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ELECTRON-BEAM PROCESSING OF KILOGRAM QUANTITIES OF IRIDIUM FOR RADIOISOTOPE THERMOELECTRIC GENERATOR APPLICATIONS

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ABSTRACT

Iridium alloys are used as fuel-cladding materials in radioisotope thermoelectric generators (RTGs). Hardware produced at the Oak Ridge National Laboratory (ORNL) has been used in Voyagers 1 and 2, Galileo, and Ulysses spacecraft. An integral part of the production of iridium-sheet metal involves electron-beam (EB) processing. These processes include the degassing of powder-pressed compacts followed by multiple meltings in order to purify 500-g buttons of Ir-0.3% W alloy.

Starting in 1972 and continuing into 1992, our laboratory EB processing was performed (ca. 1970) in a 60-kW (20 kV at 3 A), two-gun system. In 1991, a new 150-kW EB gun facility was installed to complement the older unit. This paper describes how the newly installed system was qualified for production of RTG materials. Ongoing developmental work is discussed that will potentially improve the existing process by utilizing the capabilities of the new EB system.

INTRODUCTION

Radioisotope thermoelectric generators (RTGs) convert the heat of radioactive decay to electrical energy for use in spacecraft. The fuel in the form of 238 plutonium-dioxide pellets is encapsulated in an iridium-alloy cladding. The DOP-26 alloy containing 2000 to 4000 ppm W, 30 to 90 ppm Th, and 20 to 80 ppm Al by weight is produced at the Oak Ridge National Laboratory (ORNL) in the form of 52-mm-diam by 0.64-mm-thick circular blanks. These are formed into cups, pairs of which are welded together, to encapsulate the fuel.

The requirements on the iridium-alloy cladding include high-melting temperature (above 1900°C); high-temperature oxidation resistance; strength and ductility at high temperatures and strain rates; and corrosion resistance, compatibility with graphite and the isotope fuel at high temperature and acceptable formability and weldability. In order to obtain the required properties, the purity of the iridium material must be carefully controlled. Electron-beam (EB) purification serves an important role in the control of alloy impurities.

DISCLAIMER

MANUFACTURE OF DOP-26 ALLOY SHEET METAL BLANKS

The manufacture of DOP-26 alloy sheet for RTG applications is a multistep process that is shown in Fig. 1. Each activity in the process is subject to rigorous procedural controls because of the cost of raw material, application of the end product, and sensitivity of the alloy to compositional variances.

The benefit derived from the small intentional alloy additions of thorium (30 to 90 ppm) and aluminum (20 to 80 ppm) provides a considerable enhancement in impact ductility over the base metal (1-4). This benefit is derived from grain-boundary pinning by second-phase particles of Ir_5Th. Likewise, tungsten additions enhance high-temperature properties through solid-solution strengthening. The thorium segregates strongly to the grain boundaries, which results in increased cohesive strength of the boundaries and a change in fracture made from intergranular (in undoped alloy) to ductile transgranular fracture (in the thorium-doped alloy).

DOP-26 alloy processing begins with iridium and tungsten powders that are made to the impurity limit specifications shown in Tables 1 and 2 by various refiners and vendors. Each container of powder delivered by the refiner is blended, sieved, and analyzed for impurities. The tungsten powder is processed in a hydrogen atmosphere furnace at 1000°C for 1 h to remove oxides. These components are weighed in proper proportions and mechanically blended for 4 h to homogenize the powder mixture of Ir-0.3% W; Fig. 2(*a*) shows the appearance of the iridium powder before blending with tungsten. After blending with tungsten, the mixture is pressed into compacts measuring 5 × 25 × 45 mm at a die pressure of 355 MPa. After pressing, each compact is sintered in a dry-hydrogen furnace for 1 h at 1000°C. A photograph of several compacts made in this manner is shown in Fig. 2(*b*). Each compact weighs approximately 80 g and is about 66% of theoretical density. Final operations inscribe each compact with the alloy batch number followed by final outgassing in a vacuum furnace, which holds the compacts at 1500°C for 4 h. Accountability and traceability procedures are initiated at the powder stage of processing so that each finished part is traceable to the lots of precursor powder.

These Ir-0.3% W compacts are the feedstock for the EB furnace, which is used to consolidate, homogenize, and purify multiple compacts into 560-g buttons of which one is shown in Fig. 2(*c*). A more detailed discussion of the EB process to produce the buttons will be given in later sections of this paper.

The nominal composition of the alloy after EB melting is Ir-0.3% W. In order to produce the required alloy containing 20 to 80 ppm Al and 30 to 90 ppm Th, 100-g quantities of the EB-melted buttons are converted to master alloy. Separate master alloy batches are made containing Ir-0.3% W-2.0% Th and Ir-0.3% W-2.0% Al. Appropriate amounts of the master alloys are melted with the Ir-0.3% W materials in lots of 1000 to 1400 g using a tungsten-electrode-melting arc furnace operating in a 50 kPa atmosphere of argon. Each DOP-26 alloy button is remelted six times in order to fully homogenize the material. Following the sixth melt, the buttons are sequentially melted and drop cast to form the 1500-g, 27-mm-diam castings shown in Fig. 2(*d*).

Further consolidation of the DOP-26 alloy is accomplished by EB welding nine of the segments described above into a 1060-mm-long electrode shown in Fig. 2(*e*). This electrode is remelted in a vacuum-arc-remelt (VAR) furnace to form a 63.5-mm-diam ingot

Table 1. Impurity limits for iridium powder used to produce DOP-26 alloy

Element	Maximum concentration of each element (μg/g)
Al	40
C	50
Ca	70
Cl	600
Co	20
Cr	50
Fe	100
K	100
Mn	20
Mo	40
Na	100
Ni	20
P	20
Pb	20
Pt	100
Rh	70
S	50
Sb	20
Si	70
W	50
Other	*a*
Ir	Remainder

[a]No single unspecified impurity shall exceed 50 μg/g. Total of Pt, Rh, Pd, Ru, Ag, and Au shall not exceed 200 μg/g. Powder is required to pass through a 150-μm mesh screen.

weighing about 12.5 kg. Such an ingot is shown in Fig. 2(*f*). After trimming, the VAR ingot is ground smooth to a surface rms requirement of 8 to 16 μm on all surfaces using diamond tooling.

At this stage, the DOP-26 alloy billet measures 61.0 mm diam and 165 mm length and weighs approximately 10.5 kg. This finished billet is extruded into sheet bar, which is shown in Fig. 2(*g*). This process first encases the billet in a molybdenum can with a wall thickness of 13 mm. The sheet-bar extrusion-die orifice measures 19.1 mm high by 50.8 mm wide, which gives an approximate reduction ratio of 6.4 to 1. Preheating to

Table 2. Impurity limits for tungsten powder used to produce DOP-26 alloy

Element	Maximum concentration of each element (μg/g)
Al	40
O	500
C	35
Mo	100
Co	20
Mn	20
Ni	20
P	20
Pb	20
Sb	20
Others[a]	50
W	Balance

[a]Hydrogen and nitrogen are excluded from this analysis.

1425°C for 3 h in argon is required in order to impart sufficient plasticity to the composite billet. A typical required extrusion force is 8×10^6 N and requires about 20 s to accomplish.

Following extrusion, the sheet bar is immersed in a solution of HNO_3 H_2SO_4 until all of the molybdenum is removed. The resulting sheet bar is then cut into 1200-g sections that are to be rolled. Prior to rolling, each section is etched electrochemically in a saturated KCN bath for 30 min.

Rolling of the 13-mm-thick sheet-bar sections to a final thickness of approximately 0.8 mm is a many-steps process and requires multiple stress-relief heat treatments and one recrystallization during the reduction stages. Before rolling, the sections are covered by folding molybdenum sheet over the material followed by crimping the edges. Initial cross-rolling passes (perpendicular to the extruding direction) reduce the bar to the required finish width of approximately 67 mm. The rolling temperature is 1200°C, which allows an approximate reduction of 20% per pass. Once the required width is achieved, the section is recovered with molybdenum sheet and rolled parallel to the extruding direction to approximately 4.5-mm thickness again at 1200°C. This process continues, gradually decreasing the rolling temperature and reduction per pass until final passes can be made without the molybdenum covers. These final passes are performed using tungsten carbide rolls at 900°C without exceeding 10% reduction per pass. The final sheet is shown in Fig. 2(*h*), measures 0.8 mm thick by 460 m long, and is approximately 68 mm wide.

The final processes to produce the DOP-26 alloy blank shown in Fig. 2(*i*) require electrodischarge machining and surface grinding followed by acid cleaning and a final stress relief at 900°C for 1 h. All blanks are subject to nondestructive examination which include ultrasonic, dye-penetrant, and visual inspections. Representative samples are extracted and subjected to metallographic, impurity, elevated-temperature tensile-impact, and weldability tests.

ELECTRON-BEAM-MELTING FURNACE DESCRIPTION

Until recently, our laboratory's EB processing has utilized the (ca. 1970) two 60-kW gun system shown in Fig. 3. This system has three 25-cm diffusion pumps connected to the main chamber. In addition, separate 15-cm diffusion pumps are connected to each gun space upstream of a valve, which provides the capability to pump the guns separately from the main chamber. The hearths for each gun are retractable from the bottom of the main chamber. Each hearth is downstream from a 30-cm valve to provide the capability to manipulate hearth materials without bringing the main chamber up to atmosphere. This system provides base pressures of 7×10^{-4} Pa. The main application of this furnace has been consolidation, purification, and drop casting of small quantities of research alloys in addition to the iridium alloy production previously described.

In 1991, the new furnace shown in Fig. 4 was installed in order to complement the older unit. The new furnace's principal application was to take over the EB consolidation and refining of iridium. In this configuration, the furnace has a single, 35 kV, 150-kW gun. The vacuum chamber measures $1400 \times 700 \times 680$ mm. The melting hearth for iridium alloy materials has six spherical section cavities shown in Fig. 5. Each of the iridium compacts shown measures $5 \times 25 \times 40$ mm. Vacuum equipment includes: 12.0 m^3/s oil diffusion pump, rotary piston pump and roots blower combination, 0.36 m^3/s turbomolecular pump for the gun space, and miscellaneous small mechanical pumps. An auxiliary chamber mounted on the side of the main tank allows retraction of a mold for rotation of the buttons without bringing the main tank to atmosphere. Various features were built into the furnace to allow future additions that include a second gun, a horizontal and vertical ingot feed mechanism, and a drip-melt casting withdrawal system.

NEW ELECTRON-BEAM-MELTING FURNACE QUALIFICATION TESTS

Integration of a new EB furnace to our DOP-26 alloy blank production process may appear to be a trivial undertaking, given the considerable upgrade in capability offered by the more modern furnace. It was, in fact, a rigorous process requiring considerable attention to detail. This situation was an inevitable consequence of a programmatic criterion where the new furnace must unequivocally be shown to not introduce metallurgical differences into the already well established iridium alloy material.

The strategy that was implemented to satisfy this criterion was straightforward. Its first premise was to reproduce the processing conditions in the new furnace that had been well established in the older one. Quantifiable parameters such as beam power, melt duration, pressure conditions, and cooldown duration were all easily reproduced in the new furnace. Other parameters, such as the beam sweep pattern on the work surface and beam power transients, were manually controlled in the old furnace and, therefore, subject to less exact interpretation when mimicked by the newer, numerically controlled furnace.

In order to finally establish that no deleterious effects were introduced into the material by the new furnace operation, a series of tests were devised that incrementally qualified the new furnace for routine production.

In the first series of tests that were designed to verify correct installation and to train operations staff, niobium materials were substituted for the DOP-26 alloy. The niobium test materials were the same geometry as the iridium alloy feed material. A separate melting hearth and separate liner for the furnace vacuum vessel were used to prevent niobium contamination during future operation. Analyses were performed to translate the manual manipulation of the old furnace operations into instructions that could be programmed into the new furnace melt controller. These analyses indicated that the melt profile shown in Fig. 6 was a good representation of the EB power that was incident on the melt surface of the old furnace.

Other differences that had to be taken into account were the geometry of the hearths relative to the incident beams. These differences are shown schematically in Fig. 7. In the old furnace, the button positions were located in such a way that the incident angle, which the beam makes with the normal-to-the-button surface, was a constant 2° over the five button positions. In the new furnace, this incident angle varies from 7.5 to almost 39° as shown in Fig. 7. Also shown are the backscatter electron fractions resulting from the various impact geometries (5).

In order to reproduce the constant absorbed power achieved in the old furnace, compensation in the new furnace was achieved by programming the beam power as it traversed to different positions. The compensated incident power settings that were necessary to duplicate the power absorption in the old furnace are also shown in Fig. 7. These settings were verified by melting niobium and timing the intervals required to raise a 500-g button of the material from ambient temperature to incipient melting. Using the power settings shown in Fig. 7, repeatable time intervals of ±1% were achieved in these melt tests.

Following these tests with niobium, a series of qualifying melts were started using DOP-26 alloy. In the first melt, 504 g of scrap were melted in each of the six hearth positions. These melts verified that the programs developed in the earlier tests with niobium were adequate to melt iridium. In the next two melt campaigns, flight-quality production materials were used. Qualification of the new furnace would be based on the results of the standard tests that are done in all production processing as shown in Fig. 1. For example, analytical chemistry checks are made on the initial powder batches and on the finished blanks. In order to reduce the consequences of having overlooked some aspect of integrating the new furnace into our production cycle, an additional chemical analysis would be performed on each 500-g button after EB melting. In this manner, any unknown source of contamination would be detected before the 3-kg of qualifying buttons processed in the new furnace were VAR melted with an additional 11 kg that were processed in the old EB furnace.

CHEMICAL ANALYSIS RESULTS OF NEW FURNACE-MELTED BUTTONS

Following the first melt in the new furnace, samples were taken from the resulting button and submitted for chemical analysis. The results of the analyses, shown in

Table 3, indicate that impurity levels were reduced from the precursor powder and well within the specifications for blanks and foil. The chemical analysis done on this first series of compacts (1 through 7) confirmed that the EB-melting furnace was operating as expected.

Table 3. Element concentration levels in qualifying iridium-base materials before and after EB melting

Specified elements	Limits for finished blanks[a] (wppm)	Average for iridium powder before melting[b]	Value for first button made from compacts 1 through 7[b] (509 g)	Average for buttons made from compacts 8 through 35[b] (2562 g)
Al	20 to 80	6	3	1
B	50	<50	<1	<1
C	35	<50	13	9
Ca	50	5	1	1
Cr	25	5	1	3
Cu	50	8	3	<1
Fe	150	18	3	4
Mo	50	1	<1	<1
Na	50	18	<1	1
Ni	50	5	<1	<1
O	50	<50	5	5
P	50	1	<1	<1
Rh	150	10	5	11
S	50	9	<1	<3
Si	50	18	10	10
Ta	50	<50	<1	<1
Th	30 to 90	<50	<1	<1
Ti	50	2	1	3
W	2000 to 4000	<50	3100	3050
Zr	50	2	<1	<1
Ag	--	--	--	--
Au	--	--	--	--
Pd	400	68	26	27
Pt	--	--	--	--
Ru	--	--	--	--
Each of others	50	<50	<50	<50

[a]Limits for DOP-26 alloy blanks are shown as a reference. When no range is shown, the single number is the maximum level.

[b]Analysis by spark source mass spectroscopy.

In a second melt campaign, another 28 compacts made from the same batch of powder were loaded into the furnace. Mold positions 2, 3, 4, and 5 were each filled with seven compacts. These mold positions cover the range of electron impact angles inherent in the furnace design (i.e., mold positions 1 and 4, 2 and 5, and 3 and 6 have beam incident angles of 39, 22, and 7.5°, respectively). Chemical analysis was performed on the four buttons resulting from the melting of these 28 compacts. The results indicated reduction for all impurities, and all were less than specified limits.

PREPARATION OF BLANKS FROM NEW FURNACE-PREPARED BUTTONS

Four of the new furnace-prepared buttons, weighing 2.2 kg, were melted in a nonconsumable-electrode arc furnace along with thorium and aluminum master alloys and drop cast. A VAR electrode segment of 1 kg was produced from these buttons. The electrode segment was welded to the end of a production VAR electrode. The entire electrode, which weighed 13.3 kg, was melted. The resulting ingot was ground to produce a billet of 61 mm diam by 158 mm long and weighing 10.4 kg. The billet was placed in a molybdenum can with the bottom of the ingot, containing the new furnace-prepared portion of the electrode produced from the heading buttons, at the tail of the extrusion. Following extrusion, the can material was removed chemically and the extrusion cut into appropriate lengths for rolling. The cut sections designated E5-16 and E5-17, and taken from the tail of the extrusion with a total weight of 1.1 kg, were rolled to sheet, machined into blanks, and cleaned using standard procedures.

Before the furnace can be accepted to perform flight-quality production of DOP-26 alloy, a complete series of tests shown in Fig. 1 for blanks must be performed (i.e., blanks from E5-17 sheet will receive the standard nondestructive examinations). The standard chemical and metallographic analyses performed on test pieces from each sheet show already acceptable results. Tensile impact tests will be performed using standard procedures on two blanks. Weldability testing also shows the expected results. Following successful conclusion of the qualification tests, expected by the end of November 1992, documentation will be submitted to permit routine use of the new furnace for production.

CONCLUSIONS AND FUTURE WORK

The new state-of-the-art EB-melting furnace, which was purchased to replace a 30-year-old furnace, has performed flawlessly during its initial qualifying melts. The first production of DOP-26 alloy produced with this furnace is below all required specifications regarding elemental impurity. Finished blanks were fabricated from this material. This material is currently undergoing additional metallurgical testing in order to finalize the qualification of this furnace. Successful outcome of these tests will allow the use of this furnace in the production of flight-quality blanks for encapsulating isotope heat sources.

Current activities are directed at the installation of a drip-melt system to produce 27.5-mm-diam by 1060-mm-long ingots. An application of this system will be to produce a continuously cast VAR electrode, which in the current process is fabricated by welding together short arc-melted drop-cast segments.

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ELECTRON BEAM PROCESSING OF Kg QUANTITIES OF IRIDIUM FOR RADIOISOTOPE GENERATOR APPLICATIONS*

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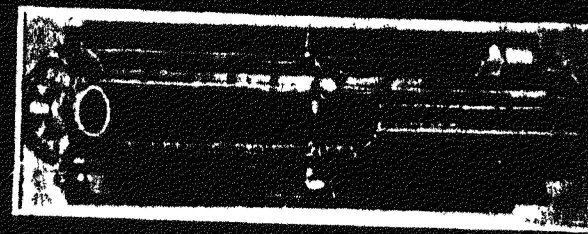
RECENT SUCCESSES

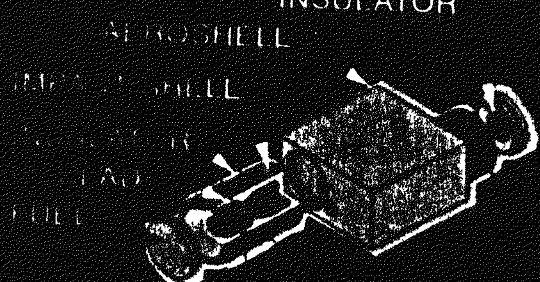
GALILEO (OCTOBER 1989)

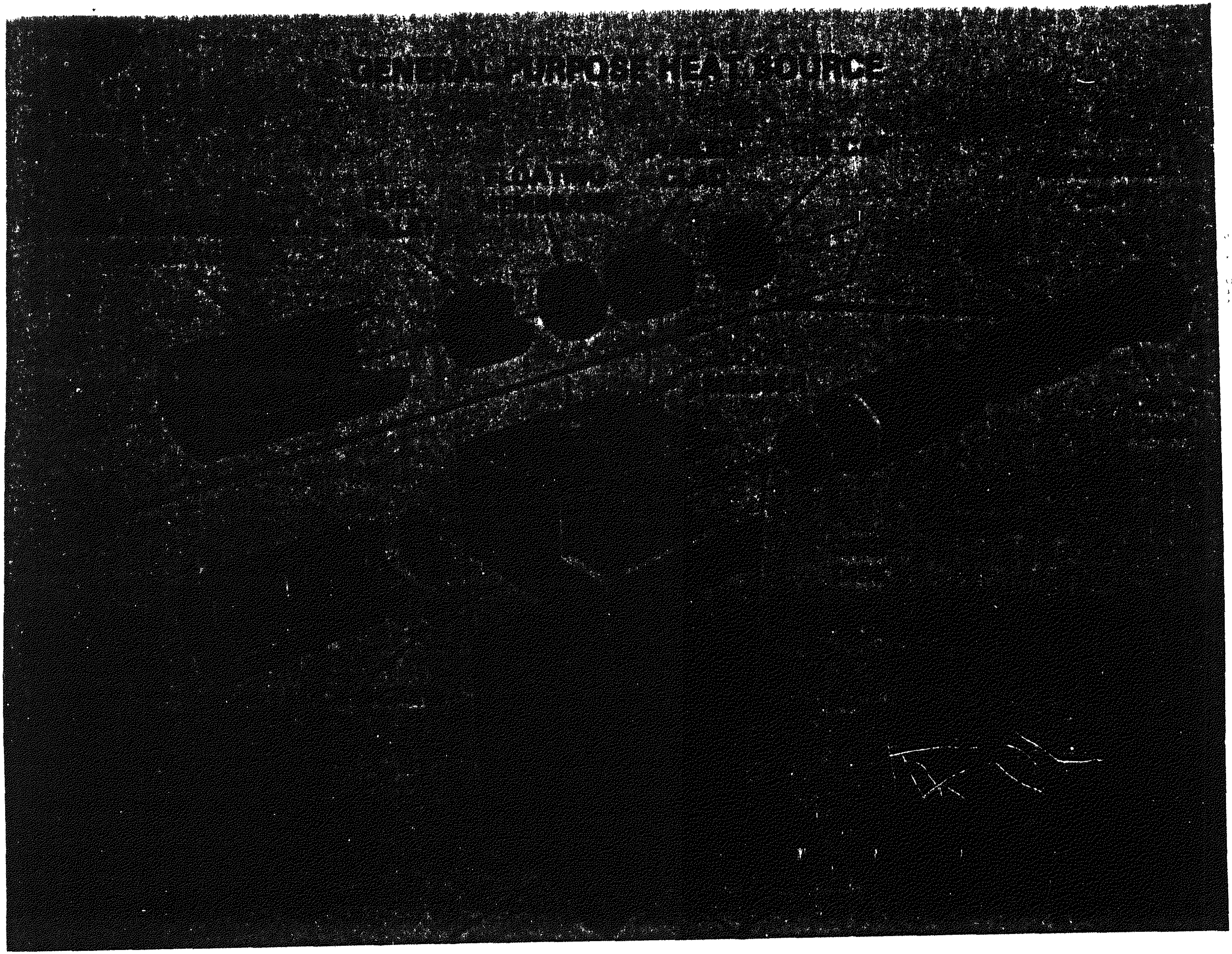
ULYSSES (OCTOBER 1990)

SYSTEM DESIGN

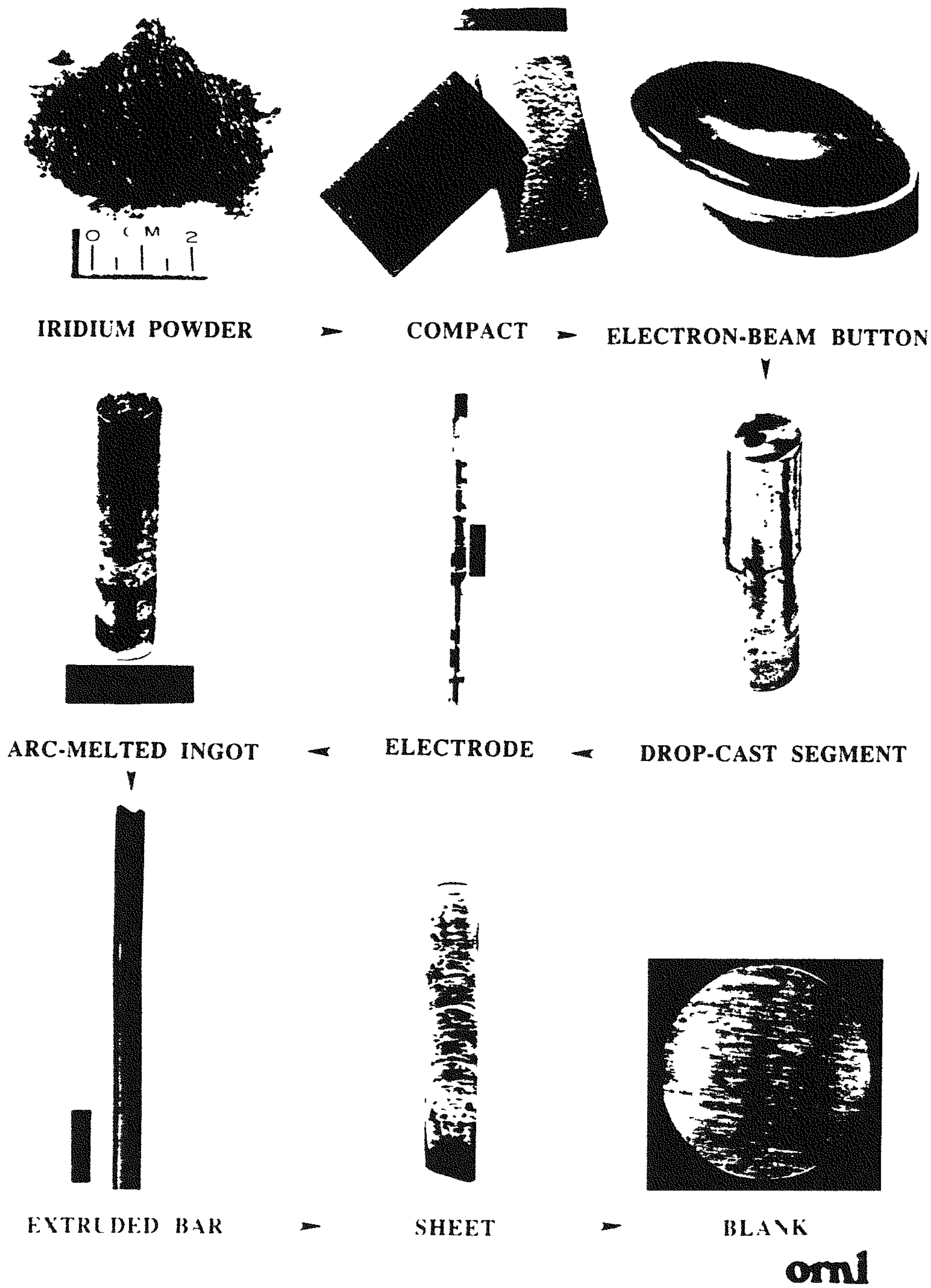


GENERAL PURPOSE HEAT SOURCE MODULE




GENERAL PURPOSE HEAT SOURCE

DOP-26 IRIDIUM ALLOY PROCESSING



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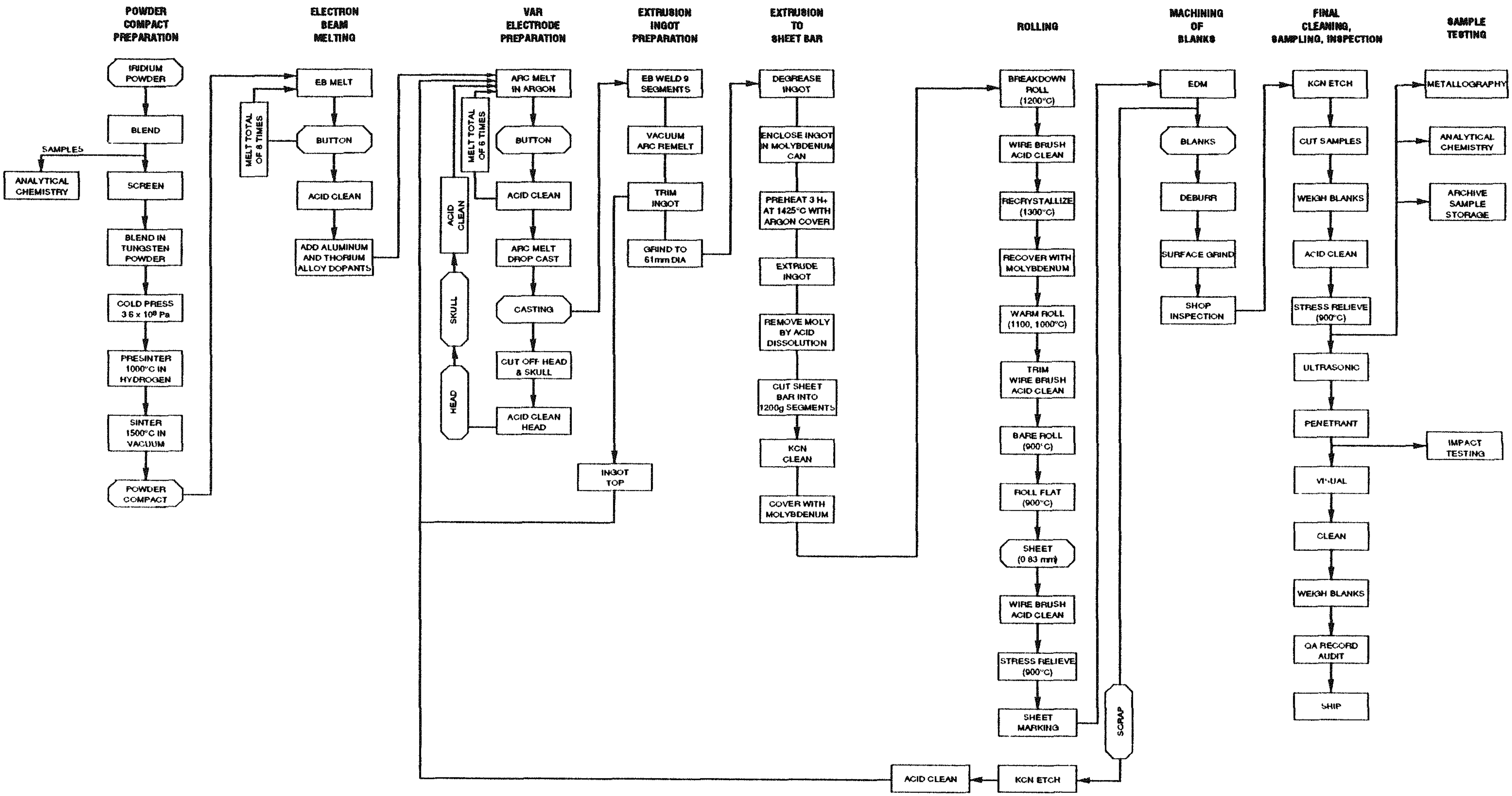
PRODUCTION OF GPHS DOP-26 IRIDIUM ALLOY BLANKS



QUALIFICATION REQUIREMENTS FOR NEW ELECTRON BEAM FURNACE

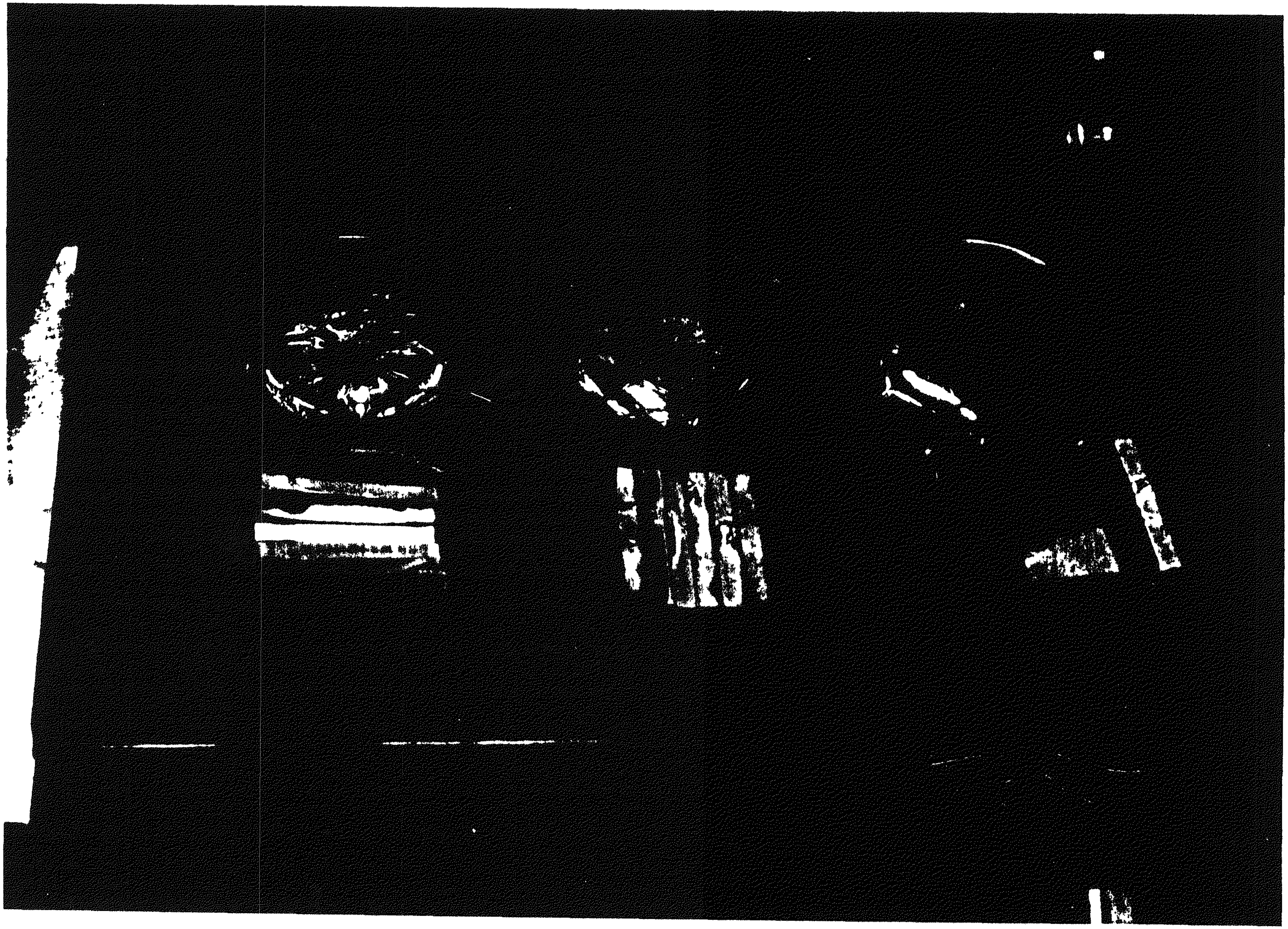
- TEST FURNACE OPERABILITY BY MELTING NIOBIUM BARS TO SIMULATE IRIDIUM POWDER COMPACTS

- INSTALL NEW VAPOR SHIELDS AND MELT ONE IRIDIUM BUTTON USING 560 g OF FLIGHT-QUALITY MATERIAL. VERIFY BY SSMS THAT THE FINISHED MATERIAL MEETS IMPURITY LIMIT SPECIFICATIONS FOR FINISHED BLANKS.

- IF THE FIRST BUTTONS MEET SPECIFICATIONS, MELT FOUR OTHER BUTTONS. VERIFY CHEMISTRY BY SSMS.

QUALIFICATION REQUIREMENTS FOR NEW ELECTRON BEAM FURNACE
(Continued)

- INTRODUCE THIS MATERIAL INTO THE BLANK PRODUCTION CYCLE BY INCORPORATING IT INTO THE LAST SEGMENT OF A REGULAR PRODUCTION VAR ELECTRODE.
- FABRICATE TEST BLANKS AND SUBJECT THESE TO: CHEMICAL AND METALLOGRAPHIC ANALYSES, TENSILE IMPACT TESTS, AND WELDABILITY TESTS.
- DOCUMENT RESULTS FOR CONTROL BOARD REVIEW.



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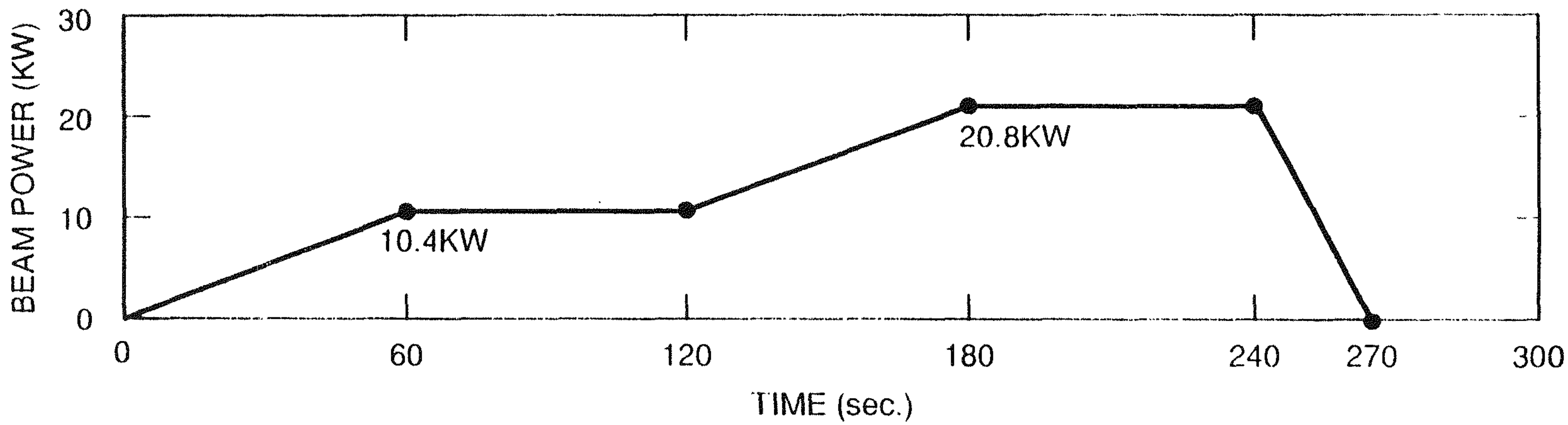
IRIDIUM BUTTON MELT PROFILE



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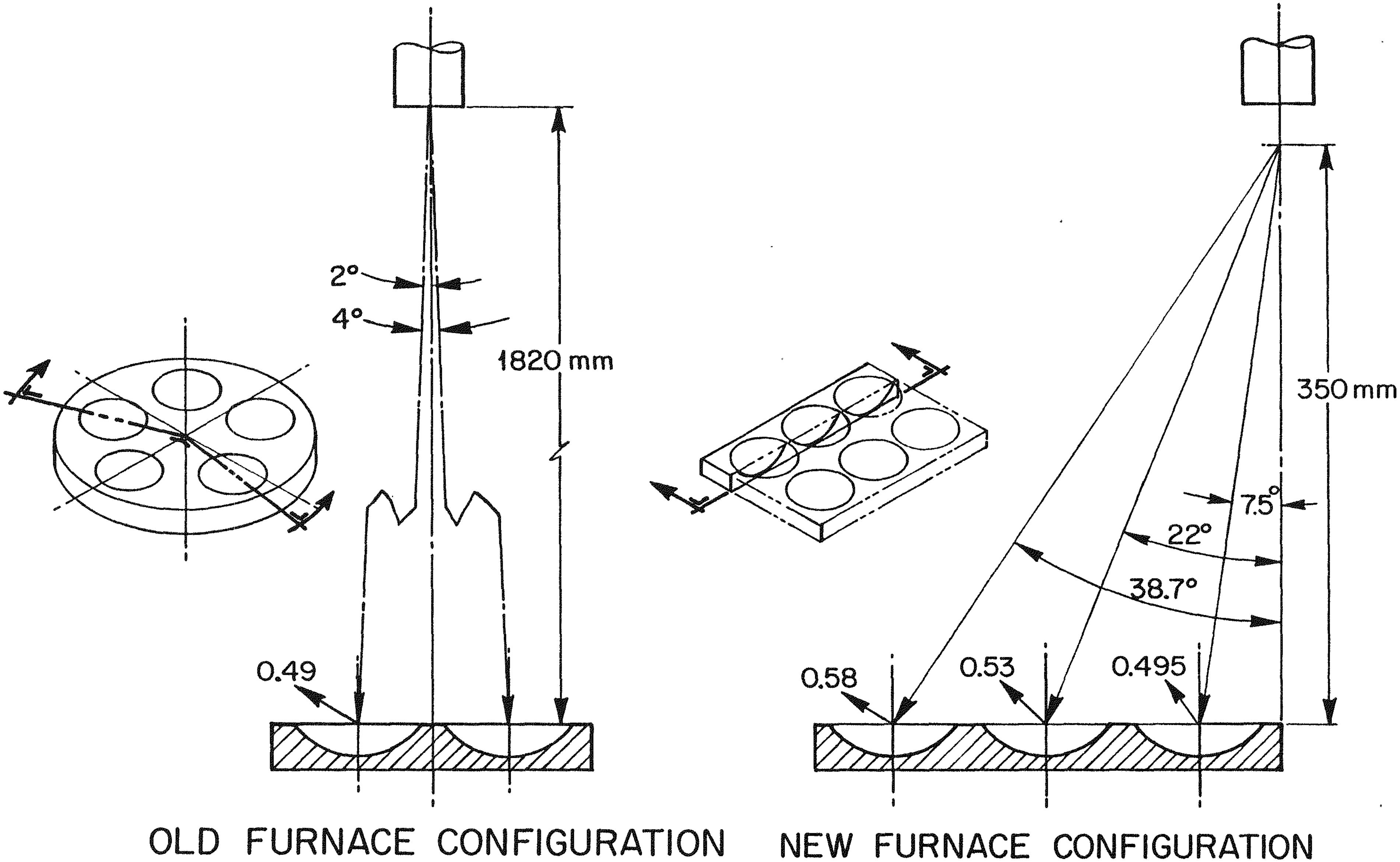
ELECTRON BEAM TRAJECTORY DIFFERENCES BETWEEN NEW AND OLD FURNACES



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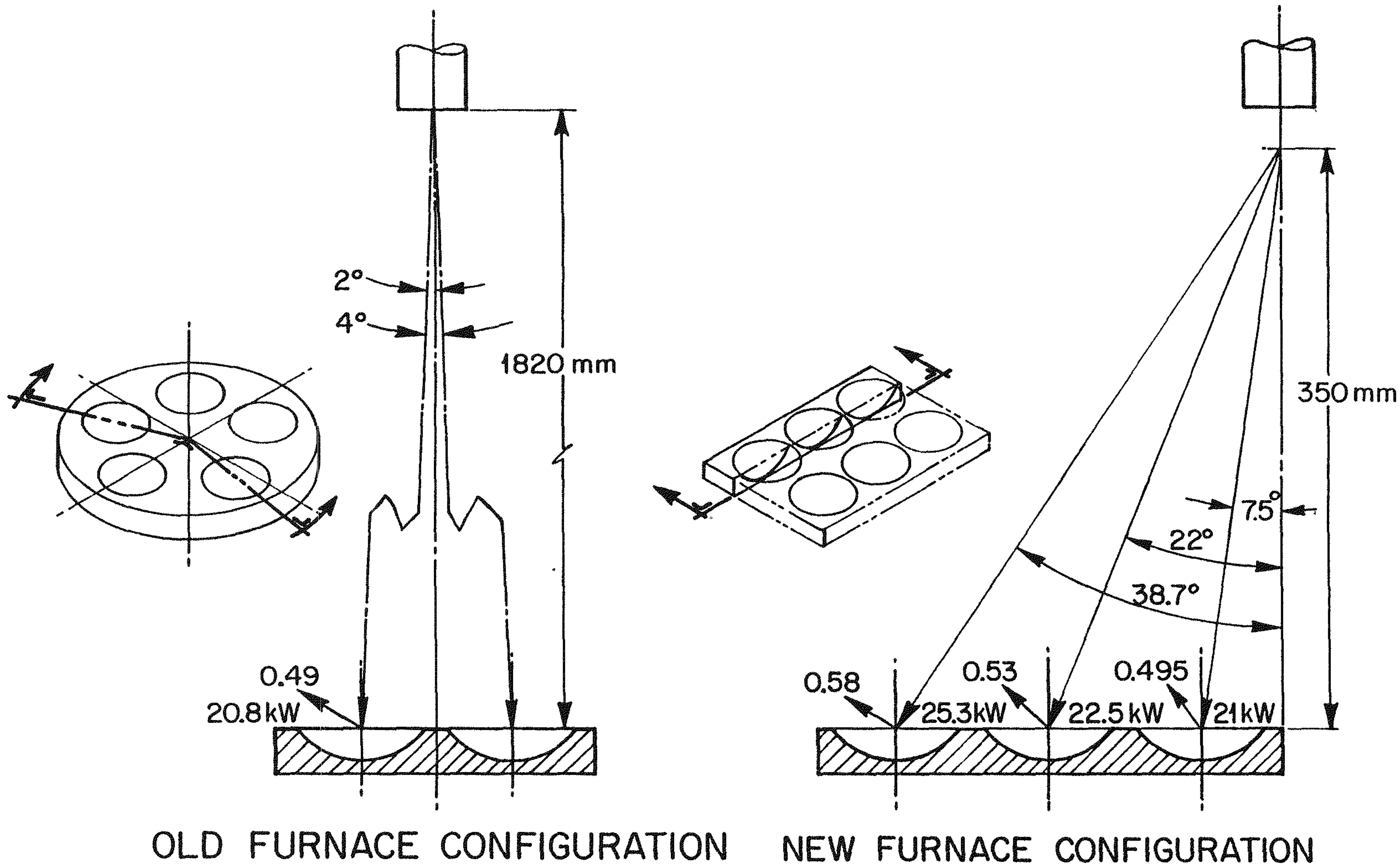
EMISSION POWER IS PROGRAMED TO CHANGE AT DIFFERENT BUTTON POSITIONS TO MATCH THE OLD FURNACE POWER ABSORPTION



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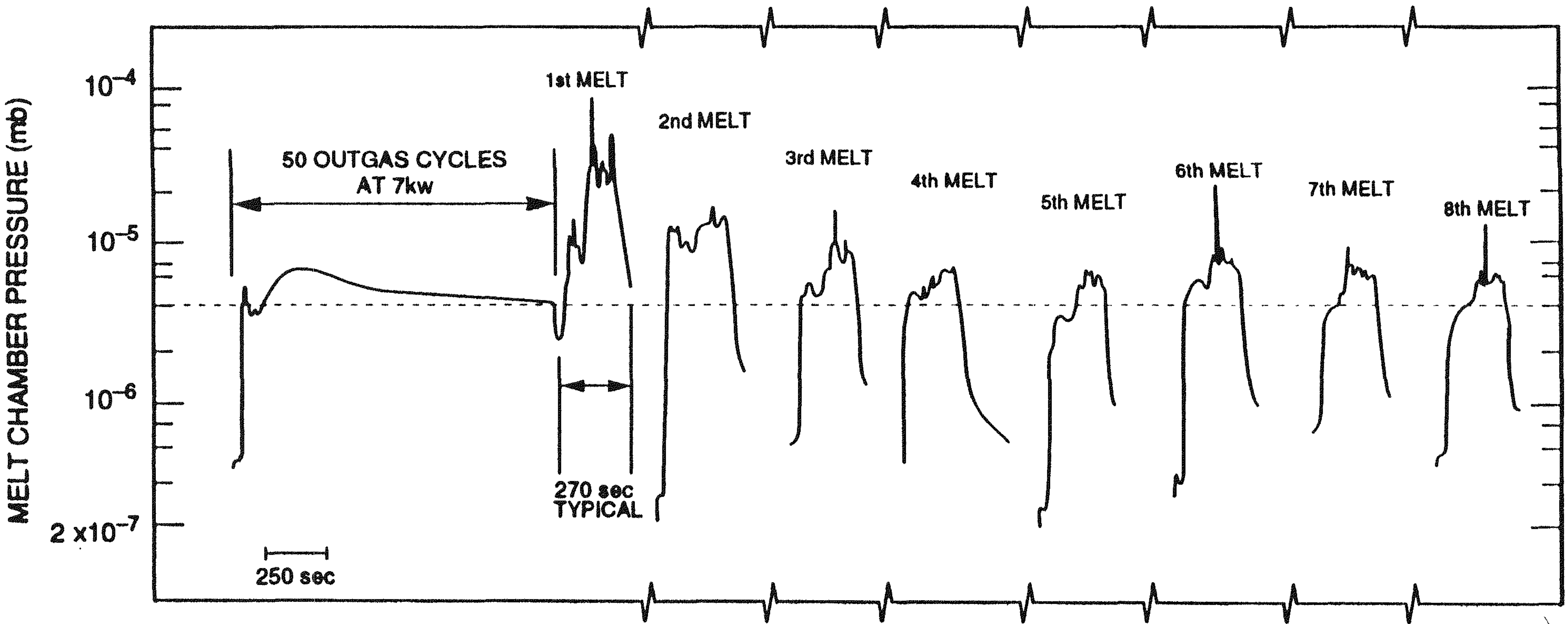
ORNL-DWG 92M-13558R

Melt chamber pressure response to melting of Iridium compacts at 25.2 kw.



IMPURITY LEVELS IN IRIDIUM BUTTONS THAT WERE MELTED DURING QUALIFICATION OF THE NEW FURNACE

Specified elements	Bulk specified limits[a] (wppm)	Average for E-batch iridium powder before melting[b]	Value for melted button made from compacts 1 through 7	Average for melted button made from compacts 8 through 35
Al	20-80	6	1	3
B	50	<50	<1	<1
C	35	<50	13	9
Ca	50	5	1	1
Cr	25	5	1	3
Cu	50	8	3	<1
Fe	150	18	3	4
Mo	50	1	<1	<1
Na	50	18	<1	1
Ni	50	5	<1	<1
O	50	<50	5	5
P	50	1	<1	<1
Rh	150	10	5	11
S	50	9	<1	<3
Si	50	18	10	30
Ta	50	<50	<1	<1
Th	30-90	<50	<1	<1
Ti	50	2	1	3
W	2000-4000	<50	3100	3050
Zr	50	2	<1	<1
Ag, Au, Pd, Pt, Ru	400	68	26	27
Each of others	50	<50	<50	<50

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ELECTRON BEAM FURNACE STATUS AND FUTURE UPGRADES

- BASED ON THE SSMS DATA, THE NEW ELECTRON BEAM FURNACE PRODUCES IRIDIUM ALLOY MATERIAL EQUIVALENT TO THE CURRENT PRODUCTION FURNACE. THE RESULTS OF ADDITIONAL METALLURGICAL TESTS ARE PENDING. POSITIVE OUTCOME WILL QUALIFY THE NEW FURNACE FOR ROUTINE PRODUCTION.

- UPGRADES INCLUDING A CONSOLIDATION TROUGH FOR RECYCLING SCRAP, A BAR FEEDER, AND A 27-MM-DIAM BY 1060 DRIP-MELT CRUCIBLE WILL BE INSTALLED THIS FISCAL YEAR. EXPERIMENTS WILL BE CONDUCTED TO ESTABLISH THE FEASIBILITY OF DRIP CASTING A CONTINUOUS, 1000-MM VACUUM-ARC-REMELT ELECTRODE.