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Production of Ir-0.3% W Disks and Foil

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OAK RIDGE NATIONAL LABORATORY

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APRIL 1975

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PRODUCTION OF Ir-0.3% W DISKS AND FOIL

D. N. Braski and A. C. Schaffhauser

ABSTRACT

The production of Ir-0.3% W disks (2 3/8 in. diam $\times$ 0.025 in. thick, 60 $\times$ 0.64 mm) for fuel cladding used in the Multi-Hundred Watt (MHW) isotopic heat source is described. Cleaning of disk surfaces was improved by electrolytic etching in KCN. Rolling temperatures were raised 100°C because production batches of iridium alloy exhibited 100°C higher recrystallization temperatures than earlier material. Improved surface finishes were obtained by using a fine grit alumina grinding wheel. A total of 484 disks were fabricated, of which 415 met specifications. Also, 687 in.² (0.443 m²) of 0.005-in.-thick Ir-0.3% foil was also manufactured for the MHW heat source.

INTRODUCTION

The Multi-Hundred Watt (MHW) isotope heat source and thermoelectric generator are being developed by the Space Nuclear Systems Division of the U.S. Atomic Energy Commission to provide electrical power for a variety of space missions. A schematic diagram of the MHW heat source is shown in Fig. 1. Iridium was selected for primary containment of the ²³⁸PuO₂ fuel spheres in the heat source because of its unique combination of high melting point, high-temperature strength, oxidation resistance, and compatibility with PuO₂. During operation, the maximum fuel-cladding interface temperature will be around 1400°C. The iridium-clad ²³⁸PuO₂ spheres are covered with a tough shell of fiber-wound graphite, which provides impact resistance in case of a mission abort and earth impact. The MHW heat source uses 24 fuel spheres to produce 2400 W of thermal energy.

During the past two years, our laboratory has carried out the development and production of Ir-0.3% W sheet for fuel cladding. This report describes the fabrication of 0.0255-in.-thick (0.648 mm) Ir-0.3% W alloy disks and 0.005-in.-thick (0.13-mm) foil. Finished disks were shipped to Mound Laboratory in Miamisburg, Ohio, where they were warm-hydroformed into hemispheres and welded together to encapsulate fuel spheres. Ir-0.3% W foil was used for weld backup strips and covers for the vent hole to permit decontamination of the cladding after fueling. The results of development work on the iridium alloy disks dealing with surface cleaning, rolling schedules, and surface grinding are also discussed. A summary of the production yield for both disks and foil is presented together with a tabulation of disks that failed to meet specifications.

MHW HEAT SOURCE

Y-121314

Fig. 1. Multi-Hundred-Watt Heat Source.

PRODUCTION OF Ir-0.3% DISKS

A flow chart showing the fabrication steps used to produce Ir-0.3% W disks is given in Fig. 2. These steps will be briefly described in the following paragraphs. Detailed descriptions of several operations, such as powder compact preparation, electron-beam melting, and nondestructive testing (NDT) will be reported separately in other publications.

Fig. 2. Production of Ir-0.3% W Disks.

Powder Compact Preparation

The iridium powder used to prepare cladding material for the MHW Program was obtained from the Office of Emergency Preparedness (OEP) in 40- to 60-kg shipments. The powder was gray and is shown in Fig. 3. The iridium powder was first blended for 2 hr in a V-blender and then washed in deionized water to remove impurities and extremely fine

Fig. 3. As-Received Iridium Powder.

iridium that floated to the surface. The water was subsequently decanted and the powder oven-dried at 100°C for at least 4 hr. To remove any oversize iridium particles, the powder was screened through a 100-mesh screen using a sieve shaker. The quantity of oversize iridium particles amounted to about 2% of the total powder screened, while the residue from the water wash was about 1%.

Tungsten powder with a purity of 99.97% and a particle size of -400 mesh was treated at 1000°C for 1 hr in a hydrogen furnace to remove the oxygen. The tungsten powder was then blended with the iridium powder to make a mixture containing 0.3 wt % W. The powder was blended in 2700- to 3000-g batches, with each batch designated as a lot for subsequent melting and fabrication. The chemical analyses of the various jars of powder making up one shipment of iridium are listed in Table 1 along with the analysis of a typical composite to which tungsten had been added. This was the shipment from which production-run compacts designated WE were made. Total impurities excluding carbon, oxygen, and tungsten were about 550 ppm.

The powder lot was next cold-pressed into 18 to 21 compacts, nominally 150 g each, with a pressure of 50 tons/in.² (0.69 GPa). The pressed compacts were presintered at 1000°C for 1 hr in a hydrogen furnace and sintered at 1500°C for 4 hr on a platinum hearth in a vacuum (pressure less than 10^{-4} torr or 0.1 MPa). The iridium alloy compacts were $1 \times 1.8 \times 0.25$ in. ($25 \times 46 \times 6.4$ mm) and are illustrated in Fig. 4. The compacts fabricated from each shipment of powder (i.e., compacts that had the same chemical analysis) were given designations of WA, WB, WC, etc. The "W" shows the iridium compact as having been alloyed with tungsten, while the consecutive alphabetic letters refer to specific shipments of powder.

Electron-Beam Melting

According to previously published studies on iridium,^{1,2} information available on commercially produced material used in early heat source development work, and our preliminary studies, the fabricability and properties of iridium were obviously extremely sensitive to low levels of trace impurities. Since high-purity iridium powder was available only in small quantities and material refined from a number of sources would have to be used, electron beam melting was selected as the most practical method for obtaining a reproducible product.

To produce an ingot for rolling it was necessary to melt the sintered iridium compacts into buttons. This was accomplished by electron-beam

¹W. Betteridge, "The Consolidation Working and Properties of Iridium and Ruthenium," pp. 525-51 in *Metalle für die Raumfahrt* (Proc. 5th Plansee Seminar, Reutte, Tirol, Austria), ed. by F. Benesovsky, Metallwerk Plansee AG., Reutte, Tirol, Austria, 1965.

²R. W. Douglas et al., *Summary Report on High-Temperature Properties and Alloying Behavior of the Refractory Platinum-Group Metals*, AD-265624 (1961).

Table 1. Spark-Source Mass-Spectrographic Analysis of Iridium Powder
From the Shipment Used in WE Compacts^a

Element	Content (ppm by weight) in Each Jar												Composite ^b
	1	2	3	4	5	6	7	8	1A	2A	3A	4A	
Ag	3	100	10	10	3	1	1	1	30	3	10	10	-
Al	20	7	20	7	20	20	4	1	1	1	1	10	20
As	0.1	<0.1	<0.1	0.1	0.1	0.1	<0.1	0.1	0.1	0.1	0.1	0.1	0.1
B	2	0.6	0.6	0.6	0.6	0.6	0.6	0.6	2	2	0.6	0.6	0.6
Ba	20	7	7	7	7	7	20	7	<0.1	<0.1	0.2	<0.1	2
Br	8	8	8	8	8	8	8	8	2	2	2	2	3
Ca	60	60	29	29	29	20	20	20	200	200	200	200	60
Cl	80	80	80	80	80	300	300	80	80	80	80	80	30
Co	0.1	0.1	<0.1	<0.1	<0.1	0.1	<0.1	<0.1	0.1	0.1	<0.1	0.1	0.1
Cr	1	1	1	1	1	1	1	1	1	1	3	1	0.8
Cu	5	5	5	5	5	5	5	2	5	5	5	5	5
Fe	20	50	20	20	20	20	20	20	50	20	50	50	100
Hf	1	1	1	1	1	1	1	0.3	<0.1	<0.1	<0.1	<0.1	-
K	200	60	0	60	60	60	60	60	60	60	60	60	20
Li	0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	0.1	-
Mg	10	10	10	10	10	10	10	10	3	3	3	30	40
Mn	1	1	1	1	1	1	1	1	1	1	1	1	-
Mo	<0.3	<0.3	1	1	<0.3	1	<0.3	1	<0.3	<0.3	<0.3	<0.3	4
Na	4	4	4	1	4	4	4	4	4	4	1	4	4
Nb	<0.1	<0.1	0.1	0.1	<0.1	0.1	<0.1	0.1	<0.1	<0.1	<0.1	<0.1	-
Ni	1	1	1	1	1	1	1	1	1	1	1	1	2
Os	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	-
P	20	5	5	5	5	5	5	2	0.5	0.5	2	2	2
Pb	6	6	6	6	6	2	2	6	6	6	2	2	-
Pd	2	6	2	2	2	2	0.6	0.6	2	2	6	2	2
Pt	4	4	4	4	4	4	1	4	1	4	1	4	-
Rb	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.7	0.2	0.7	0.7	0.2	-
Re	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	-
Rh	500	500	500	500	500	200	500	200	5	5	20	20	50
Ru	5	2	5	5	5	2	5	2	2	2	2	2	2
S	40	10	40	40	40	40	40	40	40	40	100	40	100
Sb	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1	-
Si	90	90	90	30	90	90	90	30	30	9	30	30	90
Sn	0.6	0.6	0.6	0.2	-	0.2	-	-	0.2	0.6	0.6	0.6	-
Ta	3	3	3	3	3	3	3	3	3	3	3	3	0.3
Te	-	-	-	-	-	-	-	-	-	-	-	-	0.6
Ti	2	2	0.7	0.2	0.2	0.2	<0.1	<0.1	0.5	0.5	0.5	0.5	2
V	<0.1	0.1	0.1	0.1	0.3	0.1	<0.1	0.1	<0.1	0.1	<0.1	0.1	0.1
W	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	<0.3	3300
Zn	3	10	3	3	3	3	3	3	3	3	3	3	10
Zr	10	10	10	10	10	10	10	3	0.3	0.3	0.3	0.3	-

^a(-) means not determined.

^bBlend of all jars after washing, screening, and addition of 0.3 wt % W.

Fig. 4. Sintered Iridium Compacts.

melting in a seven-cavity, water-cooled copper hearth shown in Fig. 5. Three compacts, each weighing about 150 g, were placed in each cavity and melted into a button (see Fig. 6). Each button was subsequently turned and remelted twice to remove gases and other volatile impurities present in the compacts. The buttons were then cleaned in a solution

Fig. 5. Seven-Cavity Copper Hearth.

PHOTO 1121-74

Fig. 6. Electron-Beam-Melted Ir-0.3% W Button.

of aqua regia (3 parts concentrated HCl: 1 part concentrated HNO_3) for 5 min followed by a 5-min soak in aqua regia plus 10% HF. Next, a three-cavity, water-cooled copper hearth, shown in Fig. 7, was used with the electron-beam furnace to consolidate each button with about 650 g of iridium alloy heads and skulls into a large button. The heads and skulls were obtained from previous melts and will be described later in this section. The large iridium buttons were acid cleaned in the same manner as the smaller buttons and electron-beam drop-cast into a $3/4 \times 3/4 \times 2\frac{1}{3}$ -in. (19- $\times$ 19- $\times$ 59-mm) water-cooled copper mold. Figure 8 is a

PHOTO 1118-74

Fig. 7. Three-Cavity Copper Hearth for Drop Casting.

Fig. 8. Drop-Casting Mold.

photograph of the split mold used in drop-casting. Details of the electron-beam drop-casting technique have been published.³ The furnace pressure during drop-casting was 2×10^{-4} torr (27 MPa) or lower. After the ingot was drop-cast it was allowed to cool at least 1 hr before the furnace was opened. A drop-cast ingot and skull are shown in Fig. 9. The flat annular ring or skull was the portion left in the upper portion of the mold after drop-casting. The "head" or top portion of the casting has been cut off with an abrasive (Al_2O_3) cut-off wheel, leaving the ingot. As outlined in the flow chart (Fig. 2), the heads and skulls were recycled and melted with small buttons (melted compacts) to produce more large buttons.

Ingot Preparation

After the casting heads were cut off with an abrasive cut-off wheel, the resultant ingots were cleaned in separate baths of aqua regia and aqua regia plus 10 wt % HF. The ingots were rinsed in water, dried, and cleaned in acetone with ultrasonic agitation. The top ends of the ingots were always placed downward in the ultrasonic bath to permit removal of debris (left from cutting of the head) from the shrinkage pipes. The shrinkage pipes were sealed by electron-beam welding to prevent cracking of the ingots during the subsequent warm rolling operations. If filler material was required for the repair, it was taken from the skull associated with that particular ingot. After weld repair of the shrinkage pipes,

³C. W. Dean and R. E. McDonald, *High Purity Shape Casting with an Electron-Beam Furnace*, ORNL-TM-1935 (October 1967).

Fig. 9. Ir-0.3% W Casting, Head, and Skull.

most ingots were left with cavities similar to the one shown in the radiograph in Fig. 10. Two radiographs were taken of each ingot, at 90° orientations of each other, using a 2000-kV x-ray source. Since the weld repairs were performed in vacuum, the cavities contained no gas and were eliminated by diffusion bonding during rolling of the ingot. Following weld repair and radiography of the ingots, the two opposite surfaces of the ingot that were to contact the rolls were ground or "conditioned" by means of a small hand grinder with a tungsten carbide burring tool. Grinding away the small pits or cold shuts greatly reduced the chances of edge cracking and delamination of the iridium alloy sheets during subsequent rolling. Iridium ingots before and after surface conditioning are shown in Fig. 11. The electron-beam weld repairs of the shrinkage pipes are also evident on the ends of the ingots. After surface conditioning, the ingots were again cleaned in aqua regia and annealed at 1500°C for 4 hr in vacuum to homogenize the cast iridium microstructure.

Rolling of Sheet

Before being rolled, the iridium ingots were fitted into molybdenum frames and shimmed with molybdenum sheet for a tight fit. Molybdenum cover sheets, 0.060 in. (15 mm) thick, were tightly folded over the frame and lapped over to lock the frame in place. The assemblies were preheated at 1200°C for 1 hr in air and rolled in four passes to a thickness of 0.237 in. (6.02 mm). Each pass produced a 25% reduction in sheet thickness. The sequence of the passes was one longitudinal roll, two cross rolls, and a final longitudinal roll. The assemblies were reheated at

Y-127678

Fig. 10. Radiograph of Cavity in Ir-0.3% W Casting, Which Has Been Sealed by Electron-Beam Welding.

Fig. 11. Ir-0.3% W Ingots Before (left) and After (right) Surface Conditioning. Electron-beam repair welds are seen as depressions in the tops of the ingots.

1200°C for 10 min between passes. An iridium ingot in a molybdenum frame after breakdown rolling is shown in Fig. 12. The iridium plates were recrystallized at 1300°C for 1 hr in vacuum. Further warm rolling was conducted at 1100°C in 10% longitudinal passes until a 50% total reduction was reached. The preheat temperature was then reduced to 1050°C and the sheets were rolled longitudinally to a thickness of about 0.035 in. (0.9 mm). The iridium sheet was removed from the cover sheet and frame and bare rolled longitudinally at 1050°C to a final thickness of 0.033 ± 0.001 in. (0.84 mm). The as-rolled sheet was cleaned for 10 min in a solution of 10 parts (by volume) H_2SO_4 , 45 parts HNO_3 , and 45 parts H_2O . Finally, the iridium sheets were annealed at 850°C for 1 hr between heavy Incoloy 800 platens. Iridium "waster" sheets were used to prevent the rolled sheets from being contaminated by direct contact with the Incoloy platens. The purpose of this 850°C anneal was to flatten and stress relieve the iridium sheets to facilitate machining.

Machining of Disks

Iridium alloy disks, 2 3/8 in. (60.3 mm) in diameter were cut to size by electrical discharge machining (EDM). Normally four disks could be machined from each sheet. In addition, a 0.9-in. (23-mm) test disk was EDM cut from the sheet for metallography, chemical analysis, and archive samples. A typical set of four production disks and a test disk is shown in Fig. 13. The remaining portion of the rolled sheet was called the "skeleton". After all the iridium powder from a specific lot was used, all the skeletons left from production of disks were recycled to make ingots (refer to the flow line across the bottom of Fig. 2). Disks manufactured from recycled material were given a "R" suffix (e.g., disks fabricated from recycled WE material were labeled WER). The production disks and test disks were deburred by hand filing to permit good sealing in the vacuum chuck used for surface grinding. Equal amounts of material were ground from each face of the disks so that a final thickness of 0.0255 ± 0.0005 in. (0.648 mm) was attained. The identification number and rolling direction were indicated on each disk by filing tiny notches on the edge. The disks were then inspected and the following quantities measured: (1) diameter, (2) thickness at least six randomly selected positions, with a maximum and minimum thickness reported, and (3) surface finish.

Cleaning, Sampling, and Annealing of Disks

The iridium disks were electrolytically treated in a bath containing 130 g of KCN per liter of H_2O . An ac voltage of 9 V was applied across each disk for 5 min, removing about 0.0005 in. (13 μm) of material from each face. Because of the potential danger of toxic vapors with this process, it was conducted in a well-ventilated fume hood. Next, the disks were weighed to the nearest milligram as part of an identification check set up by the Quality Assurance Plan. (As seen on the flow chart in Fig. 2, the disks were weighed again just before shipment to assure

Fig. 12. Ir-0.3% W Ingot in Molybdenum Frame After Breakdown Rolling at 1200°C.

Fig. 13. Ir-0.3% W Disks, Test Disk, and Skeleton.

that they had not been inadvertently interchanged in their plastic envelopes.) After weighing, the disks were cleaned in a solution of 30 parts HF to 70 parts HNO_3 at 90°C for 10 min. Next, they were soaked in a bath of concentrated HNO_3 at 90°C for 5 min. It was necessary to conduct these hot acid-cleaning steps in a special acid fume hood with Teflon tanks and tongs. The disks were rinsed several times in deionized water and then in absolute ethyl alcohol, dried with warm air, and packaged in separate clean plastic envelopes. The disks were given a final stress relief anneal at 950°C for 15 min in vacuum. White-glove handling of the acid-cleaned disks was required through this last step to avoid introducing any surface contamination that might diffuse into the bulk material during the final annealing treatment.

Referring back to the flow chart (Fig. 2) shows that samples cut from the test disk were sent to metallography for certification of the microstructure. Experimental hydroforming tests at Mound Laboratory on disks made at this laboratory determined that the optimum microstructure of the iridium blanks should be fibrous and should not contain any more recrystallized grains than the partially recrystallized Ir-0.3% sheet standard shown in Fig. 14. Other samples were submitted for chemical analysis of the surface and bulk by spark source spectrometry. Carbon and oxygen contents were also measured by use of the techniques of combination thermal conductivity and vacuum fusion, respectively. Iridium sheets (four disks) not passing specifications with regard to microstructure or chemical analysis were identified on QA nonconformance reports (NCR).

Meanwhile, the iridium disks themselves were subjected to nondestructive tests as outlined in the flow chart in Fig. 2. A special closed-circuit television system with a sample-to-screen magnification of $10\times$ was used to visually check the disk surfaces for flaws. The depth of any flaws noted was measured at $200\times$ with a calibrated optical microscope. The disks were given dye-penetrant and ultrasonic inspections to detect cracks, fissures, or internal flaws not detected by the visual examination. Disks failing to pass specifications were rejected and identified on a nonconformance report. In some cases, disks with nonconformance reports were shipped to Mound Laboratory for use as fabrication samples, depending on the severity of the defects.

Following the nondestructive tests, the disks were ultrasonically cleaned in acetone and ethyl alcohol to remove all examination markings, dye penetrant, etc. and reweighed for identification. The disks along with samples cut from the test disk were shipped to Mound Laboratory.

PRODUCTION OF Ir-0.3% W FOIL

Iridium alloy foil, nominally 0.005 in. (0.13 mm) thick, was used for weld shields and decontamination covers in the MHW source. Foil fabrication began with 0.033-in.-thick (0.84-mm) sheet produced according to the steps outlined in the flow chart in Fig. 2. The iridium sheet was rolled at 1050°C without cover sheets to a thickness of about 0.025 in. (0.64 mm). The sheet was subsequently recrystallized by annealing in air at 1000°C for 15 min and then warm rolled at 850°C between cover sheets of Incoloy 800 to a thickness of 0.005 in. (0.13 mm). The foil was cleaned by the

Fig. 14. Ir-0.3% W Metallographic Standard for Quality Assurance. 100 $\times$.

same procedures used for disks. Sampling was also similar, except that the test samples were cut from the end of the foil sheet rather than from test disks.

A fully recrystallized microstructure with a grain size less than ASTM-5 was specified by Mound Laboratory for the foil. Annealing studies indicated that the foil rapidly recrystallized above 900°C, and complete recrystallization was observed after 15 min at 100°C in vacuum. A fully recrystallized microstructure in iridium foil is illustrated in Fig. 15.

Inspection of iridium alloy foil included metallographic examination, bulk spark-source spectrochemical analysis, carbon and oxygen determinations, and dye penetrant. The visual examination of foil consisted of inspecting the surfaces with a large-area 5 $\times$ magnifier. Any slivers, burrs, blisters, laps, cracks, or other surface imperfections detected at this magnification were noted on the inspection report. The weight and approximate surface area of the foil was measured as a final inspection before shipment to Mound Laboratory.

PROCESS DEVELOPMENT

The production processes described are the end result of numerous months of development work in the area of iridium fabrication. Much of

Fig. 15. Fully Recrystallized Ir-0.3% W 0.005-in.-thick (0.13 mm) foil. 440 $\times$.

this development effort has been reported previously.⁴⁻⁶ As with many development programs, problems arose during final production runs and had to be solved. The following paragraphs describe several such problems and the steps taken to solve them.

Surface Cleaning

Before full production of disks began, General Electric scientists reported⁷ that aluminum was detected in the grain boundaries of hemispheres

⁴R. G. Donnelly and A. C. Schaffhauser, *Space Power Material Development Program Quart. Progr. Rep. Sept. 30, 1972*, ORNL-TM-4057, pp. 1-12.

⁵R. G. Donnelly, *Space Power Material Development Quart. Progr. Rep. Dec. 31, 1972*, ORNL-TM-4165, pp. 1-10; *Ibid. Mar. 31, 1973*, ORNL-TM-4264, pp. 1-14.

⁶R. G. Donnelly, *Isotopic Power Materials Development Progress Report for April 1973*, ORNL-TM-4263, pp. 1-8; *Ibid. May 1973*, ORNL-TM-4313, pp. 1-6; *Ibid. June 1973*, ORNL-TM-4348, pp. 1-8; *Ibid. July 1973*, ORNL-TM-4372, pp. 1-4; *Ibid. August 1973*, ORNL-TM-4383, pp. 1-2; *Ibid. September 1973*, ORNL-TM-4414, pp. 1-2; *Ibid. October 1973*, ORNL-TM-4445, pp. 1-8.

⁷R. G. Frank, W. C. Wyder, and A. C. Schaffhauser, *Backup Iridium Hemisphere Program Task Force Report*, GESF-744 (April 1974).

fabricated from ORNL sheet. The aluminum was detected by ion mass analyses (IMA) in grain boundaries near the inner and outer surfaces of the spheres, suggesting that the aluminum had diffused inward from the surface. Although this finding was not shown to be deleterious, the general consensus of opinion at that time was that unless the effect of aluminum in the grain boundaries on impact properties was understood it would be safer to eliminate the aluminum. Therefore an investigation was initiated to determine methods for removing aluminum and other impurities such as silicon from the iridium surfaces, which might diffuse into the microstructure during annealing. Analysis by spark source spectroscopy of an as-ground iridium surface showed 3000 ppm Al at the surface, dropping to about 30 ppm at a depth of approximately 100 μm . When the same surface was acid cleaned before analysis according to the procedure outlined previously, the aluminum concentration at the surface was reduced to less than 10 ppm. Based on these results, the acid cleaning treatment was adopted for use in the production line. Further analysis of other iridium samples by auger spectroscopy support the spark source findings, as illustrated by the results in Table 2. Sample WC-28 was surface ground with an alumina grinding wheel and then annealed at 850°C for 15 min. Very high concentrations of

Table 2. Auger Analysis of Acid-Cleaned Iridium Surfaces

Table 2. Auger Analysis of Acid-Cleaned Iridium Surfaces

Sample	Condition	Content, ppm by weight			
		Al	Si	Fe	Ca
WC-28	(a) Ground + 15 min at 850°C	11,000	25,000	7000	5000
	(b) Condition (a) + 25 Å (2.5 nm) removed by sputtering	5000	4000	6000	2000
	(c) Condition (a) + 400 Å (40 nm) removed by sputtering	<500	ND ^a	ND	ND
WC-14	(a) Ground + 1 hr at 1050°C + acid etch	<500	7000	5700	2000
	(b) Condition (a) + 30 Å (3 nm) removed by sputtering	200	400	2500	1000
	(c) Condition (a) + 100 Å (10 nm) removed by sputtering	ND	ND	<600	<600
WC-59	(a) Ground + acid etch	ND	ND	ND	4400

^aND = not detected, <200 ppm.

Al, Si, Fe, and Ca were detected and extended nearly 400 Å deep. Sample WC-14 was ground, annealed for 1 hr at 1050°C, and acid cleaned. These results show that acid cleaning after the anneal greatly reduced the amount of impurities as well as the depth to which they were detected.

Acid cleaning of sample WC-59 directly after surface grinding effectively removed all major contaminants except calcium below the detection of the instrument (<200 wt ppm). Therefore, acid cleaning before the final anneal appeared to remove most of the aluminum.

The results of acid cleaning treatments were further confirmed by other studies on iridium using the ion microprobe. Sample WC-17 was ground, acid cleaned, annealed at 1075°C for 1 hr, acid cleaned, and finally annealed at 1500°C for 18 hr. Any aluminum present on the surface before the 1500°C anneal would probably have had ample opportunity to diffuse into the iridium. Ion probe analysis detected Al, Na, Si, Cr, Fe, and trace amounts of Ca on the iridium surface. However, none of these elements were detected in the grain boundaries. Figure 16 shows edge views of this iridium sample as seen by the ion microprobe. Figure 16(a) is a primary ion current photograph of an area near an edge that shows several grains at the sample surface. The black line between the iridium surface and the epoxy mount was identified as separation between the two. This interpretation was verified by scanning electron microscopy. Figure 16(b) is a secondary ion photograph of the $^{27}\text{Al}^+$ ion taken of the same identical area as shown in Fig. 16(a). Aluminum was found in the separation area, but none had diffused into the grain boundaries. These results and others from the same sample show that acid cleaning before annealing removed any aluminum that might have diffused from the surface into the matrix along grain boundaries. The aluminum and other elements detected on the iridium surface were probably introduced during specimen preparation and/or handling after the anneal and before ion probe analysis. An alternate explanation of where residual contaminants originated, especially aluminum, was that the aluminum was in the form of Al_2O_3 left from the grinding process. Numerous large particles ($\sim 100\ \mu$ in diameter) containing aluminum and silicon were observed on early unetched iridium surfaces by scanning electron microscopy and energy dispersive x-ray analysis.⁸ Since Al_2O_3 is only slightly soluble in acid solutions, it may not have been completely removed by the acid cleaning. Experiments conducted by Darling et al.⁹ showed that platinum will reduce Al_2O_3 in vacuum or a reducing atmosphere. Unpublished work by General Electric¹⁰ indicated similar behavior for iridium and Al_2O_3 . Therefore, to prevent aluminum contamination from Al_2O_3 introduced by surface grinding, an additional cleaning treatment was adopted to remove thin layers of iridium from the blank surfaces. The treatment consisted of an electrolytic etching with KCN (see previous section on Cleaning, Sampling, and Annealing of Blanks). The results of

⁸A. A. Solomon and N. H. Packan, *Relation Between Mechanical Properties and Impurities in Iridium*, Argonne National Laboratory, (ANL-75-4 to be published).

⁹A. S. Darling, G. L. Selman, and R. Rushforth, "Platinum and the Refractory Oxides-I Compatibility and Decomposition Processes at High Temperatures," *Platinum Metals Rev.* 14(2): 54-60 (1970).

¹⁰H. Sayell, General Electric Space Division, private communication.

Fig. 16. Ion Microprobe Photographs of Ir-0.3% W Sample that had been Acid Cleaned and Annealed for 18 hr at 1500°C. 350×. (a) Primary ion current photograph, (b) $^{27}\text{Al}^+$ ion photograph.

Auger spectroscopic analysis on samples with and without a KCN etching followed by acid cleaning are given in Table 3. The cleaned surface was analyzed first and then sputtered *in situ* for 10 min to remove about 10 atom layers. Auger spectra were obtained from these virgin surfaces to determine the approximate thickness of the contamination layer. Surprisingly, the sample cleaned only in acid (WE-2) showed lower aluminum and silicon levels at the original surface than those having the KCN etch. The carbon and calcium were very high for all three samples. However, after 10 atom layers (~ 25 Å, 2.5 nm) were removed, the aluminum and silicon levels remained about the same for WE-2, but decreased markedly

Table 3. Auger Analysis — Effect of KCN Etching Iridium Surfaces

Sample	Condition	Content, ppm by weight			
		Al	Si	C	Ca
WE-1	(a) KCN etch + acid cleaned	1100	1100	51,800	4400
	(b) Condition (a) + 25 Å (2.5 nm) removed by sputtering	400	ND ^a	ND	ND
WE-2	(a) Acid cleaned only	500	800	141,000	14,600
	(b) Condition (a) + 25 Å (2.5 nm) removed by sputtering	700	500	ND	200
WE-3	(a) KCN etched + acid cleaned	1600	1000	151,000	9100
	(b) Condition (a) + 25 Å (2.5 nm) removed by sputtering	500	ND	ND	ND

^aND = not detected, <200 ppm.

in the KCN-etched samples. Therefore, it appeared that the KCN etch was probably successful in producing relatively clean surfaces, which were partially recontaminated upon subsequent acid cleaning and/or handling. In any case, the combination of KCN etching and acid cleaning was adopted for use on the production line for processing all the WE-series iridium alloy disks. Special handling of iridium disks between fabrication at ORNL and hot hydroforming at Mound Laboratory was not considered necessary because the disks were acid cleaned again before hydroforming.

Rolling Schedule

Iridium alloy sheets fabricated before WE-40 were rolled and annealed at temperatures 100°C below those shown on the flow chart (Fig. 2). Also, the bare rolling step at 1050°C was not used before WE-40, and the sheet was warm rolled in the molybdenum cover sheets to a thickness of 0.033 in. (0.84 mm). The need for higher rolling temperatures for the WE lot of material was not immediately obvious, and the problem revealed itself in a rather subtle way. As more of the WE-series disks were fabricated, an increasing percentage was rejected because of moderately deep (>25 μ m) pits in the surface. These pits were caused by the increased surface roughness of the final rolled sheet such that cleanup of the surface by grinding was impossible. Metallographic examination of early WE disks showed that their microstructures often contained large elongated grains such as shown in Fig. 17. This suggested that some of the original cast structure was not being recrystallized by the 1 hr anneal at 1200°C. The technicians conducting the warm rolling also reported that the material work hardened very rapidly, and mill settings had to be changed often. These observations prompted a metallographic study of recrystallization temperatures of the WE material and previous lots of material.

Samples of 0.033-in.-thick (0.84 mm) rolled sheet were cut to 1/4 $\times$ 1/4 in. (6.4 $\times$ 6.4 mm) with an AUH-150-15 abrasive cut-off wheel (ACM Products, Inc., Chicago, Ill.). The samples were annealed in an alumina tube furnace at various temperatures for 15 min and air-cooled to room temperature. The samples were subsequently mounted in Araldite (CIBA Products Co., Fairlawn, N.J.) and vibratorily polished with 0.3- μ m Al₂O₃ followed by 1.5 μ m diamond polishing compounds in water. The samples were then electroetched in a solution of 20 parts HCl and 80 parts H₂O, saturated with NaCl, using 9 V, ac, for 3 to 5 min. Details of the procedure have been reported elsewhere.¹¹ The percentage of recrystallized grains was estimated and averaged over the surface of the polished section. Five diamond pyramid hardness (DPH) values were taken on each section and averaged. The results of this study are given in Figs. 18 and 19. Figure 18 relates the percentage of recrystallization to annealing temperature for an

¹¹D. N. Braski, M. D. Allen, and A. C. Schaffhauser, "Metallography in Iridium Sheet Production," paper presented at the Seventh Annual International Metallographic Society (IMC) Conference, Gatlinburg, Tenn., Aug. 4-7, 1974; to be published in *Microstructural Science*, Vol. 3, ed. by P. M. French, R. J. Gray, and J. L. McCall, American Elsevier, New York, 1975.

Fig. 17. Ir-0.3% W Sheet with Large Elongated Grains. 100 $\times$.

annealing time of 15 min. Early lots of material (WC, WD, and WCR) recrystallized completely between 1025 and 1075°C, while the WE material recrystallized near 1200°C. The corresponding microhardness data in Fig. 19 taken on the same samples show that the microhardness began decreasing between 950 and 1000°C for the earlier material. Microhardness in the WE-1 and -22 samples (rolled at lower temperatures) began decreasing above 1000°C, while WE-42 and WER-95 (rolled at 100°C higher temperatures) demonstrated sharp drops above 1100°C. This result is reasonable because the sheet rolled at the lower temperature would have more stored energy to apply toward the recrystallization process. Also note that microhardness values for fully recrystallized material in all lots of material dropped to approximately 260 DPH. These results clearly show that the WE material recrystallizes approximately 100°C higher than the early material for a similar annealing time. Therefore, to obtain the desired microstructure and mechanical properties of iridium alloy disks, the annealing and rolling temperatures were raised accordingly. An additional warm bare rolling step was added to the end of the revised rolling schedule to ensure that the sheet surfaces would be adequately smooth. The use of the increased rolling and annealing temperatures along with the bare-rolling step for WE material eliminated the problem of surface pits. It is important to note that all the batches of Ir-0.3% W sheet exhibited similar tensile properties after annealing 1 hr at 1500°C. Table 4 lists the results of tensile tests at both room and elevated temperatures for the different batches of sheet material. All sheets tested showed similar

Fig. 18. Percent Recrystallization of Ir-0.3% Sheet as a Function of Annealing Temperature. Annealing time = 15 min.

Fig. 19. Microhardness of Ir-0.3% Sheet as a Function of Annealing Temperature. Annealing time = 15 min.

Table 4. Tensile Properties of Different Sheet Lots of Ir-0.3% W After Annealing 1 hr at 1500°C

Sheet	Test Temperature (°C)	Strength, ksi(MPa)		Elongation (%)
		Yield	Ultimate	
WC-4	room	13.4(92)	55.7(384)	12.1
WCR-93		12.3(85)	58.1(401)	12.9
WD-1		13.0(90)	61.3(423)	14.2
WE-3		13.1(90)	60.4(416)	12.4
WER-94		14.4(99)	54.3(374)	12.0
WC-4	760	7.5(52)	58.1(401)	32.5
WCR-93		8.9(61)	64.6(445)	39.8
WD-1		9.3(64)	58.7(405)	42.5
WE-3		11.0(76)	65.6(452)	34.0
WER-94		8.6(59)	59.1(407)	42.1
WC-4	1093	7.3(50)	35.0(241)	53.5
WCR-93		6.5(45)	40.1(276)	57.6
WD-1		10.6(73)	36.8(254)	55.3
WE-3		7.9(54)	34.9(241)	51.5
WER-94			35.0(241)	56.0
WC-4	1370	5.2(36)	24.4(168)	50.4
WCR-93		5.0(34)	26.3(181)	54.2
WD-1		7.8(54)	23.8(164)	56.0
WE-3		7.8-7.0(54-48)	25.5(169)	55.7-56.0
WER-94		6.1(42)	24.3(168)	53.4

tensile values despite differences in recrystallization temperature (e.g., compare WC, WCR, and WD with WE and WER) or differences in rolling temperature (compare WE-3 with WER-94).

Surface Grinding

Surface grinding was used to accurately reduce the thickness of the 0.33-in.-thick (0.84-mm) disks, cut by EDM, to the final thickness of 0.0255 in. (0.648 mm). The surface grinding operation also produced a smooth surface finish of about 8 to 10 μ in. (0.2-0.25 mm). However, as the WE series was fabricated, another problem with surface flaws developed. Inspection of ground surfaces with the closed-circuit TV system and optical microscopy showed the flaws to be narrow grooves with one end generally deeper than the other. The grooves were 0.005 to 0.010 in. (0.13-0.25 mm) long and up to 0.0015 in. (38 μ m) deep. Since the flaws were always parallel to the grinding direction, they were obviously

produced during grinding. Two remedies were devised to help alleviate the problem: (1) more frequent cleaning of the shroud surrounding the grinding wheel to remove grinding particles and (2) the use of a finer grit aluminum oxide wheel (an A60-H instead of the A46-H wheel). These changes in grinding procedure reduced both the size and number of scratches and tears quite dramatically. Out of the 40 disks produced from WE-40 through 49, 33 had visual notations. (A visual inspection notation was given to a blank if any flaw exceeded a depth of 0.0007 in. or 18 μm .) Adoption of the finer grinding wheel and more stringent cleaning procedures started with WE-50 through 59, 10 out of 40 disks had visual notations, and from WE-60 through 69, only 5 out of 40 disks were noted. A low percentage of surface defects was maintained throughout the remainder of the production program.

PRODUCTION YIELD

The production yield of Ir-0.3% W disks for November 1973 through April 1974 is presented in Table 5. The total number of iridium alloy

Table 5. Production Yield — Ir-0.3% W Disks^a

Month	Number Manufactured	Rejections						Number Shipped
		Ultrasonic		Dye Penetrant		Surface		
		Held	NCR ^b	Held	NCR ^b	Held	NCR ^b	
November 1973	42	4	0	2	0	2	0	34
December 1973	59	9	3	2	2	2	0	46
January 1974	66	3	5	0	1	6	6	57
February 1974	100	13	6	1	1	2	14	84
March 1974	118	11	2	1	3	2	0	104
April 1974	99	9	0	0	0	0	0	90
Totals	484	49	16	6	7	14	20	415
Percent of Total Manufactured		10.1	3.3	1.2	1.4	2.9	4.1	85.7

	<u>Number</u>	<u>Percent of Total Manufactured</u>
^a Gross yield (number manufactured — rejects held)	415	85.7
Number of rejects held at ORNL	69	14.3
Number of rejects shipped with NCR	43	8.9
In-specification yield (number manufactured — all rejects)	372	76.9

^bShipped with nonconformance report.

disks manufactured, the number of disks rejected in nondestructive inspection tests, and the number shipped are shown on a monthly basis. Some disks that did not strictly meet specifications were still shipped to Mound Laboratory for use in special tests, welding setups, etc. These samples were accompanied by nonconformance reports (NCR's) stating which specifications were not met. From a total of 484 disks manufactured, the number shipped to Mound Laboratory was 415, for a gross yield of 85.7%. Forty-three of those shipped (8.9%) had NCR's, so the total in-specification yield was 372 disks or 76.9%. A total of 69 disks or 14.3% were rejected as a result of ultrasonic, penetrant, and surface inspections. Most of the rejections were due to ultrasonic indications of flaws lying beneath and parallel to the disk surfaces. These flaws were probably delaminations in the respective sheets caused by rolling the extremely work-hardenable iridium alloy. Another possible explanation as to their origin points back to the shrinkage cavities in the original ingots. If some of the larger shrinkage cavities were not completely diffusion-bonded together during warm rolling, they could also produce flaws giving ultrasonic indications. In either case, a way of eliminating the flaws was not obvious without making major changes in the fabrication procedure. The number of disks rejected by surface inspection was quite small in November and December (see Table 5), but increased markedly during production for January and February. The increase was associated with the difficulties in warm rolling the early WE-series material, as described previously in the section on Rolling Schedule (see p. 20). After rolling and annealing temperatures were increased by 100°C, the number of surface rejections decreased to low number in March and April. Only 13 rejections of iridium disks were attributed to dye penetrant tests, and these were fairly evenly distributed over the six-month production period. A more detailed listing of the production yield of iridium disks for any one of the months in the table can be found in the respective monthly report.¹²

A total of 687 in.² (0.443 m²) of 0.005-in.-thick (0.13-mm) Ir-0.3% W, foil was shipped to Mound Laboratory during the period from January through April 1974.

SUMMARY

The production of Ir-0.3% W disks for fuel cladding used in the Multi-Hundred Watt (MHW) isotopic heat source has been described. The fabrication of 0.005-in.-thick (0.13-mm) Ir-0.3% W foil for weld backup strips and covers for the MHW was also outlined. A comprehensive quality assurance program was used for the production of both iridium disks and foil. An improved surface cleaning treatment was developed: the iridium material was electrolytically etched in KCN, thoroughly rinsed, and finally soaked in a hot HF + HNO₃ solution. Disks fabricated after WE-40

¹²R. G. Donnelly, *Isotopic Power Materials Development Progress Report for January 1974*, ORNL-TM-4534, p. 2 (on WE-1-9); *ibid. February 1974*, ORNL-TM-4536, p. 4 (WE-11-39); *ibid. March 1974*, ORNL-TM-4609, p. 3 (WE-40-69); *ibid. April 1974*, ORNL-TM-4632, p. 2 (WE-70-WER-96); *ibid. June 1974*, ORNL-TM-4671, p. 1 (WER-97-108).

were rolled at 100°C higher temperature than those used previously after studies showed that the WE material exhibited 100°C higher recrystallization temperatures than earlier lots of iridium. However, the room- and elevated-temperature tensile properties of the WE material were similar to those for earlier batches when all sheets were annealed 1 hr at 1500°C. Improved surface finishes were obtained on Ir-0.3% W disks by more frequent cleaning of the grinding wheel shroud cover and by using a finer grit aluminum oxide grinding wheel. A total of 415 disks were shipped to Mound Laboratory out of a total of 484 manufactured for a gross yield of 85.7%. Most of the 69 disks failing to meet specifications were rejected because of ultrasonic indications of flaws lying beneath and parallel to the disk surfaces. The flaws were probably delaminations caused by the rolling process itself and/or the presence of shrinkage cavities in the original ingot that were never completely diffusion-bonded during subsequent rolling. A total of 687 sq. in. (0.443 m²) of 0.005-in.-thick (0.13-mm) foil was also shipped to Mound Laboratory.

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