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***Mound Laboratory Isotopic
Power Fuels Programs:
January-March 1973***

April 30, 1973

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AEC Research and Development Report

MOUND LABORATORY

Miamisburg, Ohio
operated by

MONSANTO RESEARCH CORPORATION

a subsidiary of Monsanto Company
for the

U. S. ATOMIC ENERGY COMMISSION

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Foreword

This report presents the technological achievements of Mound Laboratory in its isotopic fuels programs during the past three months. The separate reports compiled here represent the work of the following sections of the Nuclear Operations Department (Director W. T. Cave): Heat Sources (Manager D. P. Kelly), and Technology Applications & Development (Manager R. E. Vallée).

This work is supported by the AEC Division of Space Nuclear Systems.

Summaries of all sections are compiled at the front of this report. Brief introductions to the work are included with each section.

Previous reports in this series are:

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MLM-1887	MLM-1983
MLM-1905	MLM-2014

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The Scanning Electron Microscope (SEM) was used to examine cracks on the surface of an iridium hemisphere. This work, done in support of the Multihundred Watt Program, showed that two surface defects were present: (1) thin line cracks and (2) depressions where metal was removed. There was an indication that the cracks extended below the surface in an irregular manner. This was later confirmed by destructive metallography.

This examination also showed that the SEM with some modifications to the sample holder system could possibly be a valuable analytical tool for use in nondestructive analysis of iridium hemisphere hardware.

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The Scanning Electron Microscope (SEM) has been used to examine holes that have been laser drilled through iridium sheet. This examination, done in support of the Multihundred Watt Program, showed that the holes were drilled completely through the 0.020-in. sheet. The drilling process produced iridium puddles on both sides of the holes. Heat-affected zones were observed surrounding each of the holes. The irregular nature of the sidewalls of the holes was confirmed. The examination also showed that a vacuum anneal at 1000°C had little or no effect on the nature of the hole.

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CAPSULE TESTING

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The quadrupole mass spectrometer was used to helium leak check welds on a fueled plutonia heat source. This method of leak testing is more sensitive than the standard helium leak testers presently available at Mound Laboratory. With the appropriate standard helium leaks, it should be capable of testing helium leaks in the 10^{-14} std. cc (He) sec^{-1} range.

Plutonium-238 Fuels

FUEL PREPARATION

Multihundred Watt Sphere Fabrication As part of the Multihundred Watt testing program, seven PPO (pressed plutonium oxide) spheres, SPO-42 through 48, were fabricated during this report period. All of the spheres were vacuum hot pressed at 1480°C and 2500 lb load. The feed was <297 μm , hydroxide-precipitated, $^{238}\text{PuO}_2$ particles that had been oxygen-16 exchanged and fired to 1200°C for 2-4 hr. The spheres SPO-45, -46, -47 and -48 are awaiting further processing. The spheres SPO-42, -43, and -44 were given an oxygen-16 treatment at about 700°C after fabrication to remove any carbon impurity from the surface. They were then processed through calorimetry, neutron counting, gaging, outgassing at 1200°C for 30 min, final weighing, and gaging; and then were transferred to the welding line for encapsulation. However, recent changes in the sphere processing operation call for an outgassing at 1500°C for one hour instead of at 1200°C for 30 min. In addition, the change requires that this 1500°C outgassing occur within two weeks of encapsulation. Thus, these processed spheres will be returned and outgassed at the higher temperature within two weeks of encapsulation. Table 1 lists the data gathered on these spheres to date. (W. D. Pardieck and W. A. Zanutelli)

Table 1

MHW PPO SPHERES - PROPERTY DATA

Sphere	Diameter Range ^a (in. at 20°C)	Weight (g)	Power (W)	Specific Neutron Emission n/sec/g Pu-238
SPO-42	<u>1.456</u> - 1.480	253.9	100.68	7.29×10^3
SPO-43	<u>1.450</u> - 1.466	254.5	100.92	6.82×10^3
SPO-44	1.460-1.474 (<u>1.461</u>)	254.9	101.31	7.30×10^3
SPO-45 ^b	<u>1.456</u> - 1.470	251.6		
SPO-46 ^b	<u>1.458</u> - 1.474	251.8		
SPO-47 ^b	<u>1.451</u> - 1.474	251.9		
SPO-48 ^b				

^a Polar dimension is underlined.

^b Data based on preliminary measurements.

PROCESS STUDIES

Plutonium-238 Dioxide Dissolution Study In the preparation of $^{238}\text{PuO}_2$ particles by the hydroxide method, oxalate-produced $^{238}\text{PuO}_2$ is dissolved in a HF-HNO_3 medium as the primary step of this production process. In the course of this dissolution, small amounts (<1 wt %) of oxalate-produced $^{238}\text{PuO}_2$ often remain undissolved after a normal 48-hr acid treatment. The original intent of this study was to determine the cause and subsequently eliminate this residue. The residue consisted of very large (e.g. 390 μm) refractory-type $^{238}\text{PuO}_2$ particles. Discussion of this residue and photomicrographs illustrating the comparison of the residue to the normally small, oxalate-precipitated $^{238}\text{PuO}_2$ particles were reported earlier.¹

These large residual particles are believed to have resulted from material which had been calcined to higher than normal temperatures, perhaps because of significant thermal gradients within a batch of material during calcination. The apparent lower dissolution rate of this residual material could be due to the lower surface area/mass ratio, to the postulated high calcination temperature, or to both. In either case, the study of this residual material precipitated an extensive study of the entire dissolution process of oxalate-precipitated $^{238}\text{PuO}_2$.

Other investigators^{2,3} have shown that increasing HNO_3 acidity and increasing fluoride ion content generally increases the extent of dissolution. Moseley² has also shown that calcination has a marked effect on dissolution.

The standard production dissolution procedure for 600 g of $^{238}\text{PuO}_2$ entails introducing 120 drops of 48% HF in three to four liters of concentrated HNO_3 (15.8 N). The specification for calcination of the oxalate-precipitated plutonium oxide is to heat to a minimum of 650°C for a minimum of 1 hr. Both of these operations have a relatively wide latitude in areas which could be critical.

Experimental The material used for this study was taken directly from the Mound Laboratory recovery process, as freshly precipitated plutonium oxalate. The isotopic ratio of plutonium was nominally 80 wt % 238; 16 wt % 239; 3 wt % 240; and the remainder was 236, 241, and 242. The maximum metallic impurity content was 1500 ppm. Other individual actinides concentrations were less than 0.5 wt %.

Because of the self-heating characteristics of plutonium-238, 10-g quantities were calcined in a tube furnace to ensure a uniform temperature throughout the sample.

All dissolutions were performed, without auxiliary heating, in a tantalum beaker.

The plutonium solution concentrations were analyzed by alpha counting.

Results The results of the investigation of calcination temperature and HF concentration as a function of dissolution are shown in Figure 1. The effect of calcination temperature is very pronounced. The 800°C calcination curve exhibits a marked resistance to dissolution and even at an HF concentration of 14 mg HF/g $^{238}\text{PuO}_2$ only 27% of the material was dissolved, whereas 73% of the material calcined at 400°C was dissolved under the same conditions.

Also illustrated in the data presented in Figure 1 is the marked effect of HF concentration. The 400°C material, which is the material least hindered by calcination effects, was only 50% dissolved after 3 hr at an HF concentration of 7 mg HF/g $^{238}\text{PuO}_2$; however, the amount dissolved increased to 73% for an HF concentration of 14 mg HF/g $^{238}\text{PuO}_2$.

Figure 2 illustrates the dependence of HNO_3 concentration on dissolution. In this study, the HF concentration as a function of the mass of $^{238}\text{PuO}_2$ was held constant while the normality of the HNO_3 with respect to the mass of $^{238}\text{PuO}_2$ was varied. Very little dissolution occurred below a HNO_3 concentration of 10 N. Above 10 N the amount dissolved increased rapidly with increased HNO_3 normality. Thus, by far, concentrated HNO_3 (16 N) should be used, as is presently the case.

FIGURE 1 - The effect of calcination temperature and HF concentration on PuO_2 dissolution.

FIGURE 2 - The effect of HNO₃ concentration on PuO₂ dissolution; HF concentration held at 10 mg HF/g ²³⁸PuO₂ / 3 cc HNO₃.

However, in marked contrast to the dependence on HNO₃ normality is the independence on the quantity or volume of concentrated HNO₃, as evidenced by the data in Table 2. These results demonstrate that the volume of concentrated HNO₃ can be varied between the investigated limits without dissolution effect provided the HF concentration is held constant as a function of the mass of ²³⁸PuO₂. The calculated HF molarity exhibits a considerable range, while the HF/PuO₂ ratio remains constant. These data show that production quantities (575 g ²³⁸PuO₂) can be dissolved in less than half of the volume presently employed.

Since the results of the calcination temperature dependence suggested the use of lower temperatures, it became necessary to investigate the temperature dependence of oxalate decomposition to ensure complete decomposition to the oxide phase, and thus, eliminate any hazard of CO₂ evolution during further processing. Published thermogravimetric analysis studies⁴ report that no further decomposition occurs above 400°C. We confirmed this in an experiment using a "Savannah River" storage container that had been modified to permit pressure measurement. This container was loaded with 330 grams of ²³⁸PuO₂ that had been calcined at 500°C for 1 hr. The filled container was allowed to reach equilibrium (center temperature was 400°C), then the pressure gage was screwed into place. There was a 7 psi increase within 3 min, then no further increase was observed for the remainder of the 24-hr test period. The initial 7

Table 2

EFFECT OF CONCENTRATED HNO_3
VOLUME ON PLUTONIUM DIOXIDE DISSOLUTION^a

HNO_3 Volume Per Gram PuO_2 (cc)	Amount Dissolved in 3 Hr (%)	HF Concentration (M)
2	55.7	0.25
3.5	55.5	0.142
5	54.3	0.1
7	54.0	0.071

^a Dissolution Conditions: PuO_2 Calcined at 500°C for 1 hr,
10 mg HF/g PuO_2
Temperature: 30°C , 1-g PuO_2 Samples

psi increase was probably due to the trapped gas in the pressure gage subsequently expanding when equilibrium temperature was reached. The absence of a continual pressure increase is indicative of no decomposition.

Conclusions The conclusions drawn here are based on the above data and that reported earlier.^{1,5} The highest dissolution rate is obtained from those particles calcined at the lowest temperatures. However, to ensure complete decomposition of the oxalate and yet maintain a reasonable dissolution rate, a calcination temperature of $500 \pm 50^\circ\text{C}$ is suggested. It must be emphasized that this temperature must be carefully maintained and thermal gradients that exceed this range must be rigidly eliminated.

The HF concentration must be considered as a function of the mass of $^{238}\text{PuO}_2$ to be dissolved. An HF concentration, or better referred to as an HF to $^{238}\text{PuO}_2$ mass ratio, of 10 mg HF/g $^{238}\text{PuO}_2$ is suggested as this ratio yields a reasonable dissolution rate. Ratios greater than 10 mg HF/g $^{238}\text{PuO}_2$ for material calcined at 500°C do not greatly increase the dissolution rate (Figure 2). It is also desirable to maintain the HF concentration at the least amount necessary, yet yielding a reasonable rate, to minimize reaction with and possible impurities from the various reaction vessels, handling equipment, and transfer lines.

As long as the suggested HF to $^{238}\text{PuO}_2$ ratio is maintained and concentrated HNO_3 used, the volume of concentrated HNO_3 can be varied widely from 2 to 7 ml/g PuO_2 without affecting the dissolution rate.

Use of the above conditions should considerably shorten the present 48 hr dissolution time to less than 24 hr and probably to 4-6 hr. In

addition, the lower and more uniformly controlled calcination temperature should eliminate the occurrence of the large residual $^{238}\text{PuO}_2$ particles. (D. E. Etter and W. R. Herald)

Plutonium Recovery From Graphite Dies Graphite dies are used in the vacuum hot pressing of molybdenum-coated $^{238}\text{PuO}_2$ shards to produce the plutonium-molybdenum-cermet (PMC) fuel discs. During the hot pressing operation, the loaded dies are subjected to a temperature of 1650°C and pressures up to 13,500 psi. After pressing and disc removal, the dies contain a recoverable amount of plutonium (~5 g plutonium-238). Surface scraping and direct HF-HNO₃ leaching was found to be of little benefit.

To determine the location and nature of the residual plutonium, a section of the die adjacent to the disc location was examined metallographically. Close examination revealed light, metallic-appearing particles which were not found in the original graphite; otherwise the section appeared normal. Electron microprobe examination of the section positively identified the metallic particles as tantalum. The tantalum is present as a liner during the pressing operation. A scan for molybdenum, tantalum, chlorine, fluorine, plutonium, and oxygen, from the specimen edge to 3 mm depth of the cross section, exhibited only plutonium and oxygen, except for the previously discussed tantalum particles.

This information suggested that the plutonium had diffused into the die as some type of carbide complex.

In order to more accurately reveal the location of plutonium diffusion into the die body, an autoradiographic technique was developed. This technique consisted of simply exposing Polaroid 3000 film by putting it in contact with a cross section of the die in a glove-port trash bag. The microprobe and autoradiographic data indicated that essentially all of the plutonium was contained within 1/2 in. of the surface. By removing and further processing only that section of graphite die within 1/2 in. of the pressing area, the volume of processed graphite was considerably reduced.

With the quantity of graphite now reduced to a workable level, the problem of separating the plutonium-238 from the graphite was addressed. A literature search revealed a report⁶ which showed that HF-HNO₃ leaching was quite successful on pulverized graphite containing plutonium from dies employed for casting plutonium metal. This information suggested that the same technique could be used for recovering the plutonium-238 from plutonium-rich graphite.

Simultaneous removal and pulverizing of the plutonium-238 rich section was accomplished by use of a milling tool adapted to a slow speed drill. The powder was subsequently leached with a 0.1M HF-conc HNO₃ solution at 30°C. After three 4-hr leaches, with filtering between each leach, 90% plutonium removal had been achieved.

This appears to be a satisfactory method for recovering plutonium from graphite dies that have been used for the hot pressing of the PMC fuel form. (D. E. Etter and W. R. Herald)

Ceramic Fixtures Slip casting procedures were developed to fabricate ceramic fixturing for use with Multihundred Watt fuel sphere transfer vessels (Figure 3). The ceramic material used was Taycor 414FH Hydro-cast Al_2O_3 from Charles Taylor and Sons.

Molds were designed and fabricated from brass stock (Figure 4). The Al_2O_3 was found to shrink 20% during processing. Thus, the dimensions of the brass mold were designed accordingly.

The casting procedure consists of the following:

Application of mold release agent (Apiezon grease).

Weighing and mixing of the Al_2O_3 with 13% water.

Pouring the mixture evenly into molds and troweling.

Self-drying overnight.

Removing from the molds and firing in air at 1300°C for 1 hr with a heating rate of 200°C/hr .

Before use, the ceramics are vacuum outgassed at 1500°C at a pressure of less than 10^{-4} torr for 1 hr.

(W. A. Zanutelli, Jr.)

FIGURE 3 - Al_2O_3 liners for Multihundred Watt sphere transfer vessels.

FIGURE 4 - Brass molds used to slip cast ceramic liners.

Capsule Design and Testing

CAPSULE MATERIALS

Scanning Electron Microscopy Examination of Surface Cracks of an Iridium Hemisphere An iridium hemisphere (S/N 854), received from Engelhard Corporation for the Multihundred Watt Program, failed dye penetrant examination and, upon closer visual examination, marks that appeared to be deep scratches were observed on the exterior surface of the hemisphere. Since the hemisphere did not meet Quality Control specifications, it was considered a reject part and was available for examination. Scanning Electron Microscopy (SEM) was chosen as one method for characterizing these suspected "deep scratches."

Since it was not possible to fit the entire hemisphere into the SEM chamber without designing a special sample holder, a sample piece was cut from the hemisphere for examination.

The SEM examination showed that the "deep scratches" were actually cracks and, in most cases, material had been removed from the surfaces. Most of the cracks and material removal occurred after the final machining or grinding operation since the machining marks were aligned on both sides of the crack affected area and no galling was observed.

In general, where material removal had occurred, thin line cracks were also observed in the proximate area. In the case of the areas where material removal had occurred, there was an indication that cracks extended beneath the surface in a jagged manner. This fact was later confirmed by metallographic examination. In other areas, thin line cracks had extended around pieces of iridium and it appeared as if these iridium pieces would eventually fall off of the iridium surface.

Figure 5 shows one of the areas where material had been removed from the surface. The largest diameter of this affected area is approximately 77 μm . Granular cleavage surfaces are present in the depths of the affected area (arrow A). Arrow B shows an undisturbed position of the iridium surface in the middle of the affected area. The machining marks present on this "island" are consistent with those on the parent material indicating that the material around the "island" was removed after final machining or grinding. The sharp edges of the depressed area tend to

FIGURE 5 - Crack on surface of iridium hemisphere.

support this contention. Arrow C indicates the deepest visible portion of the depressed area ($\sim 20 \mu\text{m}$); however, arrow D indicates an area where there is a deeper depression and possible crack continuation below the surface.

Figure 6 shows another cracked area with material removed. In this photograph a thin line crack (arrow A) can be seen running parallel to the larger damaged area. A mixed mode of material fracture is present with cleavage obvious in the upper portion of the damaged area (arrow B). Arrow C indicates the area where the damage extends below the surface of the material. Again, the edges of the depression are clean and sharp indicating that the material was removed after final machining.

Figure 7 shows the continuation of the crack in Figure 6. The thin line crack that has been running parallel to the larger one terminates by turning into the larger crack (arrow A). If the surface is undercut enough by the crack lines of both the thin line and the larger crack, it is possible that the surface area bounded by both would eventually fall off. Cleavage types of fracture surfaces are present in the area where the metal has been removed.

FIGURE 6 - Crack on surface of iridium hemisphere.

FIGURE 7 - Crack on surface of iridium hemisphere.

FIGURE 8 - Crack on surface of iridium hemisphere.

Figure 8 shows a portion of the hemispherical surface that has a thin line crack extending essentially completely around it. The fact that this occurred after final machining is obvious since the machining flow lines are clean and continuous. Arrow A shows that the crack extends below the surface.

Figure 9 shows metal removal in two directions. Most of the other areas where metal had been removed had their longest direction running essentially perpendicular to the machining direction; in this case (arrow A), however, an opposite condition exists. Cracks extending below the surface are present in both depressions. Thin line cracks are also evident, in fact one has essentially propagated completely around one portion of the surface.

This examination, while not identifying the cause of the cracks or metal removal, has indicated the extent of these defects, and has shown that the cracks extend below the surface. It has confirmed the fact that the metal removal occurred after final machining, and that two types of metal fracture (inter- and intragranular) are present in the depression.

In addition this study has demonstrated the usefulness of SEM in the gathering of information concerning metal surfaces for quality acceptance decisions in a nondestructive manner. (R. E. Zielinski)

FIGURE 9 - Crack on surface of iridium hemisphere.

Scanning Electron Microscopy Study of Laser Drilled Holes Since the optical microscope proved to be of limited use in characterizing the holes that are laser drilled in iridium discs for use in the Multi-hundred Watt PICS (post-impact containment sphere) vent system, the scanning electron microscope (SEM) was proposed as the tool to study the laser-drilled holes. Figure 10 dramatically emphasizes the differences of the two techniques. Figure 10A shows a laser-drilled hole as it appears using optical microscopy with optimum magnification and lighting; Figure 10B shows a laser-drilled hole as it appears using the SEM.

For the present study, a sample of as-received ORNL sheet material (0.020 in. thick) was laser drilled. Six holes of various sizes were drilled through the specimen using the same technique as developed for the MHW program. Figure 11 shows the top of the specimen after drilling. The puddling of the removed material is confined to the proximity of the hole. Channeling is also observed in the vicinity of some holes. The cause of this effect is not known and it is possible that these channels were present prior to drilling. The lighter area of Figure 11, evident between holes 1, 2, 4, and 5, is due to a surface stain that appeared after drilling. This stain is quite possibly the result of surface oxidation. Table 3 lists the approximate average effective diameters of the holes as determined using the SEM.

A. Optical micrograph (40X).

B. SEM photograph (650X).

FIGURE 10 - Laser-drilled hole.

FIGURE 11 - Overview of six typical laser-drilled holes in 0.020-in. iridium. Captions point out parameters of interest used in interpreting the rest of the figures.

Table 3

APPROXIMATE HOLE DIAMETERS AS DETERMINED BY SEM

<u>Hole Number</u>	<u>Average Effective Diam. (in.)</u>
1	0.0004
2	0.0006
3	0.0040
4	0.0006
5	0.0025
6	0.0040

Figures 12 and 13 show Hole #1 at higher magnification. Figure 12 was taken using the two-signal-chain method. This method allows for two signals to be presented simultaneously. Thus, Figure 12 illustrates both the topography of the hole area and the depth of the hole. The gray area with the obvious scratch marks (arrow A) is the sample holder. The sidewalls of the hole are not smooth, indicating: 1) a possible multistep drilling process, and/or 2) different solidification rates of the "layers" of the 0.020 in. thick sheet. Also observed in this photograph is a metallic stringer in the effective diameter region. In Figure 13A, an enlargement of the hole diameter area, the topography of the metal stringer is obvious. Figure 13B is a differential photograph of the hole region at approximately the same magnification as Figure 13A. (A differential photograph is one taken using a differential amplifier; it enhances minor deviations in detail.) Thus, we can see that a puddle of iridium is attached to the sidewall of the hole and a thinner iridium finger extends from the puddle. This attachment is located in the approximate middle of the hole depth. Also observed in this photograph is the ledging effect of the drilling operation, indicating the possibility of multiple step drilling.

FIGURE 12 - Hole #1.

A

B

Differential mode used to bring out iridium finger present in hole.

FIGURE 13 - Hole #1.

Figures 14 through 18 show enlargements of the other sample holes. Most of these (except Figure 17) show an irregularly shaped hole. This supports the premise that multiple-step drilling was performed. The fact that the holes are irregular in shape can indicate that the sample was checked and realigned after each step until a complete hole was drilled. The realignment possibly was not perfect and irregularly shaped holes resulted. Hole #5 (Figure 17) was the most concentric hole with uniform puddling.

Figure 19 illustrates the shape of the holes on the beam exit side. These holes are more concentric. Puddling occurs to a lesser degree than on the top of the sample, simply because a thinner layer of metal is being pushed through the hole. Splashing is also evident as minute drops of iridium are on the sample surface away from the puddled areas. Figure 20 is a higher magnification photograph of Hole #2. This photograph indicates that a heat-affected zone (arrow A) occurs adjacent to the hole. Away from the hole, the material is not affected by the operation (arrow B).

FIGURE 14 - Hole #2.

FIGURE 15 - Hole #3.

FIGURE 16 - Hole #4.

FIGURE 17 - Hole #5.

FIGURE 18 - Hole #6.

FIGURE 19 - View of beam-exit side of four of the laser-drilled holes.

FIGURE 20 - Beam-exit side of Hole #2 (upper right hole in Figure 19).

Figures 21 and 22 are higher magnification photographs of Holes #1 and #4, respectively.

At the higher magnifications, in the photographs of the isolated holes, the puddling effect seems excessively high and out of proportion with respect to the parent material. To put this feature into the proper perspective a typical laser-drilled hole was cross sectioned (Figure 23). From this profile view it is observed that the highest portion of the puddle extends no more than 0.001 in. above the surface.

To determine the effect of a vacuum anneal on the integrity of the holes, the sample was annealed in vacuum for 1 hr at 1000°C. Post-anneal examination of the sample was then performed again, using the SEM. Figure 24 shows the appearance of the top surface of the sample after the anneal. Figure 24A shows that surface cleaning occurred and the stained area obvious in Figure 11 has been removed. A very uniform contrast is present in the entire area of interest in Figure 24A. Figure 24B is a photograph of the area of interest using the two-signal-chain method. This photograph shows that the holes have not changed in dimensions and are still open. The light area in each hole again is the sample holder showing through the hole. The most drastic change that occurred during

FIGURE 21 - View of beam-exit side of laser-drilled Hole #1.

FIGURE 22 - View of beam-exit side of laser-drilled Hole #4.

FIGURE 23 - Cross section of laser-drilled hole; puddle height ~1 mil (70X).

A

B

FIGURE 24 - View of beam-entrance side of the laser-drilled holes after vacuum anneal.

annealing was that the iridium stringer in Hole #1 has changed shape. It seems to have folded up on itself and has become more uniform in thickness. Why this occurred is not known. The effective diameter of the holes did not change during the anneal.

Figure 25 shows the bottom of the sample. In this photograph some metallic slivers are observed. One in particular is located on the surface of Hole #1. These slivers are the result of surface bonding of the sample to the liner material of the furnace on which the sample was placed. Figure 26 shows an enlargement of Hole #1. The sliver appears to be bonded to the top of the puddled material and has not collapsed into the hole. Figure 27 shows some of the metal slivers in better detail (black arrows). This photograph also shows the heat-affected zone (white arrows) surrounding Hole #2. These heat-affected zones surround all the holes. Other than the metallic slivers, no other changes were observed on the beam-exit side of the sample.

Since the question was raised regarding the removal of the puddles by mechanical scraping, the sample was scraped with a scalpel blade and then reexamined. This examination showed that the scraping did remove the puddles; however, the scraped-off material is deposited in the holes. Unless a more involved procedure is employed to prevent the deposit of the iridium shavings in the holes, mechanical scraping appears to be a

FIGURE 25 - View of beam-exit side of the laser-drilled holes after vacuum anneal.

FIGURE 26 - View of beam-exit side of Hole #1 after vacuum anneal.

FIGURE 27 - View of the beam-exit side of the laser-drilled holes after vacuum anneal.

detrimental operation. If any of the iridium shavings remain in the holes, the possibility of impeding the gas flow through the holes is present.

In summary, this examination has shown that the laser beam effectively drills holes in 0.020 in. iridium sheet. No matter what the size of the laser-drilled hole, characteristic puddling is present after drilling on both the entrance and exit sides of the sheet material. The sidewalls of the holes are not smooth and in some cases indicate multiple-step drilling. Heat-affected zones surround the drilled area, but these do not appear to deleteriously affect the sample. Vacuum annealing of the drilled specimens at 1000°C has no effect on the size or nature of the holes. (R. E. Zielinski)

CAPSULE TESTING

Method of Ultra-Sensitive Helium Leak Detection Presently, the helium leak testing equipment available at Mound Laboratory cannot accurately check for leaks on the order of 10^{-11} st. cc (He) sec^{-1} or lower. Since future programmatic requirements will require accurate readings of helium leaks of this magnitude, a method had to be devised to perform this operation.

It was possible, using a quadrupole mass spectrometer tuned for helium with a calibrated standard helium leak, to quantitize helium leak rates in these low orders of magnitude. A 6-W (thermal) plutonia heat source was used to determine whether this was an effective means of helium leak detection. This heat source consisted of an outer Hastelloy metal container that had been TIG welded. [Previous helium leak checking of this source indicated a helium leak rate of 1×10^{-10} std. cc (He) sec^{-1} , which was the maximum sensitivity of the equipment which was used.] The source was placed in the sampling system of the quadrupole mass spectrometer and the system was allowed to stabilize in a vacuum environment prior to testing.

Once the system was stabilized, four different leak tests were performed on the heat source and the results of the four runs were consistent, as shown in Table 4.

Table 4

HELIUM LEAK RATE DETERMINATIONS

<u>Run No.</u>	<u>Helium Leak Rate (Std cc of He sec^{-1})</u>
1	2.3×10^{-12}
2	2.16×10^{-12}
3	2.88×10^{-12}
4	2.22×10^{-12}
	<hr/>
	2.4×10^{-12}

The results in Table 4 show that the quadrupole mass spectrometer can be used to determine helium leak rates in a region where presently available standard helium leak testing equipment is unusable. (B. W. Hartzell and R. E. Zielinski)

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