Lewis Research Center. Figure 10 shows a drop casting of tungsten-5 weight percent osmium and a specimen of tungsten-10 weight percent tantalum.

RM44047

FIGURE 10. A TYPICAL DROP CASTING AND FINAL SPECIMEN

DISCUSSION

The melting and casting to shape of 200-gram charges of ultrahigh purity, fine-grained tungsten or tungsten alloys and maintaining their purities in doing so is not an easy task. Various requirements must be met before such a task can be accomplished. Among these requirements are (1) a high-energy source (for melting), (2) a noncontaminating environment, and (3) a proper mold design. The electron-beam furnace drop-casting technique which was developed and was utilized in this program appeared to have satisfied these requirements except for fine grain size. Satisfactory cylindrical castings 3/4 in. in diameter and from 3/4 in. to 1-3/4 in. long were prepared with a minimum of difficulty.

Perhaps the major problem in the casting of the tungsten alloys was shrinkage. In all alloy drop castings, shrinkage, which resulted in an internal pipe, occurred to some extent. In most cases, however, the shrinkage cavity was located near the top of the casting and was completely or partially removed during the preparation of specimens.

TABLE 6. RESULTS OF X-RAY RADIOGRAPHIC AND DYE-PENETRANT EXAMINATIONS

Composition	Specimen Number	Approximate Length: Full Specimen, in.	Approximate Length: Sound Material, in.	X-Ray Radiographic Observations	Dye Penetrant Observations
Pure W	1	1	>3/4	Sound specimen except for 2 very small voids	No defects
Pure W	2	1	1	Sound specimen	No defects
W-5Ta	1	7/8	13/16	Sound specimen except for 1/32-in. exposed pipe	Pits at top and bottom
W-5Ta	2	1/2	1/4	Sound specimen except for 5/32-in. exposed pipe	Pits at bottom
W-10Ta	1	1/2	7/16	Sound specimen	Pits at bottom
W-10Ta	2	1	1	Sound specimen	No defects
W-20Ta	1	1	13/16	Sound specimen except for 1/8-in. exposed pipe	No defects
W-5Re	1	5/8	1/2	Sound specimen except for 1/8-in. exposed pipe	No defects
W-5Re	2	5/8	5/16	Sound specimen except for 1/4-in. internal pipe	No defects
W-15Re	1	11/16	11/16	Sound specimen	Cold shut on top; pits on the sides
W-15Re	2	5/8	7/16	Sound specimen except for 3/16-in. exposed pipe	No defects
W-15Re	3	7/8	7/16	Sound specimen except for 7/16-in. internal pipe	Satisfactory except for several small pits
W-23Re	1	>1	3/4	Sound specimen except for 1/4-in. internal pipe	No defects
W-23Re	2	1/2	3/8	Sound specimen except for 1/8-in. exposed pipe	No defects
W-1Ir	1	3/4	5/8	Sound specimen except for 3/32-in. pipe at top	No defects
W-1Ir	2	3/4	5/8	Sound specimen except for 1/16-in. internal pipe	Several small pits
W-2Ir	1	3/4	7/16	Sound specimen except for 5/16-in. internal pipe	No defects except rough ends
W-2Ir	2	11/16	5/16	Two small (<1/16-in.) internal voids; otherwise sound	Cold shut on the surface
W-2.5Os	1	1/2	7/16	Sound specimen except for 1/16-in. exposed pipe	No defects
W-2.5Os	2	7/8	9/16	Sound specimen except for 5/16-in. internal pipe	No defects
W-5Os	1	1-3/8	7/8-15/16	Sound specimen except for 3/8-in. internal pipe	No defects except hole on surface

The achievement of a fine-grain size in the alloys in question was difficult owing to (1) the nature of the alloys — all alloys were single-phase solid solutions and, therefore, no nucleation of a second phase could take place, and (2) the purity of the alloys — few impurities were present which could act as nucleation sites. The target as-cast grain size range for the tungsten alloy drop castings was ASTM No. 4 to No. 7. This grain-size range was achieved in drop castings of tungsten-5 and -15 weight percent rhenium and of tungsten-2.5 weight percent osmium and, to a lesser extent, in drop castings of tungsten-23 weight percent rhenium and of pure tungsten. The grain size of all tungsten-tantalum and tungsten-iridium alloys, on the other hand, was notably larger (ASTM No. 1 or 2). Grain sizes much smaller than ASTM No. 2 in these latter alloys are believed not possible by this technique unless the size (particularly diameter) of the final castings is reduced.

Purification of the tungsten alloys was expected upon electron-beam melting. Close examination of the analyses of materials before and after electron-beam melting will show that some purification did occur (for example, oxygen levels were reduced in most cases), although it must be concluded that, in general, the overall purity of materials remained essentially constant. Perhaps one reason why no noticeable amount of purification occurred was simply that the purity of the materials was so high to begin with that further purification was difficult to achieve.

CONCLUSIONS

Based upon this study, the following conclusions can be made:

(1) Macrohomogeneous, 50-gram tungsten alloy button castings can be prepared with little difficulty in a nonconsumable tungsten-tipped electrode arc furnace. Little or no copper pickup from the crucible and only slight attack of the electrodes occur. Great difficulties, however, are usually encountered if attempts are made to prepare alloy buttons with weights greater than 50 grams in the arc furnace. Inhomogeneity of castings and/or severe copper and electrode attack may be expected.

(2) Cylindrical castings of tungsten and tungsten alloys 3/4-in. in diameter and up to 1-3/4-in. long can be prepared by an electron-beam furnace drop-casting technique which was developed at Battelle. Castings prepared by this technique retain or improve the purities of the starting materials.

(3) The shrinkage which takes place in tungsten alloy drop castings is perhaps the most serious problem in the use of the electron-beam drop-casting technique. Shrinkage usually results in a single pipe that generally occurs at or near the top of the casting. With exception of pipes, however, most castings appear X-ray sound.

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