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SUBJECT: Technical Highlights of Space and Terrestrial Systems Programs at Oak Ridge National Laboratory for December 1980

TO: Distribution

FROM: R. H. Cooper

This monthly report is intended to inform the Space and Terrestrial Systems Division, DOE, and their contractors of significant technical highlights of technology and systems support programs in progress at Oak Ridge National Laboratory. A detailed technical presentation of information will be published in topical reports and open literature publications.

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SOLAR-POLAR MISSION MATERIALS SUPPORT

(Activity AE 15 15 20 0, WPAS 02313)

R. L. Heestand

This task provides the fabrication development, characterization, and design verification of materials for the General Purpose Heat Source (GPHS) and Thermoelectric Converter for the 1983 Solar-Polar Space Mission.

Creep Properties of 2219 Aluminum Housing Materials (J. P. Hammond)

The development of creep curves on the 2219 aluminum forging used to retain the RTE generator under elastic loading is continuing. Data for a detailed review of the status will be available for the January 1981 progress report.

FLIGHT SYSTEMS HARDWARE

(Activity AE 15 20 00 0 WPAS 01322)

R. L. Beatty and R. L. Heestand

The objective of this task is to supply Mound Facility with flight quality carbon-bonded carbon-fiber insulators and iridium hardware components for use in the assembly of isotope heat sources. The major iridium activities are associated with fabrication of iridium alloy forming blanks for isotope fuel capsules and the fabrication of iridium foil for vents, decontamination covers, and weld shields. This task also includes management of the precious metal inventory and iridium refining activities for space systems and the coordination of iridium transfers between contractors involved in the General Purpose Heat Source Program.

In addition ORNL is fabricating cylindrical insulator sleeves and end discs from a carbon-bonded carbon-fiber material. Techniques to fabricate this light-weight high-temperature insulating material in the components needed for the General Purpose Heat Source were developed at ORNL in FY-1980.

Iridium Forming Blank Fabrication (R. L. Heestand)

Blanks from "MER" material double electron beam melted "M" batch material and the initial blanks from the "N" batch were ground, inspected, and ready to ship early in the month. However, early this month Mound Facility observed numerous radius cracks in "as-formed" cups from the first group of blanks shipped from ORNL with ground surfaces. At the last heat source meeting held in December it was recommended that ORNL lap the surface of all available blanks before shipment to Mound Facility. As a result the sixty-two blanks from "MER" and "N" batch material which were available for shipment and all "N" batch blanks that had completed the grinding operation were returned to the machine shop for lapping. Because these blanks had been ground to the middle of the blank tolerance, it was judged necessary to lap only one side of the blanks in order to hold the blanks within the required tolerance limits. Ground blanks which had been inspected and rejected due to visual or dye penetrant indications were returned to the shops to be reworked by lapping. The rolling schedules for all sheets in process were modified to allow additional thickness for grinding and lapping. This modification of the rolling schedule will significantly decrease the number of blanks that can be obtained per ingot.

Double electron beam melting wa completed on the "M" batch yielding sufficient material for eighteen ingots. The 3.3 kg of powder received from GSA as a part of an inventory adjustment has been electron beam melted and is designated as the "O" batch and will be used for foil fabrication. This batch of powder was too small for use as a separate batch of ingots for blank fabrication. Electron beam melting of the "P" batch of powder was initiated and is approximately 60% complete. All batches of

powder subsequent to "M" batch have been double electron beam melted with the exception of a small portion of the "N" batch. Selected sheets from this material have been impact tested and found satisfactory.

Revisions and updating of iridium production and quality assurance procedures is underway and are approximately 50% complete. New procedures written for lapping, grinding, and preparation of master alloy are being reviewed for approval.

Iridium Management (W. O. Graves and R. L. Heestand)

Contractors were contacted to determine inventories of iridium on hand and the availability of material which could be released for refining. Visits were made to DOE to discuss analysis of Platina iridium shipments and justification for return of the material and to Engelhard Corp. to witness receipt of material to be refined and assay sampling.

CBCF-3 Insulation Fabrication and Characterization (J M Robbins)

This report period was spent in preparing sleeves and discs for the angle verification test (AVT) and the safety verification test (SVT). Six sets of parts (6 sleeves, 12 discs) were required for the AVT and were supplied from sleeve run C-27 and plate runs P-3-1 and P-8-1. The sleeves from C-27 all revealed small linear indications when examined radiographically. These indications were discussed at the November heat source meeting where it was agreed that these sleeves were usable for the AVT. The sleeves for the AVT were shipped on December 11 and the matching discs were shipped December 19, 1980.

The SVT required 16 sets and were supplied from sleeve runs C-29, C-33, and C-34 along with discs from plate run P-8-1. These parts were shipped on December 23, 1980.

Table 1 summarizes the current production and shipment status for CBCF insulation.

Table 1

Run No.	Status
C-27	Shipped for AVT - 6 sleeves
C-28	Waiting machining - 9 sleeves
C-29	Shipped for SVT - 7 sleeves
C-30	Waiting machining - 6 sleeves
C-31	Waiting machining - 9 sleeves
C-32	In machine shop - 5 sleeves
C-32	Waiting machining - 2 sleeves
C-33	Shipped for SVT - 4 sleeves
C-34	Shipped for SVT - 5 discs
P-8-1	Shipped for SVT - 32 discs
P-3-1	Shipped for SVT - 8 discs

MATERIALS TECHNOLOGY SUPPORT

(Activity AE 15 35 00 0, WPAS 01495)

C. T. Liu

The primary objective of this task is to characterize and improve the metallurgical and mechanical properties of noble base alloys, mainly DOP-26 (Ir-0.3% W doped with 60 ppm Th and 50 ppm Al), to meet the requirements of cladding material in radioisotope heat resources for the Galileo and Solar-Polar space missions. The current efforts are focused in four areas: (1) to provide continuing technical support of the ORNL iridium production activity; (2) to coordinate and interface ORNL materials technology activities with the needs of LANSL, MF, SRP/L and the systems integration contractor; (3) to determine the effect of impurities and environment on the impact properties of the DOP-26 iridium alloy; and (4) to characterize hot cracking, fusion zone structure, and impact properties of arc welded and laser welded DOP-26 iridium alloy. With formation of the Iridium Task Force, there has been a major shift in emphasis to the first two efforts described above.

Rolling Schedule Optimization (C. T. Liu, H. Inouye, and R. L. Heestand)

The rolling schedule currently in use was developed for undoped iridium, and no significant changes have been introduced to compensate for the change to the DOP-26 composition. In a recent Iridium Task Force meeting it was suggested that modification of the rolling schedule may yield a more desirable structure for cup forming. In particular, it is suspected that the 1400°C recrystallization after rolling approximately 70%, see Fig. 1, does not produce a uniform structure for subsequent rolling. To study this problem the end of an ingot was removed after 70% rolling and the metallographic structure was examined in the as-rolled condition, see Fig. 2, and after 1 h recrystallizations at temperatures of 1400, 1300, and 1150°C. From this information it was found that a mixture of fine and coarse grains was produced at 1400°C, see Fig. 3, indicating insufficient work prior to recrystallization. The remainder of the ingot was cut into two sections for further recrystallization and rolling studies.

Results from this analysis suggest that a more optimized rolling schedule for the DOP-26 alloy would require a greater rolling reduction before the intermediate recrystallization step. A rolling schedule that meets this requirement has been developed, see Fig. 4. One ingot is being rolled by this schedule and will be metallographically evaluated to assess the effect of this rolling schedule on the final sheet microstructure.

It should be noted that both ingots used in the analysis will be recycled and will not result in any significant reduction in the iridium available to meet GPHS mission requirements.

INGOT (0.75 × 0.75 INCH CROSS SECTION)

4 ROLLING PASSES AT 1200°C
70% REDUCTION FROM INGOT

INTERMEDIATE RECRYSTALLIZATION
1 HOUR AT 1400°C

9 ROLLING PASSES AT 1100 to 1150°C
PLUS BARE ROLLING AT 900°C

GAGE THICKNESS

87 to 90% REDUCTION FROM RECRYSTALLIZATION STEP

Fig. 1. Summary of the Current Thermal Mechanical Processing Schedule Used for the DOP-26 Alloy.

200 μm 100X

Fig. 2. Microstructure of As-Rolled DOP-26 Ingot (N-513) After the First Four Rolling Steps (70% Reduction in Thickness) Reveal Nonuniform Distribution of Deformation.

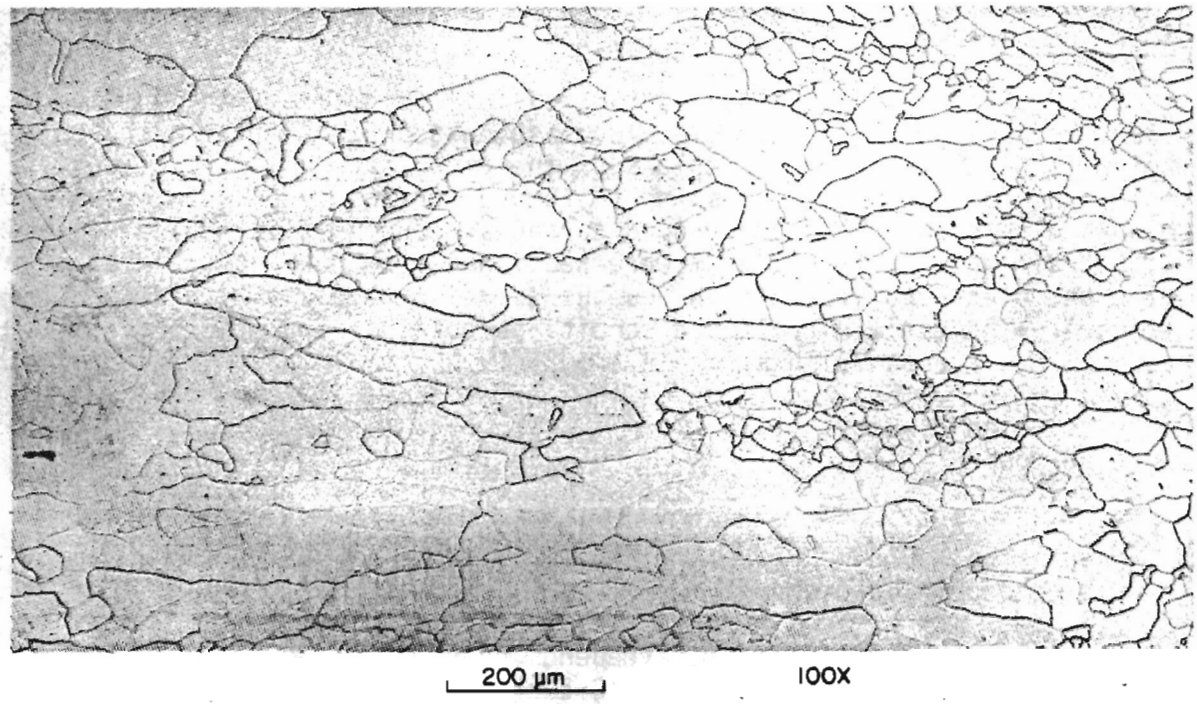


Fig. 3. Microstructure of DOP-26 Alloy Plate (N-513) Rolled to 70% Reduction in Thickness and Recrystallized for 1 h at 1400°C. Note the large variation in grain size.

IMPROVED PROCESS

INGOT (0.75 IN. × 0.75 IN. CROSS SECTION)

6 PASSES AT 1200°C
82% REDUCTION FROM INGOT

INTERMEDIATE REXL.
1 HOUR AT 1400°C

7 PASSES AT 1100°C
PLUS BARE ROLL AT 900°C

GAGE THICKNESS

78 TO 80% REDUCTION RECRYSTALLIZATION STEP

Fig. 4. Summary of the Revised Thermal Mechanical Processing Schedule Being Evaluated on One DOP-26 Ingot.

Improved Surface Preparation (C. T. Liu)

We currently are evaluating the feasibility of preparing iridium blank surfaces by use of an electropolishing technique. The approach being taken by this work is to assess the effectiveness of electropolishing to clean impurities and to remove surface defects on iridium blanks with bare-rolled and ground surfaces. During this period coupons with a dimension of 1 × 1.6 cm were sectioned from a rejected iridium disc and electropolished in an aqueous solution of $CaCl_2$ with 3.5% HCl (conc.). The optimium polished conditions obtained from these initial experiments are as follows:

solution density: 1.35 g/cm^2
AC power
voltage: 25 V
temperature:
solution: 10°C
electrode: iridium

We have found the polishing rate depends on voltage and solution density and is almost independent of AC current level. The average polishing rate of the coupons was about 0.036 mm/1 h at 25 V.

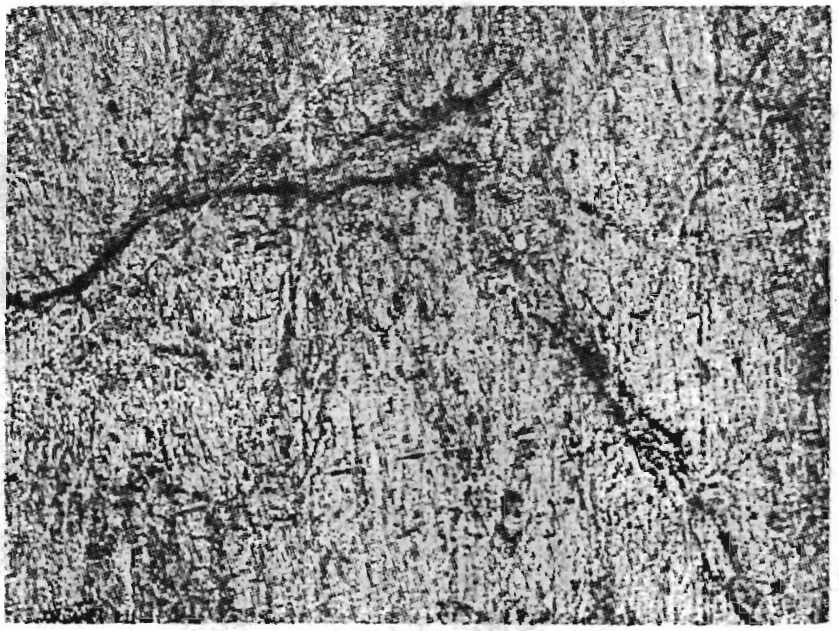
The polished surfaces were examined by optical microscopy, with results shown in Figs. 5 and 6 for bare-rolled and ground surfaces, respectively. Fig. 5(a) shows craze cracks on a rejected bare-rolled blank. A segment of the blank was electropolished for 30 min at 25 V. As shown in Fig. 5(b), a major part of a similar craze crack has been removed through electropolishing. Figure 6 compares the ground surface before and after electropolishing. Fine grinding marks were removed by electropolishing; however, traces of heavy grinding marks are visible in the polished surface after a removal of approximately 0.02 mm (60 min polishing).

The initial results indicate that electropolishing is a promising way to remove surface defects on iridium blanks. Chemical analysis of the polished surface is in progress.

Gas Voids in Ingots (H. Inouye and R. L. Heestand)

A recently sectioned ingot showed large gas voids in addition to a central shrinkage cavity. In addition, small porosity was distributed throughout the ingot. The formation of porosity and gas voids may be caused by a decrease in solubility of a dissolved gas or a decomposition product on freezing. Defects of this type could contribute to ultrasonic defects in the finished sheet and, if minimized, would increase blank yield. To further support the observations a disc containing ultrasonic defects was vacuum annealed 1 h at 2000°C causing the formation of blisters, further suggesting trapped gas or a decomposition product. An attempt to analyze one of the blisters by an auger electron spectroscope equipped with a residual gas analyzer is underway and metallographic sections of another area are being prepared. On the basis of the above

DEFECT IN A BARE ROLLED SURFACE
NO POLISH (200×)



SURFACE AFTER ELECTROPOLISHING
30 MINUTES (NOT THE SAME AREA
AS THE ABOVE PHOTO)

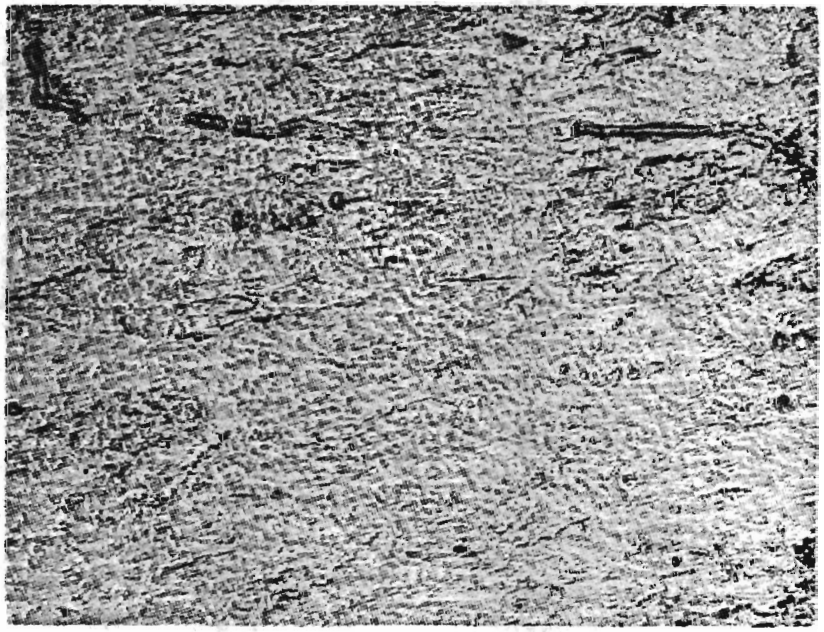
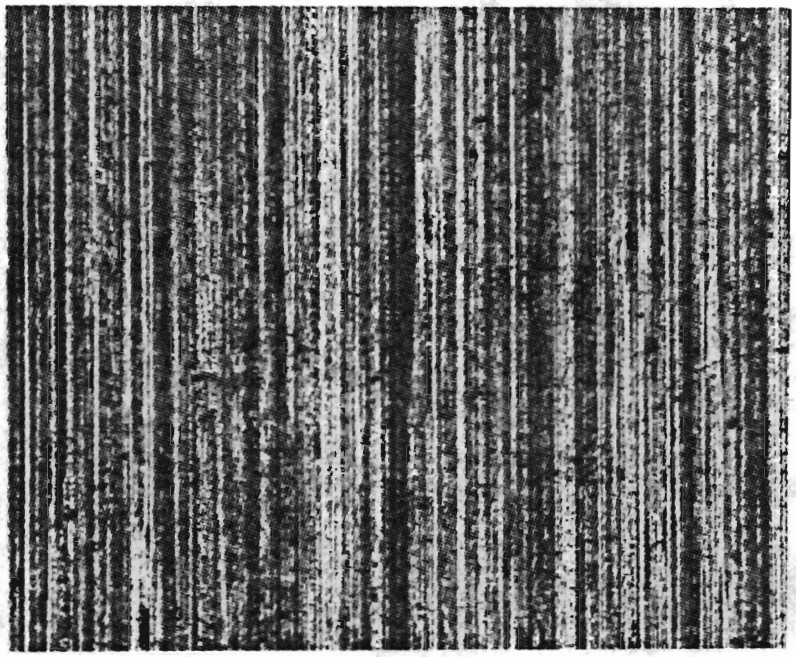


Fig. 5. Comparison of a Bare Rolled Surface Before and After Electropolishing.

GROUND SURFACE NO POLISH
(200×, 6–8 MICRO INCH FINISH)



GROUND SURFACE AFTER
ELECTROPOLISHING FOR 60 MINUTES
(0.6 MIL REMOVED)

Fig. 6. Comparison of a Ground Iridium Surface Before and After Electroplating.

observations it was suspected that oxygen or iridium oxide could be the problem. Conventional metallic deoxidizers dissociate in the presence of iridium making them ineffective. However, hydrogen or carbon may prove effective. To check the above a series of melting experiments using an argon-hydrogen atmosphere, carbon additions in the form of tungsten carbide, and iridium carbon master alloys for addition to the arc melt for deoxidation were devised.

The initial experiment of melting in a static argon-4% hydrogen atmosphere was ineffective in reducing porosity. Melting in a flowing argon-4% hydrogen mixture will be attempted along with the additions of tungsten carbide and the iridium carbon master alloy.

Again it should be noted that the iridium powder used in these experiments contained a high impurity level when received from the vendor and were rejected at ORNL. This material has never been available nor is a candidate for use in fabricating hardware for the GPHS Program.

Ir-0.3 wt % W (Plus C) Powder Compacts for Melting (J. I. Federer)

The purpose of preparing Ir-0.3 wt % W powder compacts with C additions is to determine whether C is effective in degassing the material during melting. The iridium powder used for these compacts contained concentrations of Ag, Cl, K, Na, Pt, Rb, and Rh in excess of the maximum specified amounts. This powder, weighing 3107 g, was divided into four portions, one weighing 1607 g and the other three weighing 500 g each. The larger portion was washed in pure water five times according to standard procedures. The first and fifth wash waters were retained for analysis. The three smaller portions of powder were treated with separate acid solutions. One portion was treated with H_2O-40 vol % HNO_3-10 vol % HF at 60 to 70°C for 3 h with occasional stirring. Afterwards, the acid was retained for analysis. The other two 500 g portions of powder were treated similarly with H_2O-40 vol % HNO_3-10 vol % HCl and H_2O-40 vol % HCl-10 vol % H_2SO_4. Each of these treatments resulted in powder that was within all impurity specifications except for Cl and S.

The treated powders were recombined and blended. Three batches of compacts containing 0.3 wt % W were prepared from the blend of powders. The first batch, designated WC0, was prepared in the usual way and contained only a tungsten powder addition. In the second batch (WC01) the tungsten was added both as tungsten powder and as WC powder, which resulted in a C addition of 0.01 wt %. Only WC was added to the third batch (WC02) resulting in 0.3 wt % W and 0.02 wt % C.

Statistical Analysis of Iridium Process Variables (T. Hebble)

A statistical analysis of process variables associated with L and M batch material is being conducted. As a first step in this analysis combinations of process variables are being evaluated for linear relationships. Process variables that are being evaluated in both L and M batch include impact ductility and impurity content sheet and powder. Additional

L batch process variables that are being evaluated include ingot void volume, dye penetrant rejection frequency, and ultrasonic rejection frequency.

Initial results show that for L batch there is little or no correlation between impact ductility and impurity content. However, for M batch there is a measurable correlation between impact ductility and the respective concentrations of silver, aluminum, silicon, thorium, and tantalum.

Although M batch was found to exhibit a strong correlation between impact ductility and ingot number, (an indication of the order and/or date of melting) this correlation is clearly due to the adoption of double electron beam melting as a method of recovering the impact ductility of the M batch material. No association between impact ductility and ingot number was observed with L batch.

An estimate of the total volume of voids in an ingot has been determined from radiographs of selected L batch ingots and has been compared to other process variables. Although there were no significant correlations between ingot void volume and any individual impurity, the average ingot void volume was found to decrease with the increasing level of total impurity content. It was surprising to find that no apparent relationship was noted between ingot void volume and the frequency of dye penetrant or ultrasonic rejection. It should also be noted that dye penetrant and ultrasonic rejection data did not correlate with any individual impurity concentration or total impurity concentrations.

TERRESTRIAL RADIOISOTOPE APPLICATION DEVELOPMENT

(Activity AE 15 35 00 0, WPAS 01367)

F. N. Case, K. W. Haff, F. J. Schultz, and J. A. Tompkins

Cesium-137 Low Solubility Compounds

Leaching of the sectioned and unsectioned fully loaded ^{137}Cs aluminosilicate pellets is in progress. Some of the results are listed in Table 2.

Table 2. Cesium-137 Aluminosilicate Pellet Cesium Leach Rates

Leach Experiment	Sample I.D.	Pellet No.	Leaching Period Interval (days)	Accumulated Leaching Period (days)	Leach Rate	
					$gcm^{-2}d^{-1}$	$kgm^{-2}s^{-1}$
Soxhlet Extractor (sectioned pellets)	6-22/25	C2	15.79	173.5	5.36×10^{-6}	6.21×10^{-10}
	6-23/25	C2	4.78	178.3	1.33×10^{-5}	1.54×10^{-9}
	7-22/25	C3	15.79	151.7	1.11×10^{-6}	1.28×10^{-10}
	8-24/25	C4	6.77	185.0	9.69×10^{-6}	1.12×10^{-9}
Immersion (unsectioned pellets)	1-34	F1	16.00	136.9	5.18×10^{-8}	5.99×10^{-12}
	1-35	F1	4.99	141.9	3.27×10^{-8}	3.78×10^{-12}
	1-36	F1	7.00	148.9	2.65×10^{-8}	3.07×10^{-12}
	2-20	F2	6.99	31.9	2.39×10^{-8}	2.77×10^{-12}
	2-31	F2	5.03	104.9	1.11×10^{-8}	1.29×10^{-12}
	2-35	F2	5.00	141.9	2.07×10^{-8}	2.40×10^{-12}
	2-36	F2	7.00	148.9	1.57×10^{-8}	1.82×10^{-12}

Reviewing the data presented above and previously reported results one can conclude that the unsectioned, grafoil sheathed aluminosilicate pellets immersed in distilled water have attained their respective steady-state values, while the unsectioned pellets being leached in the Soxhlet extractor have yet to attain steady-state.

The draft of the interim report on pellet characterization studies is complete.

Krypton-85 Light Source Development

A room is being prepared for the various light measurements that will be made during the development effort. This room, approximately 12 ft by 16 ft, is painted black on the walls, ceiling, and floor, and has a track in the form of a 6 ft radius half circle to position the light sensor relative to the light source placed at the center of the half circle.

Delivery of an EG&G light measuring device promised in December has now been extended to late January 1981. In order that work schedules do not slip too far a light measuring device was obtained from another division at ORNL on loan and will be assembled for program use. This will permit us to proceed with phosphor evaluation and with light pipe geometry testing.

Plans for a meeting of the advisory committee at ORNL on January 22, 1981 are in progress.

Methods to recover enriched ^{85}Kr from the existing lights are being tested. While some loss may occur due to the large surface of the phosphor pellets from which the krypton must be removed, we expect >90% recovery will occur. This material will be used to prepare a 30-curie source.

Pressure testing of the quartz capsules used to contain the ^{85}Kr and phosphor has been carried out. The spiral geometry has been tested to 750 psi. The first bulb type failed at 520 psi and a second one at approximately 125 psig. The bulb does not appear to be a promising geometry at this time. One more attempt will be made with a different inner bulb support. However, by all current measurements the spiral geometry is most favorable.

A new light pipe design, tapered to a larger geometry at the top than bottom, has been fabricated. Also, a simple cylinder source holder with white reflective paint has been prepared and will be compared with the existing parabolic reflector and light pipe.

MEETINGS

R. H. Cooper and C. Bieber attended The Iridium Task Force held at Los Alamos National Laboratory December 10—11, 1980.

R. L. Heestand and W. O. Graves attended an Iridium Management Meeting at DOE December 15, 1980.

R. H. Cooper, R. L. Heestand, C. T. Liu, and C. S. Morgan attended a GPHS Heat Source Review Meeting at Savannah River December 16, 1980.

B. L. Tidwell visited Engelhard Corp. to witness weight certification of iridium for refining December 19, 1980.

B. Heshmatpour visited Engelhard Corp. to witness iridium sampling for assays December 22, 1980.

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