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^{238}Pu FUEL-FORM PROCESSES QUARTERLY REPORT

OCTOBER - DECEMBER 1982

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PREPARED FOR THE U. S. DEPARTMENT OF ENERGY UNDER CONTRACT DE-AC09-76SR00001

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Approved by

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Hydrogen and Ceramic Technology Division

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FOREWORD

This report is one of a series that has been issued quarterly to summarize progress in the Savannah River Laboratory ^{238}Pu Fuel Form Program. This program is supported primarily by the DOE Advanced Nuclear Systems and Projects Division (ANSPD).

Goals of the Savannah River Laboratory (SRL) program include providing technical support for the production of $^{238}\text{PuO}_2$ fuel forms in the Savannah River Plant's (SRP) Plutonium Fuel Form (PuFF) Facility. This part of the program includes:

Demonstration of processes and techniques developed by the Los Alamos National Laboratory (LANL) for production at SRP. Information from the demonstration will provide the technical data for technical standards and operating procedures.

Technical Support to assist plant startup and to ensure continuation of safe and efficient production of high-quality heat-source fuel.

Technical Assistance after startup to accommodate changes in product and product specifications, to assist user agencies in improving product performance, to assist SRP in making process improvements that increase efficiency and product reliability, and to adapt plant facilities for new products.

The above program goals have been met. Technical assistance continues at a reduced level consistent with programmatic decreases. The next report in this series will therefore cover the full year 1983.

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GENERAL-PURPOSE HEAT SOURCE (GPHS) PROCESS SUPPORT

ANALYTICAL STUDIES OF DOP-26 IRIIDIUM ALLOY WELDS

INTRODUCTION AND SUMMARY

Intergranular, underbead cracks occasionally occur in girth welds on DOP-26 iridium alloy clad vent sets (CVS) in the region where the gas tungsten arc is quenched.¹ Weld-quench crack surfaces are characterized by ridge network structures that constitute extended grain boundary cavities. Proposed mechanisms that could cause ridge network formation and weld-quench cracking include gas porosity, liquation at grain boundaries, and high-temperature creep.^{2 3} Ultrasonic nondestructive examination of CVS welds made in the PuFF Facility showed that weld-quench cracking severity varies with each batch of DOP-26 iridium alloy. Batches MER and S resulted in severe weld-quench cracking when welded using a two-pole arc oscillator. However, recent use of a four-pole arc pattern shaper has reduced weld-quench cracking.

A welding test in which multiple weld quenches are made in a single CVS has been shown to give a good assessment of the weld-quench cracking severity of a batch of DOP-26 alloy.^{3 4} This test was used to compare the weld-quench cracking susceptibilities of S, V, and X batches of alloy before V and X CVS's were used in production welding. CVS's from each alloy batch were welded by SRP-EED using a four-pole arc pattern shaper. Weld-quench cracks were detected by visual and dye penetrant examinations by SRP-EED and by scanning electron microscope (SEM) examinations at SRL. The X-batch CVS exhibited much more severe weld-quench cracking than CVS's from batches V and S. Weld-quench cracking was slightly more severe in the V-batch CVS than in the S-batch CVS. The top and bottom cups in the V and X CVS's showed large differences in weld-quench cracking severity. It is thought that the weld-quench cracking susceptibility of V-batch alloy is sufficiently low for acceptable production welding, but use of X-batch alloy remains questionable. Results of weld-patch tests at ORNL have also indicated that X-batch alloy may have unacceptable weldability. SRP plans further evaluations by using eighteen CVS's each from V and X-batches in production welding. Final decisions on acceptance of V and X alloys for further production welding will be based on evaluation results. ORNL plans to stop shipment of X-batch blanks to MF until SRP demonstrates an adequate acceptance rate with production welds.

DISCUSSION

CVS's of cups from S(T42), V(T43), and X(T44) batches of DOP-26 iridium alloy were welded by SRP-EED using a four-pole arc pattern shaper as used presently in PuFF Facility production welding. Full rotation welds were made in the girth and in the top and bottom cups. Each weld was terminated with a long taper quench. Each full weld was overlapped with four short welds terminated with a short taper quench. Thus, each CVS contained fifteen quench areas.

Each CVS was cut into sections and interior surfaces were examined visually and by the dye penetrant method by SRP-EED. The specimens were cleaned and examined with the SEM at SRL. Results of these examinations are given in Table 1. The SEM examinations generally confirmed the dye penetrant test results. However, the SEM did not reveal cracks at many locations indicated by the dye. Either the crack widths were too small ($<2\text{ }\mu\text{m}$) to be detected at 50-100X magnifications or rough grain boundaries produced ledges that retained dye causing false crack indications. SEM examinations allowed classification of weld-quench cracks according to length and width. Examples of wide (width $>10\text{ }\mu\text{m}$) and narrow (width $<10\text{ }\mu\text{m}$) cracks are shown in Figures 1 and 2. Wide cracks generally occurred along the centerline of the first pass weld just ahead of the point where full penetration ceased during quenching. Wide cracks penetrate deeply into the weld (sometimes into the columnar grain region of the quench taper) and produce large signals during ultrasonic nondestructive examination (NDE). Narrow cracks occur along grain boundaries in alloy welded during the first pass and are generally oriented perpendicular to the weld direction. These cracks occasionally extend into the twice-welded alloy in the quench taper. Grain surfaces exposed by both wide and narrow cracks are characterized by the same ridge network structure observed previously in weld-quench cracks in welds made with a two-pole arc oscillator.

Relative weld-quench cracking susceptibilities for the S, V, and X CVS's and for each cup were assessed using the following criteria:

- 1) number of cracked quench areas,
- 2) number of cracks,
- 3) number of wide cracks,
- 4) total crack length,
- 5) average crack length, and
- 6) average number of cracks per quench.

Results are given in Table 2. All the criteria except "average crack length" indicate that X-batch alloy undergoes much more severe weld-quench cracking than S and V batches. V-batch alloy appears slightly more susceptible to weld-quench cracking than S-batch. There were large differences in numbers of cracks (and, hence, total lengths of cracks) for the two cups in each CVS. These differences were reported to ORNL so that weld-quench cracking susceptibility might be correlated with alloy processing.

TABLE 1

Weld-Quench Cracking in S, V, and X Batches of DOP-26 Iridium Alloy

Results of Examinations of Multiple Quench Areas in CVS's Welded by SRP-EED Using a 4-Pole Arc Pattern Shaper

CVS	Weld	Cup	Dye Penetrant Examinations		SEM Examinations			Total Length of Cracks (μm)	Average Crack Length (μm)	Average No. of Cracks in a Quench Area
			No. of Cracked Quench Areas	No. of Crack Indications	No. of Cracked Quench Areas	No. of Cracks	No. of Wide Cracks ($W > 10\mu\text{m}$)			
42	Top	S31-5	4	7	2	2	0	660	330	0.4
	Center	Both	2	3	1	1	0	100	100	0.2
	Bottom	S33-2	4	15	4	15	1	3660	244	3.0
	Total		10	25	7	18	1	4420	246	1.2
43	Top	V217-5	5	27	4	11	4	5280	480	2.2
	Center	Both	3	12	1	1	0	200	200	0.2
	Bottom	V211-2	1	1	2	4	0	1090	273	0.8
	Total		9	40	7	16	4	6570	410	1.1
44	Top	X302-1	4	7	4	5	2	850	170	1.0
	Center	Both	4	11	3	3	0	440	147	0.6
	Bottom	X309-4	5	56	5	24	5	7370	307	4.8
	Total		13	74	12	32	7	8660	271	2.1

Ridge network on grain surface in crack

FIGURE 1. A Wide Weld-Quench Crack in the Bottom Cup (X309-4) of CVS T44

Ridge network on grain surface in crack

FIGURE 2. Narrow Weld-Quench Cracks in Bottom Cup (X309-4) of CVS T44

TABLE 2

Weld-Quench Cracking Susceptibilities Based on SEM Examinations

	<u>Criteria</u>	<u>By Alloy Batch</u>	<u>By Cup Designation</u>
1	Number of Cracked Quench Areas	X >> V = S	X309-4 > X302-1 = V217-3 = S33-2 > V211-2 = S31-5
2	Number of Cracks	X >> S > V	X309-4 > S33-2 > V217-3 > X302-1 > V211-2 > S31-5
3	Number of Wide Cracks	X >> V >> S	X309-4 > V217-3 > X302-1 > S33-2 > V211-2 = S31-5
4	Total Length of Cracks	X > V > S	X309-4 > V217-3 > S33-2 > V211-2 > X302-1 > S31-5
5	Average Crack Length	V >> X > S	V217-3 > S31-5 > X309-4 > V211-2 > S33-2 > X302-1
6	Average Number of Cracks/Quench	X >> S > V	X309-4 > S33-2 > V217-3 > X302-1 > V211-2 > S31-5
	Overall*	X >> V > S	X309-4 > V217-3 = S33-2 > X302-1 > V211-2 > S31-5

* Doesn't include criterion #5 (Average Crack Length).

PuFF PELLET FRACTURE

Introduction

Analysis of fracture incidence of vacuum outgassed GPHS production pellets from spring of 1981 (pellet 85) through August, 1982 (up to about pellet 475) showed a cyclical trend in pellet fracture incidence (Figure 3). The average period of the cycle was about every 50 pellets, meaning that, approximately every 50 pellets the cycle of the quality of the pellets peaked at a maximum fracture tendency. A fracture index was established to quantify the fracture tendency based on frequency of fracture over groups of 10 pellets and the quality of fracture, i.e., the extent of breakup of the pellets. A high fracture index indicated a period of high fracture tendency of the pellets. The cause of the cyclic fracture tendency has not been identified. Although use of argon fired low fired shards benefited the production process, it did not noticeably change the fracture tendency of pellets, either with respect to the magnitude of the fracture index or to the frequency of the cycle. A noticeable reduction in the magnitude of the fracture index was observed starting with the beginning of two shift operation in May 1982. However, the cyclic nature persisted.

Recent Production History

Beginning in September 1982, the fracture index went down and has remained the lowest in production history through December 1982 (Figure 3). Figure 3 shows that the cyclic pattern was broken during the last quarter production. However, based on the most recent data (Figure 3) it is not certain whether the break in the cyclic pattern represented a permanent change or only an interruption.

Although the specific cause or causes of the improved fracture performance of the pellets have not been identified, two observations suggest that there has been a change in the sintering properties of the $^{238}\text{PuO}_2$ powder: (1) die closure times have been much shorter, on the order of 5-10 minutes versus 15-30 minutes during the period of peak fracture index; (2) there has been a higher incidence of long pellets. Tests by Separations Technology found no direct correlation between pellet length and time at temperature after die closure, top and bottom ram loading differences, or die closure time. However, the extra pellet length may help reduce fracture. Earlier work at SRL⁵ showed that cracking is mitigated as density decreases.

A change in sintering properties of the PuO_2 suggests either a change in the feed preparation in HB-Line or in PuFF powder processing. However, no easily identifiable changes in feed preparation or powder processing have been observed. Monitoring of the fracture index will continue. Work will also continue by both Separations Technology and SRL to correlate the improved fracture resistance of the GPHS pellets with process conditions.

*10-Group Fracture Index minus
Mean Fracture Index from
5-Group (7).

FIGURE 3. Observed Fracture Index

FUEL/IRIDIUM CLADDING COMPATIBILITY STUDIES -- CHEMICAL ANALYSIS OF CONTROL IRIDIUM

Introduction

A study of the chemical compatibility between $^{238}\text{PuO}_2$ fuel and its iridium cladding is being carried out jointly by the Savannah River Laboratory (SRL), Los Alamos National Laboratory (LANL), and Oak Ridge National Laboratory (ORNL). The study follows the revised SRL Ir/ $^{238}\text{PuO}_2$ Compatibility Test Plan, which was designed to evaluate the effects of fuel impurities such as phosphorus, calcium, silicon, aluminum and iron, on the chemistry and microstructure of iridium cladding and plugging of the vents in the iridium cladding. This report describes work done by ORNL to cross calibrate their contained and uncontained spark source mass spectrometers (SSMS), and the results of their SSMS analysis of unaged control iridium.

Calibration of Contained and Uncontained SSMS Instruments

ORNL uses a contained spark source mass spectrometer for analysis of iridium samples aged in contact with PuO_2 , and an uncontained mass spectrometer for analysis of all other uncontaminated iridium including iridium blanks produced by Oak Ridge for forming into cups at Mound Facility. To ensure that the results obtained on each machine would be correlatable with each other, ORNL prepared two DOP 26 iridium standards—one for each machine. The analyses of each standard run on the uncontained machine are compared in Table 3 for three elements. Using tungsten as the internal standard the ratio of the measured concentration of aluminum in each standard sample was very nearly 1, showing excellent agreement between standards. The ratio of thorium concentration was not as good (although within error limits) which probably reflects the inhomogeneous distribution of thorium within the iridium.

SSMS of Unaged Archive Rings

Archive rings cut from the iridium cups used to encapsulate the compatibility test pellets were analyzed and the results compared with the SSMS analysis of the blanks from which the cups were made. Table 4 gives the measured concentrations of tungsten, aluminum and thorium of the archive rings. Table 5 compares the average concentration of each element from all the rings from the same batch with the average batch composition after rolling into sheets. The ratio of the concentration of elements in the archive rings to that of the original batch (Table 5) shows that the concentration of aluminum in the archive rings (and hence the finished cups) is significantly lower than in the original batch.

There may also have been a decrease in the thorium concentration. Each of the archive rings used in this study has been given the same heat treatments as flight quality cups (Table 6). It is possible that the aluminum is lost during the one hour, high temperature recrystallization and final outgassing steps.

TABLE 3

Comparison of DOP-26 Standards for Uncontained and Contained Spark Source Mass Spectrometers

(Ratio = Uncontained/contained)

<u>Element</u>	<u>Ratio</u>	<u>Points Sampled</u>	<u>Remarks</u>
W	1.0	-	Internal Standard
Al	0.98 ± 0.24	7	
Th	1.38 ± 0.40	14	

TABLE 4

SSMS Analysis of Unaged Archive Rings (Wt. ppm)

<u>Pellet</u>	<u>T-40</u>	<u>T-40</u>	<u>44t*</u>	<u>44b</u>	<u>45t</u>	<u>45b</u>	<u>48t</u>	<u>48b</u>	<u>49t</u>	<u>49b</u>
<u>El</u>	<u>PR719-6</u>	<u>NR520-2</u>	<u>L256-8</u>	<u>L254-7</u>	<u>L257R2</u>	<u>L255-6</u>	<u>P701-4</u>	<u>N502-3</u>	<u>P714-5</u>	<u>PR715-2</u>
Al	49	13	27	15	43	10	26	34	27	48
Th	42	46	50	36	60	20	27	53	46	65
W	3.5k	2.5k	2.8k	2.5k	3.1k	2.9k	2.8k	3.1k	3.2k	3.6k

* t = vented cup

b = unvented cup

TABLE 5

Comparison of Composition of Unaged Control Archive Rings to Iridium Batch Composition

<u>Avg of Compatibility Cups</u>	<u>Batch Avg*</u>	<u>Ratio</u>
<u>L-Batch</u>		
Al = 23.8	55.8	0.43
Th = 41.5	53.6	0.77
W = 2.8k	3.2k	0.88
<u>N-Batch</u>		
Al = 23.5	55.1	0.43
Th = 49.5	58.0	0.85
W = 2.8k	3.0k	0.93
<u>P-Batch</u>		
Al = 37.5	57.8	0.65
Th = 45.0	57.5	0.78
W = 3.3k	3.2k	1.03

* Based on analyses of sheets for cups sent to SRP.

TABLE 6

Heat Treatments Given GPHS Flight Quality Iridium Cups

Forming

- First draw: $925 \pm 25^{\circ}\text{C}$ for 10 min.
- Second draw: $925 \pm 25^{\circ}\text{C}$ for 10 min.

Recrystallization

- $1300 \pm 25^{\circ}\text{C}$ for 1 hr in vacuum ($<1 \times 10^{-4}$ torr)

Final Outgassing

- $1500 \pm 25^{\circ}\text{C}$ for 1 hr in vacuum ($<1 \times 10^{-4}$ torr)

SURFACE AREA ANALYSIS OF PU-238 OXIDE

Introduction

There is a need for powder characterization techniques other than particle size to characterize PuFF feed powder and shards so that correlations between feed/shard properties and GPHS pellet quality can be assessed. Two methods are currently available to characterize powder: Coulter counter and scanning electron microscopy (SEM)*. Coulter counter particle size distribution analysis provides useful quantitative information on one aspect of powder properties: particle mode size and size distributions. However, experience in PuFF has indicated that the Coulter counter size data may not have enough resolving power to discriminate among feed powders that impart small but possibly significant differences to pellet fracture behavior. In addition, Coulter counter cannot be used to characterize shards because shard size lies outside the range of the instrument. Scanning electron microscopy (SEM) provides important qualitative information on particle morphology, but cannot quantitatively distinguish powders on the basis of morphology. SEM is also useful to verify Coulter counter data to within an order of magnitude.

Surface area is a critical powder property that is often used to correlate powders with their sinterability. It is usually measured using BET nitrogen adsorption techniques. However, BET cannot be used with Pu-238 because of the self-heating effects of this isotope. Another surface area measuring technique, Blaine fineness analysis, has been tested recently at SRL with promising results. The Blaine method is an air permeability technique and measures surface area inferentially by assuming that for a given quantity of powder in a fixed volume, the surface area of the powder will be proportional to the time it takes for a fixed volume of air to be drawn through the powder plug. By contrast, BET measures the surface area directly by measuring the amount of nitrogen desorbed from the surface. For porous particles, such as agglomerates, the BET surface area will be much larger than the Blaine surface area. For some applications, such as for shards, the Blaine surface area may be more useful than the BET total surface area. The Blaine shard surface area would be a measure of that portion of the surface dealing with shard-to-shard sintering and therefore should correlate with intershard porosity. Hence, the Blaine method may be able to differentiate among shards that have different size distributions due to differences in the sharding operation. The means to measure such possible differences has not heretofore been available to PuFF. If the Blaine method works out, it will be the first technique available to quantitatively characterize both feed and shards.

* Contained SEM capability is expected to be available again in 1983.

Comparison of Blaine vs BET Surface Areas

Surface area analyses were completed on samples of five different nonradioactive powders using a Blaine fineness tester. The powders had regularly shaped particles with a fairly narrow size range (Figure 4). Each of the five powders differed in average particle size, ranging from an average particle size of about 500 μm to 2-3 μm . Duplicate samples of the powders were sent for BET analysis by Micromeritics Corp., Atlanta GA. The Blaine surface area was also measured on a sample of NBS calibrated glass beads (Figure 4). The results of these measurements (Table 7) showed approximately factor of two agreement between the BET method and the Blaine method of surface area measurement. One discrepancy was observed in which the BET result was about 30 times greater than the Blaine results. Subsequent SEM work revealed that the standard heat treatment given the BET sample by Micromeritics caused extensive cracking on individual sample particles (Figure 5). Many of the particles were observed to crumble into smaller particles. This led to the large measured surface area. Factor of two agreement is considered excellent for two methods based on different principles.

In addition to its accuracy, the Blaine method has been reproducible of itself to within about 4% in SRL tests. It is expected that the accuracy and reproducibility that has been demonstrated by the Blaine method will be adequate to differentiate between feed powders of significantly different characteristics, i.e., powders that will result in noticeable differences in fracture behavior of hot pressed GPHS pellets. Nevertheless, the power of differentiation of the method is being assessed using thorium oxalate powders precipitated according to a parametric pattern. For this assessment surface area measurements will be correlated with particle size measured by Coulter counter and morphology observed by SEM. Actual pellets will not be made. After measurements on the oxalate powders are complete, the oxalates will be calcined to the oxide and measurements will be repeated.

100 μm

100 μm

100 μm

FIGURE 4. Morphology of Particles Tested by Both BET and Blaine Surface Area techniques (a) KCl ; (b) $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$; (c) KNO_3

20 μm

20 μm

20 μm

FIGURE 4 cont. (d) $\text{Ce}_2(\text{C}_2\text{O}_4)_3 \cdot 9\text{H}_2\text{O}$; (e) $\text{I}/2\text{O}_3$; (f) NBS Glass Beads

TABLE 7**Surface Area - Measured by Blaine Fineness Tester and BET Methods**

<u>Sample</u>	<u>Blaine, cm²/g</u>	<u>BET, cm²/g</u>	<u>Ratio</u>
KCl	256	145	0.57
K ₂ C ₂ O ₄ • H ₂ O	357	9600	26.9
KNO ₃	539	311	0.56
Ce ₂ (C ₂ O ₄) ₃ • 9H ₂ O	3117	7244	2.3
Y ₂ O ₃	11,274	43,000	3.81
NBS Glass Beads	1805	1730 ± 50*	0.96

* Measured by National Bureau of Standards.

FIGURE 5. Breakup of $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ Particle on Heating at 90°C for 12 hr in Vacuum

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