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^{238}Pu FUEL FORM PROCESSES
QUARTERLY REPORT

JULY - SEPTEMBER 1981

E. I. du Pont de Nemours & Co.
Savannah River Laboratory
Aiken, SC 29808

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

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FOREWORD

This report is one of a series 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.

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GENERAL-PURPOSE HEAT SOURCE (GPHS) PROCESS SUPPORT

ANALYTICAL STUDIES OF WELD CRACKING IN DOP-26 IRIDIUM ALLOY CLAD VENT SETS

Cracking of welds in DOP-26 iridium alloy clad vent sets beneath the quench taper appears to be caused by grain-boundary porosity in the form of ridge networks. A region several atom layers thick with unusually high thorium content and trace carbon was detected in ridge networks. Ridge network porosity was also detected in the heat-affected zone beneath the quench taper of the girth weld on a Multi-Hundred Watt DOP-26 iridium alloy Post Impact Containment Shell.

Introduction and Summary

Inspection of welds of DOP-26 iridium alloy clad vent sets (CVS) made in the Plutonium Fuel Form (PuFF) Facility prior to April 1981 revealed that about one weld out of six had interior cracks in the single-pass region beneath the quench taper. Scanning electron microscopy (SEM) of welds made in the PuFF Facility and welds made by the Engineering Equipment Division of the Savannah River Plant (SRP-EED) showed that grain boundaries in cracks and in areas susceptible to cracking had ridge networks that were not present in other weld areas.¹ These ridge networks were thought to contain low melting phases but Scanning Electron Microprobe Quantometer (SEMQ) analyses showed the ridges and areas between the ridges to have the same composition as that of the DOP-26 alloy. Areas of a thorium-bearing grain-boundary phase were detected at the edges of ridge networks.

The GPHS Iridium Welding Team met several times between March and May and proposed several "fixes" to reduce weld cracking. One of these "fixes", a long quench taper, was tested and adopted for PuFF Facility production welding which resumed in July. During this reporting period, an ultrasonic nondestructive examination technique was developed by SRP-EED and installed in the PuFF Facility for examining fueled clads for weld cracking. Welds with the long quench taper cracked with the same frequency (about one out of six) as did those with the shorter quench taper, but SRP reported that cracks beneath the quench taper were smaller.

During this reporting period, the contained SEM was out of service and contaminated specimens of welds from the PuFF Facility could not be examined. Uncontaminated specimens from CVS No. T2 which was welded by SRP-EED, and a spherical Multi-Hundred Watt (MHW) Post Impact Containment Shell (PICS), 101T, which was welded in the PuFF Facility in 1976, were analyzed using the scanning Auger microprobe (SAM), SEM, and SEMQ at SRL. Specimens from CVS No. T2 were also analyzed with the high-resolution SAM (SUPER SAM) at Oak Ridge National Laboratory (ORNL). As reported previously,¹ only small, transverse cracks at one edge of the girth weld were detected on CVS No. T2. No cracks were detected in the girth weld on MHW PICS 101T.

Analyses performed during this reporting period have shown that the ridge networks are regions of grain-boundary porosity. Except for contact through the ridges, adjacent grains in these areas are separated by $\sim 0.1 \mu\text{m}$. This grain-boundary porosity, rather than low-melting grain-boundary phases, is now thought to be the primary cause of cracking in DOP-26 alloy during production welding in the PuFF Facility prior to April 1981. This type of weld cracking, which only occurs in heat-affected zones beneath the quench taper and at the edges of the molten zone, differs from cracking observed by ORNL along the centerline of single pass weld areas in iridium alloys containing $>100 \text{ ppm}$ thorium.²

Thorium at $\sim 60 \text{ ppm}$ is added to DOP-26 iridium alloy to strengthen the grain boundaries.³ Strengthening is generally attributed to formation of a thorium-enriched region at the grain boundaries several atom layers thick.⁴ SUPER SAM analyses confirmed the existence of this region on grain boundaries in both welded and unwelded alloy from CVS No. T2. The average $\text{Th/Ir} = \sim 0.2$ (atom fraction) suggests that the phase in these regions may be ThIr_5 . In contrast, $\text{Th/Ir} = \sim 0.4$ and $\text{C/Ir} = \sim 0.1$ on grain surfaces in ridge networks. These results do not correlate with any known Ir-Th-C compound. It is thought that thorium and carbon are involved in the formation of the ridge network porosity that causes weld cracking.

Several types of thorium-bearing grain-boundary deposits have been detected in DOP-26 alloy. Thorium-bearing areas at the edges of the ridge networks are $\sim 0.1 \mu\text{m}$ thick and $\sim 10 \mu\text{m}$ wide. Thorium-bearing stringers on grain boundaries in welded alloy near edges of the molten zone are $\sim 1 \mu\text{m}$ wide and $50\text{--}100 \mu\text{m}$ long in the direction of grain elongation. Micron-size thorium-bearing inclusions occur on grain boundaries in unwelded alloy. Analyses of these deposits have indicated $\leq 8 \text{ wt } \%$ thorium or $\text{Th/Ir} \leq 0.07$. Phases with such low thorium contents are not expected to melt at temperatures significantly below the 2440°C melting point of iridium, and therefore, are not considered to be contributors to weld cracking.

Analyses of welds in DOP-26 iridium will continue. Welds made with the long quench taper proposed by the GPHS Iridium Welding Team will be analyzed. Savannah River Laboratory (SRL) is also coordinating development of a patch weldability test for DOP-26 alloy at ORNL.

Discussion

Analyses of Specimens from CVS No. T2

The girth weld in CVS No. T2 was made by SRP-EED under conditions similar to those used in the PuFF Facility in January 1981. Three overlap areas with short quench tapers were made on the girth weld to provide specimens for analytical studies. Analyses begun during the previous reporting period were continued. Small, transverse cracks detected at one edge of the girth weld were described in the previous report.¹ No additional weld cracks have been detected.

First quench

The specimen with the first quench was fractured during SAM analyses described previously.¹ The same ridge network on the two mating surfaces has been analyzed with the SEMQ. Figure 1 shows backscattered electron (BSE) images and corresponding thorium maps of the two surfaces. The ridge patterns on the two surfaces are exact mirror images of each other. This observation indicates that contact between adjacent grains occurs through the ridges. The areas between the ridges constitute grain-boundary porosity. Adjacent grains are separated by about 0.1 μm in these areas. Such network structures in the grain boundaries reduce adhesion between adjacent grains and promote intergranular cracking as observed in weld quench regions.

The thorium-bearing grain-boundary phase present at the edges of the ridge networks appears to be adherent to the grain surfaces. Figure 2 shows the locations of the thorium-bearing, grain-boundary phase (shaded areas) with respect to the ridge network on Surface B shown in Figure 1. When the specimen was fractured, some of this phase adhered to Surface A (vertical shading) while the remainder stayed on Surface B (horizontal shading). The thickness of the thorium-bearing phase appears to be about the same as the thickness of a ridge or about $\sim 0.05 \mu\text{m}$.

Results of SEMQ analyses for Ir, W, and Th at 14 points on Surface A are given in Table 1. Ridges and areas between the ridges had the same composition with thorium concentrations near

the detection limit of ~ 0.02 wt % thorium. Analysis Points 1, 3, and 10 with 3.50 - 3.70 w/o thorium correspond to areas where the thorium-bearing grain boundary phase adhered to Surface A while points 5, 11, 12, and 13 with less thorium correspond to areas where the thorium-bearing phase was detached during fracturing and adhered to Surface B. The SEMQ analysis also revealed a gradient in the tungsten concentration which varied from 0.00 wt % at the grain edge to 0.51 wt % at 16 μm from the grain edge (Figure 3).^{*} This tungsten variation is probably not related to formation of the ridge network.

Third quench

The specimen with the third quench had been fractured across the weld through the quench in earlier analyses of cracks located at the edge of the molten zone.¹ Portions of the specimen were sent to ORNL where SUPER SAM analyses were performed on specimens from the quench and single-pass areas of the girth weld and unwelded alloy from the cup wall (Figure 4). Specimens were fractured in the SUPER SAM under ultra-high vacuum of $1.2 - 1.5 \times 10^{-10}$ torr. Analyses made within a few minutes of fracturing detected no oxygen on the fracture surfaces. (Oxygen in vacuum contaminants often absorbs on fracture surfaces, but none was observed in these analyses.) After analyses of the as-fractured specimens, the quench and single pass weld specimens were sputtered for 5 minutes with 5-keV argon ions at $\sim 24 \mu\text{A cm}^{-2}$ and reanalyzed to get an indication of the thicknesses of surface phases.

Quench Taper. The specimen containing the quench taper fractured in the SUPER SAM along the weld centerline. Unfortunately, this specimen did not include the region most susceptible to cracking, which occurs under the quench taper close to where full penetration ceases. However, a small ridge network was located and analyzed.

Six points in and around the ridge network (Figure 5) were analyzed after fracturing and after 5 minutes of sputtering. Results are given in Table 2. No difference in composition was noted between a ridge and points between ridges. Points in the ridge network had $\text{Th/Ir} = 0.36 - 0.45$ (atom fraction) and $\text{C/Ir} = 0.10 - 0.15$ compared to $\text{Th/Ir} = 0.15 - 0.26$ and no detectable carbon ($\text{C/Ir} < 0.06$) for points outside the ridge network. No other elements were detected. Sputtering for five minutes removed

^{*} This variation in tungsten is not caused by an instrumental effect since tungsten was measured with the same spectrometer as was iridium, which varied less than 5% (relative).

the thorium at all the points except No. 6. The presence of a thorium-bearing area at this point was confirmed in subsequent SEMQ analysis (Figure 6).

Single-Pass Weld. This specimen of the single-pass weld fractured primarily intergranularly along the weld centerline. The exposed grain boundaries had the same appearance as that of the single-pass weld area near the first quench.¹ No ridge networks were observed. Results of SUPER SAM analyses are given in Table 3. Analyses of eight points on as-fractured grain boundaries showed Th/Ir = 0.12 - 0.34 with an average value of 0.21; Th/Ir = 0.04 for a fractured grain. No other elements were detected. Sputtering for 5 minutes removed the thorium.

Unwelded Alloy. This specimen of unwelded alloy fractured both intergranularly and transgranularly like the unwelded specimen described previously.¹ Results of SUPER SAM analyses are given in Table 4. Analyses of four points on grain boundaries showed Th/Ir = 0.13 - 0.25 with an average of 0.20. Two points on fractured grains showed Th/Ir = 0.05. No other elements were detected. Analyses were not performed after sputtering this specimen.

Analyses of specimens from MHW PICS 101T.

No weld cracks were detected in SEM examinations of the girth weld in MHW PICS 101T near the quench taper. Specimens containing the quench taper, single pass and double pass weld areas and unwelded alloy from MHW PICS 101T were fractured in the ultra-high vacuum of the SRL SAM. The single pass weld and unwelded specimens were analyzed briefly. A malfunction in the SAM prevented analysis of the quench and double-pass areas. Another specimen was fractured in air across the single-pass weld area. Fracture surfaces on all the specimens were examined on the SEM. Selected areas on several specimens were analyzed with SEMQ.

Quench Taper.

The specimen containing the quench taper fractured along the weld centerline (Figure 7). Grain boundary structures in several areas of the quench region are shown in Figures 7b and 7c. Ridge networks occurred only on grain boundaries close to where full weld penetration ceased. SEMQ analysis showed areas of a thorium-bearing, grain-boundary phase at the edges of the ridge networks (Figure 8).

Single-Pass Weld.

The specimen of the single-pass weld fractured along the weld centerline (Figure 9). The exposed grain boundaries had rows of 0.2 μ m diameter pores and inclusions oriented parallel to the length of the grain. No ridge networks were observed. SAM analysis showed Th/Ir = 0.165 at a point on this surface.

Sputtering rapidly removed the thorium as shown in Figure 10. This sputtering profile is similar to the one recorded for CVS No. T2 which was welded by EED.¹ Analyses after sputtering revealed several points of residual thorium with Th/Ir ≤ 0.08 .

Double-Pass Weld.

The specimen of the double-pass weld fractured along the weld centerline (Figure 11). Rows of 0.4 μm diameter pores and inclusions were oriented parallel to the length of grains. More of these pores and inclusions were observed on this double-pass-weld grain boundary than on the single-pass-weld grain boundary. No ridge networks were observed.

Unwelded Alloy.

The fractured surface of the unwelded alloy showed both transgranular and intergranular fractures (Figure 12). Grain boundaries has no ridge networks and relatively few pores and inclusions. SAM analysis showed Th/Ir = 0.15 at a point on this surface. The Th/Ir profile (Figure 10) indicated that the thickness of this thin thorium-bearing, grain-boundary phase is about the same as that on grain boundaries in welded alloy. This profile differs from the one recorded for unwelded alloy from CVS No. T2.¹ This difference suggests that the unusual shape of the profile for the unwelded alloy from CVS No. T2 may have been caused by rough surface topography interfering with sputtering.

Specimen Fractured Across the Single-Pass Weld.

Figure 13 shows the surface of MHW PICS 101T exposed by fracturing across the single pass weld region. The weld structure is similar to that observed for CVS No. T2 fractured in a similar manner.¹ Ridge networks were detected only on grain boundaries in the weld near the edge of the molten zone (Figure 14a). Thorium-bearing areas existed at the edges of the ridge-network and on the grain face. Thorium-bearing areas were detected along a grain edge and on a grain face on grains closer to the weld centerline (Figure 14b). Grains in the weld centerline fractured transgranularly (Figure 14c). Unwelded alloy fractured both intergranularly and transgranularly. Micron-size, thorium-bearing inclusions were detected on grain faces.

Future Studies

Long Quench Taper

CVS No. T25, which was welded by SRP-EED with the long quench taper "fix" proposed by the GPHS Iridium Welding Team, will be analyzed. Several large weld cracks have been detected by optical

examinations. Compositions and morphologies of grain boundary features associated with the cracks will be compared with those observed in short quench taper specimens.

DOP-26 Alloy Weldability Test

SRP has funded ORNL to develop a weldability patch test for DOP-26 alloy. SRL will coordinate this development. The test is to be performed on alloy disks that are heat treated to produce a grain structure like that in CVS's welded in the PuFF Facility. Welding conditions should simulate those used in the PuFF Facility, including overlap and quench regions. This development should take three to four months.

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TABLE 1

Results* of SEMQ Analyses of Surface A of
Fractured Quench Area of CVS No.T2

Element	Analysis Points in Ridge Network						
	Ridges		Area Between Ridges				
	No. 7	No. 8	No. 2	No. 4	No. 6	No. 9	No. 14
Ir	99.96	99.68	99.95	99.65	99.80	99.69	99.45
W	0.02	0.28	0.00	0.31	0.14	0.28	0.51
Th	0.02	0.04	0.05	0.04	0.06	0.03	0.04

Element	Analysis Points in Area Around Ridge Network						
	No. 1	No. 3	No. 5	No. 10	No. 11	No. 12	No. 13
	No. 1	No. 3	No. 5	No. 10	No. 11	No. 12	No. 13
Ir	96.25	96.26	97.82	96.14	99.48	98.77	99.58
W	0.05	0.24	0.07	0.19	0.50	0.08	0.06
Th	3.70	3.50	2.11	3.67	0.02	1.15	0.36

* Weight per cent.

TABLE 2

Results of SUPER SAM Analysis of a Ridge Network
on the Quench Specimen from CVS No. T2

Analysis Point No.	As Fractured	Auger Peak Intensity Ratios*			Estimated Atom Fractions			
		Th(67eV) Ir(54eV)	C(272eV) Ir(229eV)	O(503eV) Ir(229eV)	Ir	Th	C	O
1	Ridge	0.36	0.35	<0.2	0.68	0.24	0.07	<0.01
2	Points	0.45	0.43	<0.2	0.62	0.28	0.09	<0.01
3	Between Ridges	0.42	0.43	<0.2	0.63	0.27	0.09	<0.01
4	Grain Boundary	0.15	<0.2	<0.2	0.81	0.12	<0.05	<0.02
5	Outside	0.22	<0.2	<0.2	0.77	0.17	<0.05	<0.02
6	Ridge Network	0.26	<0.2	<0.2	0.75	0.19	<0.04	<0.02
Same Points After Sputtering 5 minutes								
1	Ridge	<.02	0.80†	<0.3	0.77	<0.02	0.20†	<0.02
2	Points	<.02	0.72†	<0.3	0.79	<0.02	0.18†	<0.02
3	Between Ridges	<.02	0.82†	<0.3	0.77	<0.02	0.20†	<0.02
4	Grain Boundary	<.02	0.86†	<0.3	0.78	<0.02	0.21†	<0.02
5	Outside	0.02	0.70†	<0.3	0.79	0.02	0.17†	<0.02
6	Ridge Network	0.07	0.61†	<0.3	0.78	0.05	0.15†	<0.02

* Peaks reported as < were not detected.

† Carbon probably introduced by sputtering.

TABLE 3
Results of SUPER SAM Analysis of the
Single-Pass Weld Specimen from CVS No. T2

Analysis Point No.	Area 1	<u>Auger Peak Intensity Ratios*</u>			<u>Estimated Atom Fractions</u>			
		<u>Th(67eV)</u> <u>Ir(54eV)</u>	<u>C(272eV)</u> <u>Ir(229eV)</u>	<u>O(503eV)</u> <u>Ir(229eV)</u>	<u>Ir</u>	<u>Th</u>	<u>C</u>	<u>O</u>
1	Grain Boundary	0.18	<.3	<.3	0.77	0.14	<.08	<.02
2	Fractured Grain	0.04	<.3	<.3	0.86	0.03	<.09	<.02
3	Grain Boundary	0.20	<.3	<.3	0.76	0.15	<.08	<.02
	<u>Area 2</u>							
1	} Grain Boundaries	0.12	<.3	<.3	0.81	0.10	<.08	<.02
2		0.23	<.3	<.3	0.74	0.17	<.07	<.02
3		0.34	<.3	<.3	0.68	0.23	<.07	<.02
4		0.19	<.3	<.3	0.76	0.15	<.08	<.02
5		0.18	<.3	<.3	0.77	0.14	<.08	<.02
6		0.25	<.3	<.3	0.73	0.18	<.07	<.02
	<u>Area 3</u>							
	After Sputtering 5 minutes							
1	} Grain Boundaries	<.02	1.29†	<.2	0.68	<.02	0.29†	<.02
2		<.02	1.00†	<.2	0.73	<.02	0.24†	<.02

* Peaks reported as < were not detected.

† Carbon probably introduced by sputtering.

TABLE 4
Results of SUPER SAM Analysis of the
Single-Pass Weld Specimen from CVS No. T2

Analysis Point No.	Area 1	<u>Auger Peak Intensity Ratios*</u>			<u>Estimated Atom Fractions</u>			
		<u>Th(67eV)</u> <u>Ir(54eV)</u>	<u>C(272eV)</u> <u>Ir(229eV)</u>	<u>O(503eV)</u> <u>Ir(229eV)</u>	<u>Ir</u>	<u>Th</u>	<u>C</u>	<u>O</u>
1	Grain Boundary	0.18	<.3	<.3	0.77	0.14	<.08	<.02
2	Fractured Grain	0.04	<.3	<.3	0.86	0.03	<.09	<.02
3	Grain Boundary	0.20	<.3	<.3	0.76	0.15	<.08	<.02
	<u>Area 2</u>							
1	} Grain Boundaries	0.12	<.3	<.3	0.81	0.10	<.08	<.02
2		0.23	<.3	<.3	0.74	0.17	<.07	<.02
3		0.34	<.3	<.3	0.68	0.23	<.07	<.02
4		0.19	<.3	<.3	0.76	0.15	<.08	<.02
5		0.18	<.3	<.3	0.77	0.14	<.08	<.02
6		0.25	<.3	<.3	0.73	0.18	<.07	<.02
	<u>Area 3</u>							
	After Sputtering 5 minutes							
1	} Grain Boundaries	<.02	1.29†	<.2	0.68	<.02	0.29†	<.02
2		<.02	1.00†	<.2	0.73	<.02	0.24†	<.02

* Peaks reported as < were not detected.

† Carbon probably introduced by sputtering.

BSE Image

Th Map

Surface B

BSE Image

Th Map

Surface A

Figure 1. The Same Ridge Network on Mating Surfaces of the Specimen of the First Quench on CVS No. T2 Fractured Along Weld Centerline

Figure 2. Location of Thorium-Bearing Grain Boundary Phase with Respect to a Ridge Network on Surface B (see Figure 1)

Figure 3. Variation of Tungsten Concentration Across Surface A (See Figure 1)

Figure 4. Location of Specimens from the Third Quench on CVS T2 Analyzed Using the High-Resolution Scanning Auger Microprobe (SUPER SAM) at ORNL

Figure 5. Ridge Network on Grain Boundary in Heat-Affected Zones Beneath Third Quench Taper On CVS No. T2 Showing Location of Points Analyzed Using the SUPER SAM

BSE Image

Th Map

Figure 6. SEMQ Analysis of the Ridge Network on CVS No. T2
Analyzed Using the SUPER SAM

a. Structure of Quench Area

b. Heat-Affected Zone

c. Quench Taper

d. Single-Pass Area

Figure 7. Quench Area On MHW PICS 101T Fractured Along Weld Centerline

BSE Image

Th Map

Figure 8. SEMQ Analysis of Ridge Network on Grain Boundary in Heat-Affected Zone Beneath Quench Taper on MHW PICS 101T.

Figure 9. Single-Pass Weld Area On MHW PICS 101T

Figure 10. SAM Th/Ir Profiles for Specimens from MHW PICS 101T

Figure 11. Double-Pass Weld Area On MHW PICS 101T
Fractured Along Weld Centerline

Figure 12. Fractured Unwelded Alloy From MHW PICS 101T

Figure 13. Structure of Single-Pass Weld Area On MHW PICS 101T Fractured Across Weld.

BSE Image

Th Map

a. Edge of Molten Zone (A)

BSE Image

Th Map

b. Nearer Center of Weld (B)

Figure 14. Grain Boundaries in MHW PICS 101T Fractured Across Single-Pass Weld Area.

SEM Image

c. Transgranular Fracture in Weld Centerline (C)

Thorium-bearing inclusions

BSE Image

Th Map

d. Unwelded Alloy (D)

Figure 14 (Continued)

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