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Iridium-Alloy Processing Experience in FY 1990

E. K. Ohriner

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FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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IRIDIUM-ALLOY PROCESSING EXPERIENCE IN FY 1990

E. K. Ohriner

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IRIDIUM-ALLOY PROCESSING EXPERIENCE IN FY 1990*

E. K. Ohriner

ABSTRACT

Iridium-alloy blanks and foil are produced at the Oak Ridge National Laboratory for use as fuel cladding material in radioisotope thermoelectric generators for space power sources. Until 1984, the material was produced from small, 500-g drop castings. A new process has been developed in which consumable electrodes of about 10 kg are arc melted, extruded, and then rolled to produce the sheet products. The work performed during FY 1990 included the consumable-electrode arc melting of four ingots and the extruding and rolling to sheet of four billets. Significant improvements made in the extruding and arc-melting processes during FY 1989 have been demonstrated to dramatically increase the rate of blank acceptance in nondestructive evaluations. Efforts to improve the rolling practice and to better characterize intermetallic particle distributions in the sheet are also described.

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1. INTRODUCTION

The DOP-26 iridium alloy (Ir-0.3% W-0.006% Th-0.005% Al) was developed by the Oak Ridge National Laboratory (ORNL) for use as fuel cladding in radioisotope thermoelectric generators (RTGs) used for space power.[1] Iridium alloy sheet products have been produced at ORNL for about 20 years. The products are currently produced in the form of DOP-26 alloy blanks and DOP-4 alloy (Ir-0.3% W-0.003% Th-0.005% Al) foil. Flight-quality products were produced from small, 500-g, drop-cast ingots and were used in the manufacture of flight hardware for both the Multi-Hundred Watt and General Purpose Heat Source RTGs. In 1984, development of a new iridium-alloy sheet and foil manufacturing process, the consumable-electrode arc melting and extrusion of large, 10-kg ingots, was begun.[2] Since that time, a total of 11 ingots (both experimental and production) has been processed to blanks.

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This report summarizes the production experience for DOP-26 alloy blanks and DOP-4 alloy foil during FY 1990. The results of experimental rolling trials with sheets of iridium scrap are also included. The most important accomplishments have been the production of blanks and foil and the demonstration of a dramatic decrease in the rate of rejection of blanks for ultrasonic indications, using the improvements in arc-melting and extrusion practices made during FY 1989 (ref. 3). The experience with each of the processing steps of electrode fabrication, consumable-electrode melting, ingot conditioning, extrusion, rolling, and inspection are discussed below.

The processing of all materials described in this report was performed under full configuration control. Approved procedural revisions were obtained whenever necessary for all processing of these materials. The sole exception was the RS5 material that consisted of solid scrap material not suitable for recycling for flight-quality products.

2. ELECTRODE FABRICATION

During the year, fabrication of three electrodes from iridium powder was completed, and fabrication of a fourth electrode was begun. The E2 and E3 electrodes were fabricated from electron-beam-melted buttons produced from E-batch powder. The D2 electrode was fabricated from electron-beam-melted buttons produced from D-batch (41%), B-batch (39%), and C-batch (20%) powders. No changes were made in the procedures for powder processing, electron-beam melting, or arc melting and alloying of buttons. The important technical change made in FY 1989 and implemented for all production this year is the elimination of water washing of the iridium powder. (Water washing remains optional in the powder-preparation procedure in the event that future lots of powder are not as clean.)

An additional electrode (CR4) was fabricated from recycle material in accordance with the established procedure. The recycle material consisted of cleaned, melted, extruded, or rolled iridium alloy from C1, D1, CR3, and BF7 ingots manufactured during the years 1987 through 1989. Two pieces of the recycled iridium material, which appeared different in luster, were chemically analyzed and found to be within specification. Thus, CR4 is recycled material from powder lots B, C, and D.

In addition, chemical analysis by isotopic dissolution was performed on a sample from the top center of the E3 ingot to evaluate the extent of thorium macrosegregation in recycle material used in electrode fabrication. The 72-ppm Th value for this sample indicates that macrosegregation is not significant.

3. CONSUMABLE-ELECTRODE MELTING

Arc melting of four ingots was performed during the year using the MP-22 furnace. All of the melts were made using a 27-mm-diam electrode in a 63-mm-diam mold. Measurements of the spacing between the electrode and the mold flange were made with the electrode at the top, middle, and bottom of its allowed travel. Calculated clearances between the electrode and the mold were, in all cases, maintained above the specified 13-mm minimum (as compared to a clearance of 19 mm for a perfectly straight electrode and an ideally aligned system). The initial evacuated pressure levels were between 2 and 3 mPa; they generally rose during melting to between 20 and 40 mPa. The nominal current of the arc for all melts was 3000 A, and the nominal voltage (dc electrode negative) after establishing the arc was 30 to 31 V. At the end of each melt, the current was decreased to minimize the size of the internal cavity near the top of the melt.

The plots of current, voltage, and electrode position versus time are shown in Figs. 1 through 3 for E2, D2, and E3 electrodes, respectively. (Due to a computer error, no plot was obtained for the CR4 electrode.) The plots are very similar for all three melts. Each plot shows a startup lasting about 90 s, a steady-state melting, and an ending hot-top melting. The voltage is allowed to rise to 34 or 35 V during the first 20 s of the melt in order to establish an estimated initial arc length of 25 to 50 cm to avoid sticking of the electrode to the molten pool. The potentiostat control of the electrode feed rate initially drives the electrode at a relatively high rate of 6 mm/s for the first 60 to 80 s. At this time, the arc length has shortened to decrease the voltage to between 29 and 30 V and is estimated to be less than 6 mm. In each case, a number of short circuits indicated by sharp decreases in voltage are seen. These are indications of molten-metal drops from the electrode bridging to the pool for short periods of time. The electrode-feed controller decreases the feeding rate to about 3.5 mm/s for about 10 s. The lower feeding rate causes the arc length to increase. This completes the startup portion of the melt.

The steady-state portion of each melt is characterized by a current of 3000 to 3100 A and a voltage of 30 to 32 V. The electrode-feed rate is relatively constant and is in the range of 3.9 to 4.2 mm/s for the three plots shown. The final hot-top portion of the melt is begun when about 50 mm of electrode length remains, and the current is manually decreased. The hot-top portion is ended when the the electrode has melted back to the start of the iridium stub, and the power is shut off. The three plots (Figs. 1 through 3) show different hot-top parameters. At present, it is not known which parameters are most

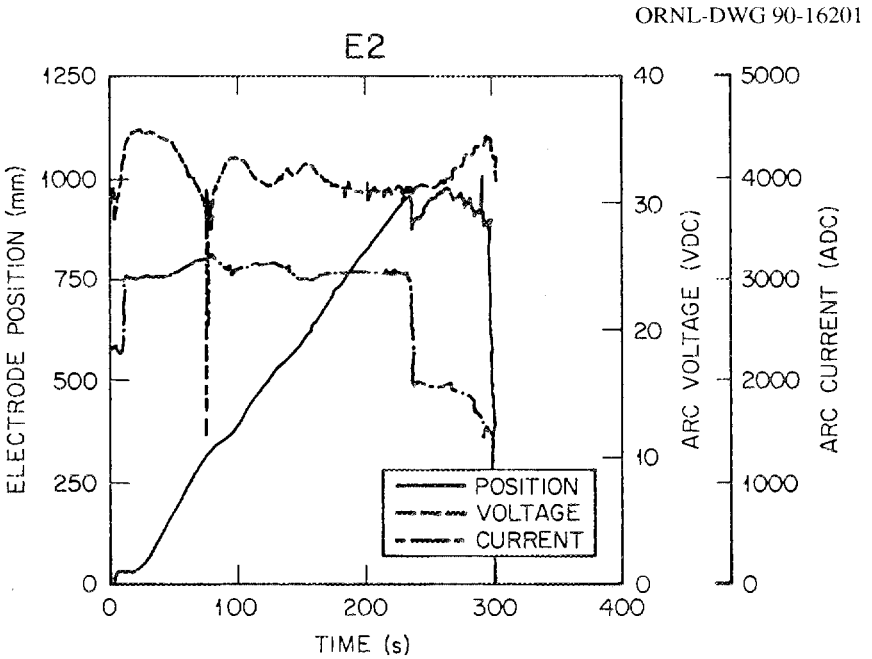

Fig. 1. Plot of arc-melting parameters for the E2 ingot shows maintenance of nominal potential of 30 V and nominal current of 3000 A after the initial 60-s melting.

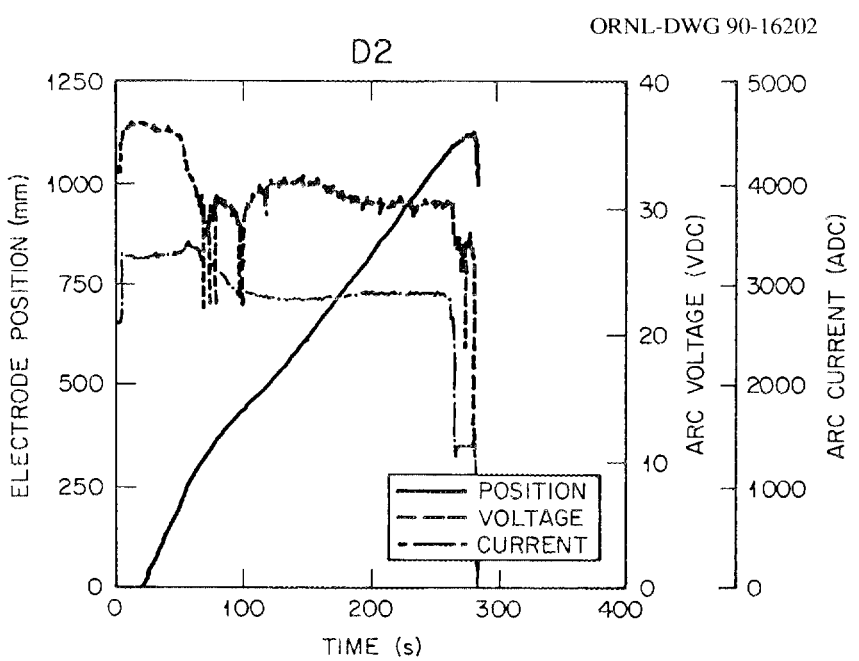

Fig. 2. Arc-melting parameters for the D2 ingot are similar to those seen in Fig. 1 for the E2 ingot.

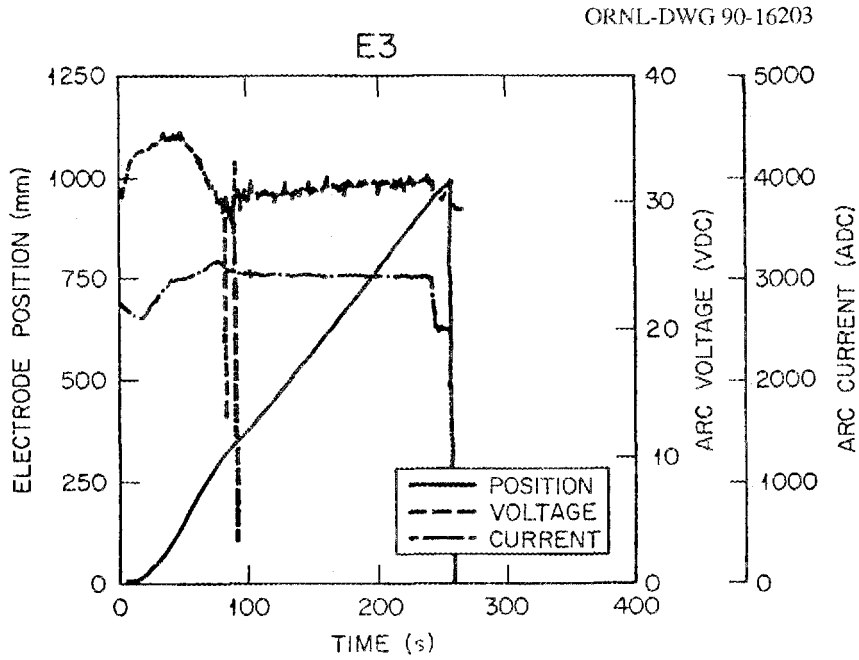

Fig. 3. Arc-melting parameters for the E3 ingot are similar to those seen in Fig. 1 for the E2 ingot.

effective in minimizing the depth of the internal pore, which must be cut from the top of each ingot for use as recycle material.

All four consumable-electrode, arc-melted ingots made during the year show similar appearance. All of the ingots are smooth over 90% or more of the surface. Some areas are rough due to unremelted splatter from the melting. The ingots were all cylindrically ground to remove a minimum of 0.5 mm from the surface, and the circular ends were cut and ground flat and perpendicular to axis. This removed all but a few regions of unremelted splatter.

4. INGOT EXTRUSION

The extrusions of the four arc-melted ingots described above were successfully performed using the same extruding parameters. The ground ingot is placed in a molybdenum can of 86.4-mm diam and heated to 1425°C for 3 h. The canned ingot is then extruded through a zirconia-coated, conical, steel die with a 19- by 51-mm-rectangular opening. The detailed extrusion parameters are listed in Table 1. The press capacity for this diameter of tooling (8.2 MN) has, in the past, resulted in incomplete extrusions.

Table 1. Current extrusion parameters for iridium alloys

Can	
Material:	Molybdenum
Outside diameter:	86.4 ± 0.5 mm
Inside diameter:	Clearance of 0.25 to 0.10 mm to ingot
Base length:	Maximum 1 mm greater than ingot length
Nose:	45 ± 3° cone with a flat diam of 24 ± 1 mm
Tail plug:	Approximately 25-mm thick
Die	
Material:	Extendo-die
Hardness:	47 to 48 HRC
Coating:	0.5 mm ZrO_2 over 0.05 mm nichrome
Outside diameter:	90.22 ± 0.05 mm
Length:	46.2 ± 0.1 mm
Die Opening:	19.05 ± 0.1 mm by 50.8 ± 0.1 mm
Entry flat:	1.6 ± 0.2 mm
Die exit angle:	3 ± 1°
Die exit length:	6.4 ± 0.2 mm
Die throat length:	6.4 ± 0.2 mm
Pinning hole diam:	95.25 ± 0.03 mm
Pinning hole separation:	70.07 ± 0.03 mm
Pinning hole depth:	15.9 ± 0.2 mm
Liner	
Material:	Extendo-die
Hardness:	47 to 48 HRC
Inside diameter:	90.4 ± 0.05 mm
Temperature:	470 ± 25°C
Die backer	
Opening:	22.2 ± 1.0 mm by 54.0 ± 1.0 mm
Pinning holes:	Same as for die
Billet heating	
Temperature:	1425 ± 25°C
Time:	3 to 6 h
Argon flow:	20 ± 5 cfh
Follower block	
Material:	POCO graphite
Diameter:	89 ± 1 mm
Length:	76 ± 3 mm
Preheat temperature:	1300 ± 25°C
Preheat time:	15 ± 5 min
Lubricants	
Billet:	67 wt % No. 567 glass (-200 mesh) 33 wt % No. 3KB glass Manufacturer: Ceramic Color and Chemical Manufacturing Company, New Brighton, Pa.
Liner:	Necrolene (J. F. Hall and Company, Carnegie, Pa.) Fiske 201 (G. Whitfield Richardson Co., Philadelphia, Pa.)
Die:	Same as liner
Transfer time:	25 s maximum
Nitrogen pressure:	15.5 MPa (2350 psi)
Cooling medium:	Vermiculite

Since the implementation of improved extruding parameters in FY 1989 (ref. 1), all extrusions have been successfully completed, although, in some cases, the maximum extruding load approached the press capacity.

The load-versus-time curves for all extrusions made with the improved parameters are shown in Fig. 4. The E2 and CR4 extrusions exhibit loads approaching the press capacity. The curve for CR4 differs from the other curves in that there is no sharp, initial rise in the load, and the load increases continuously for most of the extrusion. A possible explanation is that the difference may be due to the molybdenum can, which was procured from a different vendor and is known to have a different thermomechanical history. Thus, the CR4 can may be the cause of the initially lower flow stress, which would give initially lower load values that increase with work hardening of the molybdenum as the can temperature drops during the extrusion.

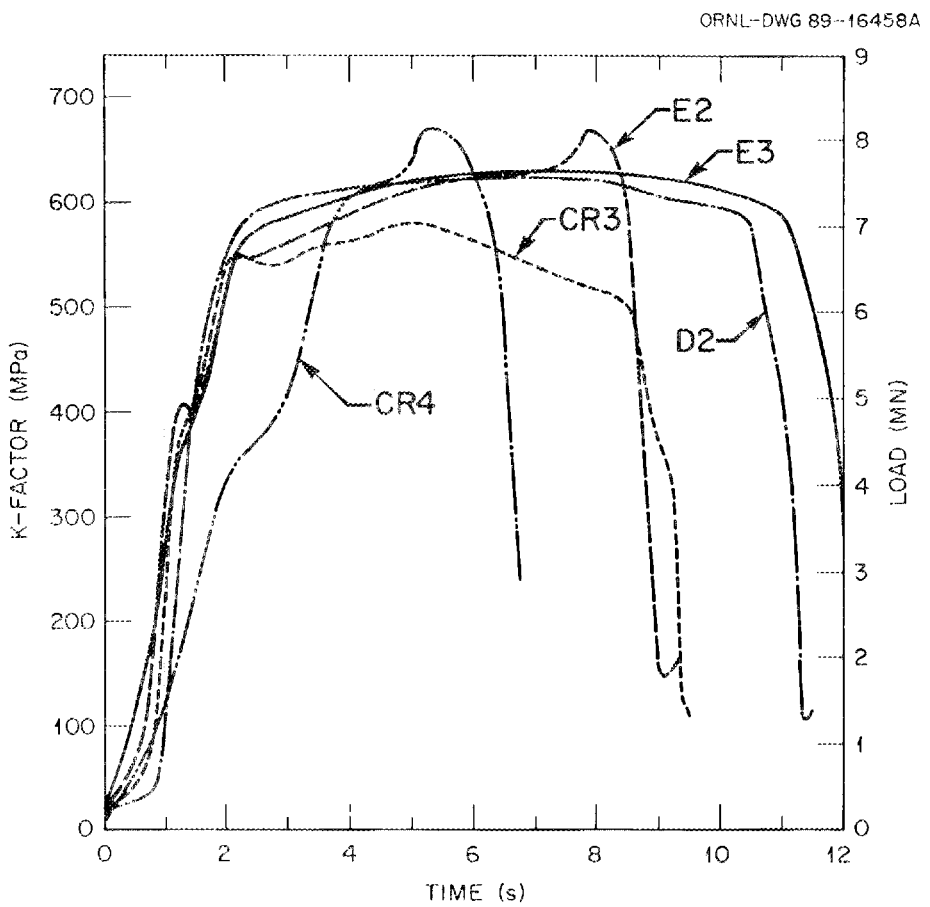

Fig. 4. Plots show that the CR4 extrusion exhibited significantly lower loads early in the extrusion than the D2, E2, and E3 extrusions but also a relatively high load similar to E2 that approached the press capacity of 8.2 MN.

5. SHEET ROLLING

The extruded bars from E2, D2, and E3 ingots were rolled to produce sheet for machining into blanks. In addition, rolling was completed on eight sheets from the CR3 ingot. All rolling was performed using identical parameters. The only technical change made during the year was the addition of a single pass in the longitudinal direction for the first piece of bar from the nose of each extrusion. This change gave a more uniform thickness prior to the normal cross rolling and minimized subsequent variation in sheet width. During the final stages of rolling without molybdenum covers, one of the 17 E2 sheets cracked along nearly its entire length. A total of 112 blanks were machined. Seventeen sheets from the D2 ingot were all successfully rolled and produced 118 blanks. The rolling of 5 of 16 sheets with molybdenum covers from the E3 ingot produced indentations or areas of insufficient thicknesses for production of blanks. Ten blanks were rejected for localized areas of insufficient thickness. The indentations are believed to be due to relative motion between the sheets and their associated covers. Similar behavior (described below) was observed previously on three sheets from the RS5 iridium-scrap ingot.

A study was conducted to identify methods of minimizing the risk of edge cracking or splitting along the rolling direction during the rolling of bare sheet using tungsten-carbide rolls. The cause of the cracking is attributed primarily to the nonuniformity in the sheet thickness following rolling in the molybdenum covers. Measurements performed on the sheets from RS5 iridium-scrap ingot show thicknesses that are maximum at the center and decrease toward both the sides and the ends. After the initial cross rolling of the extrusion from 13 to 8 mm, the variation in thickness is typically 0.4 mm. After further rolling to about 4.5 mm, the variation in thickness is typically 0.2 mm. Upon completion of the covered rolling, the variation in thickness is about 0.15 mm. However, the variation was more on two sheets that slipped within the covers during rolling. In these cases, the iridium-sheet material moved under a portion of the molybdenum cover that had not received as much reduction in thickness and, which in subsequent rolling, left an impression in the sheet.

Several special, tapered covers were prepared and used for rolling at a preheat temperature of 1000°C from an initial thickness of about 2 mm to a final thickness of about 1 mm in 6 or 7 passes of 10% each. Three of the covers were prepared by masking a 25-mm-wide strip along the center of one side with tape and acid etching to remove 0.13, 0.25, and 0.38 mm of material, respectively, to produce a lengthwise step of the same

height. Two of the three covers did produce a depression in the iridium-alloy sheet. For the cover with the 0.25-mm step, the depression varied from 0.06 mm at the nose to nil at the tail. For the cover with 0.38-mm step, the depression varied from 0.06 mm at the nose to 0.02 mm at the tail. The resultant 25-mm-wide depressions were not centered due to initial curvature of the sheets. Three additional covers with a tapered thickness, rather than a step change in thickness, were prepared. The tapers from the thicker center to thinner edge were 0.25, 0.33, and 0.36 mm, respectively. It was hoped that this would produce a rolled iridium-alloy sheet with a more uniform thickness across the width. However, the movement of the sheets within the covers produced several regions up to 0.05-mm thinner than anticipated. The initial curvature of the sheets also contributed to the problem of obtaining greater rolling reduction along the thicker central portion of the sheet.

Additional efforts to improve the use of the molybdenum covers are planned. One simple approach is the placement within the cover of molybdenum strips on both sides and of similar thickness to the iridium-alloy sheet. This is expected to minimize (1) variations in gage thickness across the width of the sheet, (2) warping of the sheet associated with nonuniform reduction in thickness of the molybdenum cover, and (3) tendency of the sheet to move within the cover during rolling.

A total of 0.2 m^2 of foil was produced from the extruded bar of BF7 ingot. The yield of foil in FY 1990 is significantly reduced due to edge cracking. The foil is rolled in molybdenum covers to a thickness of 0.63 to 0.76 mm and recrystallized. The cracking, which begins after the recrystallized sheet is bare rolled to about 20% reduction, becomes less prevalent after about 50% reduction. The efforts described above to improve the uniformity of sheet thickness during covered rolling should also benefit the foil rolling operation.

6. PRODUCT INSPECTION

The inspections for acceptance of blanks per specification GPHS-M-185 consist of dimensional, nondestructive, chemical analysis, and metallographic testing. Three ingots (CR3, E2, and D2) were processed to produce 313 blanks for inspection during this fiscal year. Four blanks were rejected for dimensional nonconformances. These D2 blanks did not clean up during grinding. A total of eight other blanks with dimensional nonconformances were accepted through materials review board action (two CR3s, four E2s, and two D2 blanks).

The nondestructive testing of blanks consists of ultrasonic, dye-penetrant, and visual inspections. Rejection of blanks for ultrasonic indications of defects has averaged 5% during FY 1990. The yield of blanks through ultrasonic inspection is shown graphically in Fig. 5 for the last six years. The consistent high yields during FY 1990 above the goal of 85% are the result of the improved melting practice using the 63-mm-diam ingots combined with ingot surface conditioning and the improved extruding practice. The size and powder-batch source of each ingot are listed in Table 2.

The dye-penetrant and visual inspections of blanks produce few ultimate rejections because reworking by sanding, with subsequent reinspection, permits salvage at relatively low cost. Inspection of 85 blanks from the CR3 ingot resulted in dye-penetrant indications for 4 and visual indications for 12. All but two were acceptable after a single reworking operation. Inspection of 112 blanks from the E2 ingot resulted in no dye-penetrant indications and visual indications for 13, all of which were successfully reworked by sanding. A recent inspection of 116 blanks from the D2 ingot resulted in dye-penetrant indications in 3 and visual indications in 21. Reworking operations on these blanks are

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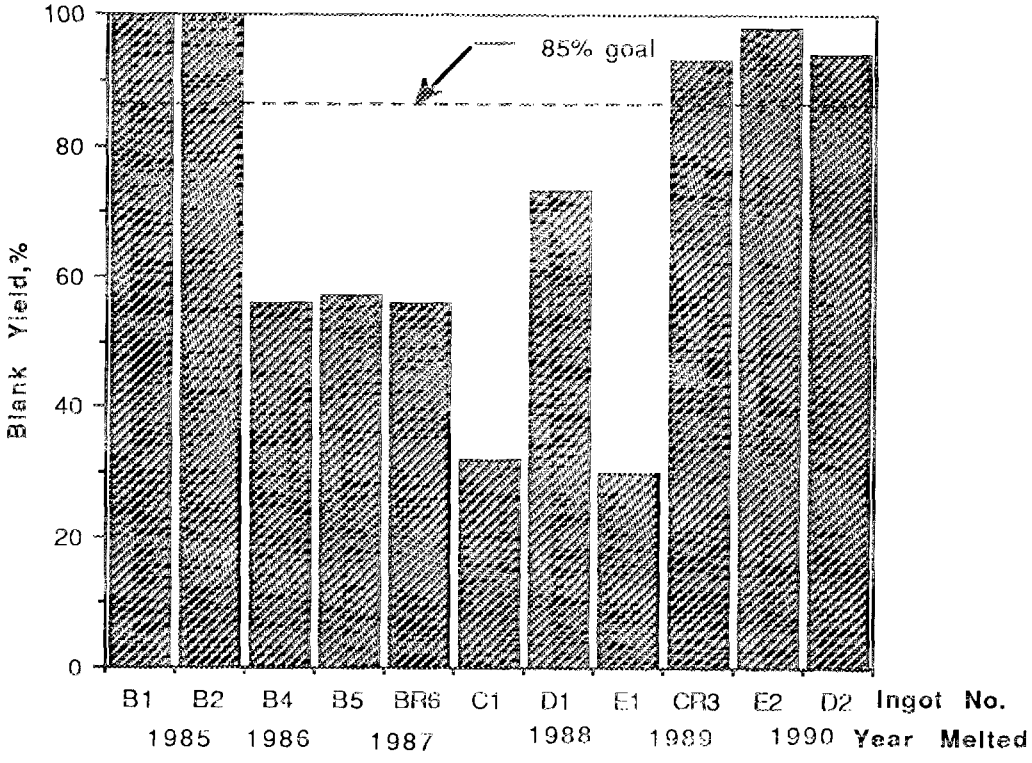

Fig. 5. Yields of blanks through ultrasonic examination have increased due to improved processing in FY 1990 and are above the task goal of 85%.

Table 2. Summary of ingot size and powder-batch source for consumable-electrode, arc-melted ingots

Ingot	Diameter (mm)	Weight (kg)	Powder batch source, %				
			B	C	D	E	Z
B1	51	6.1	100	0	0	0	0
B2	51	6.4	100	0	0	0	0
B4	51	5.6	100	0	0	0	0
B5	51	5.5	100	0	0	0	0
BR6[a]	51	7.6	100	0	0	0	0
C1	51	7.7	0	100	0	0	0
D1	51	7.1	0	0	100	0	0
E1	63	8.5	0	0	0	100	0
CR3[b]	63	8.9	47	25	0	0	28
E2	63	10.9	0	0	0	100	0
D2	63	11.0	39	20	41	0	0

[a]Recycle material obtained from B1, B2, B4, and B5 ingots.

[b]Recycle material obtained from ZR (28%), C1 (25%), BR6 (32%), and B5 (15%) ingots.

not completed. The larger number of visual indications (mostly for inclusions) for the D2 blanks is of concern, however, and is under investigation.

Metallographic inspections of iridium-alloy sheet include porosity, inclusions, degree of recrystallization, delaminations, and microhardness. No porosity or delaminations are observed on any of the sheets examined. All sheets show substantially less than the maximum limit of 5% recrystallization in the fibrous condition. Only one of 47 sheet samples showed any visible inclusions. A single inclusion (about 4-μm wide by 40-μm long) was seen in one sheet from the E2 ingot and was identified as an alumina particle. All sheets show visible particles in the as-polished condition of an iridium-thorium intermetallic that are typically 1 μm in height and 2 to 4 μm in length. The intermetallic particles are larger and more prevalent than in sheet prepared from the 19-mm-rectangular, cross-section drop castings. Sheet prepared from 63-mm-diam ingots generally shows more and larger intermetallic particles than are seen in material from 51-mm-diam ingots. Representative, polished surfaces at a magnification of 200× are shown in Figs. 6 through 8. The distribution of larger intermetallic particles in sheet produced from the larger ingots is also seen in the as-cast materials and may be associated

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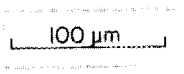

Fig. 6. Optical micrograph of as-polished specimen sheet MR424 produced from a 0.75-mm-square drop casting shows only a few small intermetallic particles.

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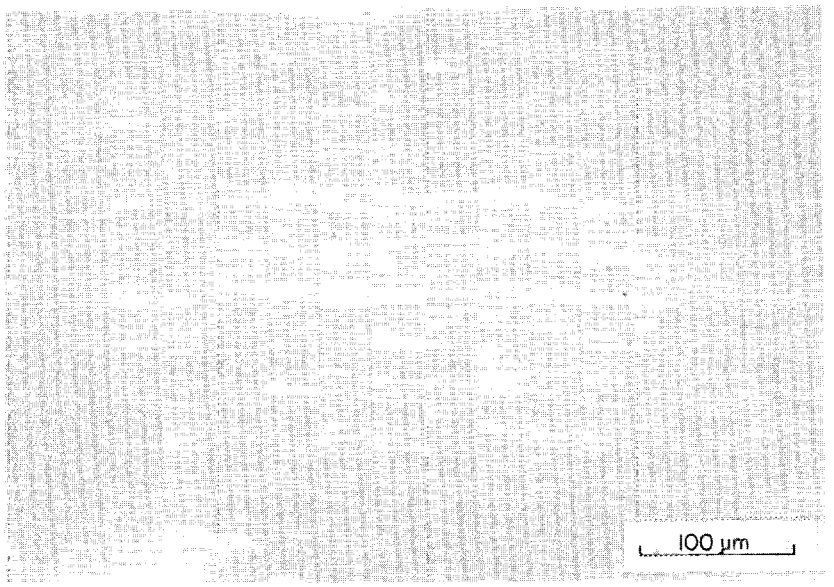

Fig. 7. Optical micrograph of as-polished sheet specimen B2-3 produced from a 51-mm-diam ingot shows more and larger intermetallic particles than seen in sheet produced from drop castings.

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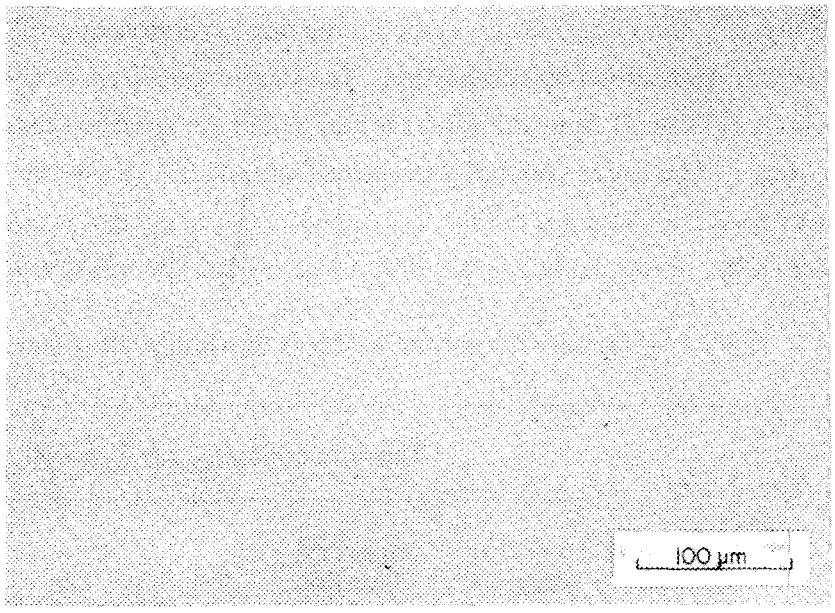

Fig. 8. Optical micrograph of as-polished sheet D2-12 specimen produced from a 63-mm-diam ingot shows intermetallic particles, some of which have been elongated in the rolling direction.

with a decrease in the solidification rate with increased ingot size. The effect of intermetallic-particle distributions and morphology on material properties will be studied in FY 1991.

Microhardness measurements are made on the metallographic sample from each sheet using a Vickers indenter at a load equivalent to 1000 g. The average of five hardness values is obtained from impressions made near the centerline of each sheet. All of the sheets examined show average hardness values within the specified range of 400 to 550 HV. The results are summarized in Table 3.

Table 3. Summary of Vickers microhardness of iridium-alloy sheet

Ingot	Number of sheets	Microhardness of sheets, 1000-g load			
		Mean	Standard deviation	Minimum	Maximum
CR3	14	465	10	450	484
E2	16	446	11	430	463
D2	17	430	6	417	443

Chemical analysis of iridium-alloy sheet includes thorium by isotopic dissolution; aluminum, tungsten, and impurities by spark source mass spectroscopy (SSMS); oxygen by vacuum fusion; and carbon by combustion. The thorium analyses of sheets from CR3, E2, and D2 ingots (Table 4) show the values to fall within the specified range of 30 to 90 ppm by weight. The larger variability in sheets from the CR3 ingot may be associated with the use of recycled materials, but the variation in thorium content is not larger than that of blanks from some ingots made directly from the powder in previous years.

Table 4. Summary of thorium analyses of iridium-alloy sheet

Ingot	Number of sheets	Thorium content, ppm by weight			
		Mean	Standard deviation	Minimum	Maximum
CR3	14	67	6	51	77
E2	16	63	1	61	64
D2	17	69	1	68	71

The analyses for tungsten, aluminum, and impurities by SSMS are performed on every second sheet from each ingot and are within the specified limits for all sheets from all ingots. The impurity levels analyzed ≤ 10 ppm for all sheets from the CR3 ingot with the exception of iron, which was 20 to 30 ppm; molybdenum, which was 20 ppm in one sheet (CR3-2); and sulfur, which was 30 ppm in one sheet (CR3-7). The impurity levels analyzed ≤ 10 ppm in all sheets from the E2 ingot with the exception of 30-ppm copper in the E2-7 sheet. The impurity levels analyzed ≤ 10 ppm for all sheets from the D2 ingot without exception.

The carbon and oxygen analyses of the sheets were all within the specified limits of 35 ppm maximum for carbon and 50 ppm maximum for oxygen, with the exception of one analysis for D2-14 sheet, which is being repeated. The carbon analyses have historically shown a large scatter in values with occasional, nonrepeatable values above the specification limit. Additional work is planned to determine if this variability in carbon analyses from adjacent samples of a sheet is due to inhomogeneity in the sheet or associated with the analysis technique.

7. CONCLUSIONS

The following conclusions are drawn from the experience in processing iridium-alloy-sheet products during FY 1990:

1. The use of the 63-mm-diam ingot in combination with an improved extruding process has dramatically decreased the rate of rejection of finished blanks for ultrasonic indications of defects.
2. The production process has met the program goal of a minimum 85% acceptance rate for finished blanks.
3. An intermetallic compound of iridium and thorium is present in the DOP-26 alloy and is seen as larger size particles in material produced from 63-mm-diam ingot than from 19-mm-square, cross-section drop castings. The particles are somewhat larger for material from 63-mm-diam ingots than for material from 51-mm-diam ingots.
4. Efforts to achieve a more uniform sheet thickness during rolling of iridium-alloy sheet within molybdenum covers, by use of a tapered cross section on the covers, were not successful. Other variations in the design of the covers should be evaluated as a means to increase product yields by minimizing sheet splitting and cracking during bare rolling.

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