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GRAIN-BOUNDARY CAVITATION AND WELD-UNDERBEAD CRACKING IN DOP-26 IRIDIUM ALLOY

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GRAIN BOUNDARY CAVITATION AND WELD UNDERBEAD CRACKING IN DOP-26 IRIDIUM ALLOY*

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Plutonium-238 oxide fuel pellets for the General Purpose Heat Source Radioisotopic Thermoelectric Generators to be used on the NASA Galileo Mission to Jupiter and the International Solar Polar Mission are produced and encapsulated in DOP-26 iridium alloy (Fig. 1) at the Savannah River Plant. DOP-26 iridium alloy was developed at the Oak Ridge National Laboratory (ORNL) and contains nominally 0.3 weight-percent tungsten, 60-ppm thorium and 50-ppm aluminum.[1] Underbead cracks occasionally occur in the girth weld on the iridium alloy cladding in the area where the gas tungsten arc is quenched.[2] A variety of electron beam techniques have been used to determine the cause of cracking.

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Experimental

Specimens from $^{238}PuO_2$-fueled capsules were examined with a contained SEM (Cambridge Stereoscan 600) at the Savannah River Laboratory (SRL). Studies of grain surface structure and elemental distributions were performed on specimens from unfueled capsules using SEM/EDX (AMR 900/Kevex 5000A), EMPA (ARL SEMQ) and SAM (Perkin-Elmer 545 at SRL and Perkin-Elmer 590 at ORNL). The following observations were made:

1) Cracks occurred beneath the quench along grain boundaries in alloy (both welded and unwelded) affected by heat from quenching (Fig. 2). Some cracks extended into the columnar-grain region of the quench (Fig. 3). Cracking in welds away from quench areas was not significant.

2) Grain surfaces in cracks exhibited ridge networks extending over many grains (Fig. 4). Ridge network patterns on mating grain surfaces were mirror images of each other (Fig. 5). Mating depressions between ridges constituted grain-boundary cavities a few tenths of a micrometer thick and up to 10 μm across. Cavities had smooth, almost mirror-like surfaces. Fracturing of uncracked weld-quench areas generally revealed small ridge networks covering only a portion of a grain facet. Some small ridge networks were located near the centers of grain facets where it is apparent that mating grains had not separated during welding. Ridge networks were not present on grain surfaces in welded alloy away from quench areas.

3) Two other types of cavities have been detected in DOP-26 iridium alloy. Elongated cavities consisting of mating depressions a few tenths of a micrometer deep, a few micrometers wide and several tens of micrometers long were detected along grain edges (often along with small ridge network cavities) on grain surfaces in weld-quench areas, but not in welded alloy away from quench areas. Submicrometer pores were detected in grain boundaries and within grains of welded alloy, but not in unwelded alloy.

4) Thorium-bearing deposits in the form of particles, stringers and patches (some with eutectic structure) have been detected on grain boundaries of welded DOP-26 iridium alloy. Small cavities were located in areas without thorium-bearing deposits (Fig. 6) with only iridium and tungsten at concentrations corresponding to bulk alloy being detected by SEM/EDX and EMPA in ridges and cavity surfaces. Thorium-bearing particles were detected on surfaces of extended ridge networks. Cavities and thorium-bearing patches were found to respond differently to heat treatment. Heating a weld-quench region at 1500°C for one hour had no effect on grain boundary cavities but caused thorium-bearing patches to coalesce into micron-size particles and stringers. Heating at 2000°C for a few minutes apparently healed grain boundary cavities.

5) SAM analyses showed that ridge network surfaces contained carbon and calcium that were not present on uncavitated grain surfaces (Table I). Ridge networks surfaces also had thorium concentrations higher than those detected on grain surfaces in thorium-doped iridium alloys.[3]

Conclusions

Since the ridge networks that characterize weld-quench cracks in DOP-26 iridium alloy have been shown to constitute grain-boundary cavities, it is natural to consider cavity-forming mechanisms that weaken grain boundaries as possible causes of cracking. Gas porosity and high-temperature creep are two such mechanisms.

Gas trapped or formed in the alloy could migrate to grain boundaries and form cavities in areas affected by heat from quenching. Pores within grains of welded alloy are indications of a source of gas in DOP-26 iridium alloy. Pores, grain-edge cavities, and ridge networks could form as increasing quantities of gas are released to grain boundaries. Formation of ridge networks near the center of grain facets indicates a pressurization process. Ridge spacing indicates a greater cavity depth near the centers of ridge networks as expected for gas porosity. The smooth surfaces of cavities are a general characteristic of gas porosity. The SAM results suggest the pressurizing gas contains carbon and, possibly, calcium but not oxygen. Hydrogen and helium, two gases used in DOP-26 iridium alloy processing that are not detectable by Auger electron spectroscopy, could also be involved in cavity formation.

Creep is normally regarded as a very slow process; but, at temperatures near the melting point, intergranular cracking due to creep can occur rapidly.[4] Two types of grain-boundary cavities are formed by high-temperature creep. Cavities on grain faces (called r-type cavities), that form at temperatures very close to the melting point and at low stresses, often lead to cracking failures. Ridge networks in DOP-26 iridium alloy could be r-type cavities. Cavities at grain-boundary triple lines (called w-type cavities), that form at lower temperature and higher stress levels, produce small cracks. Grain-edge cavities in DOP-26 iridium alloy could be w-type cavities.

Grain-boundary liquation is usually not considered as a mechanism that produces grain-boundary cavities. The structural and compositional differences between ridge networks and thorium-bearing grain boundary deposits suggest that weld-quench cracking in DOP-26 iridium alloy may not be caused by the liquation mechanism that produces weld-metal cracking in iridium alloys containing greater than 100-ppm thorium.[5]

References

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TABLE 2

SAM Results for a Small Ridge Network

Analysis Point No.	As Fractured	Auger Peak Intensity Ratios*			Estimated Atom Fractions			
		Th (67 eV) / Ir (54 eV)	C (272 eV) / Ir (229 eV)	Ca (291 eV) / Ir (229 eV)	Ir	Th	C	Ca
1	Ridge	0.36	0.35	0.45	0.66	0.24	0.07	0.03
2	Points	0.45	0.43	<0.1	0.68	0.28	0.09	0.00
3	Between Ridges	0.42	0.43	<0.1	0.64	0.27	0.09	0.00
4	Grain Boundary	0.15	<0.2	<0.1	0.88	0.12	0.00	0.00
5	Outside	0.22	<0.2	<0.1	0.83	0.17	0.00	0.00
6	Ridge Network	0.26	<0.2	<0.1	0.81	0.19	0.00	0.00
	Same Points After Sputtering 5 minutes							
1	Ridge	<0.02	0.80†	<0.1	0.79	0.00	0.21†	0.00
2	Points	<0.02	0.72†	<0.1	0.81	0.00	0.19†	0.00
3	Between Ridges	<0.02	0.82†	<0.1	0.79	0.00	0.21†	0.00
4	Grain Boundary	<0.02	0.86†	<0.1	0.79	0.00	0.21†	0.00
5	Outside	0.02	0.70†	<0.1	0.81	0.02	0.17†	0.00
6	Ridge Network	0.07	0.61†	<0.1	0.80	0.05	0.15†	0.00

* Peaks reported as < were not detected.

† Carbon probably introduced by sputtering.

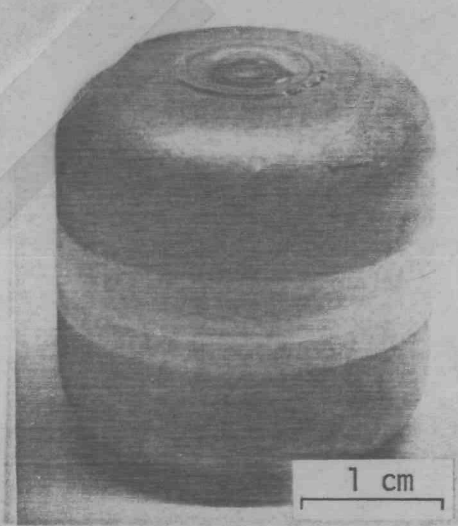

FIG. 1

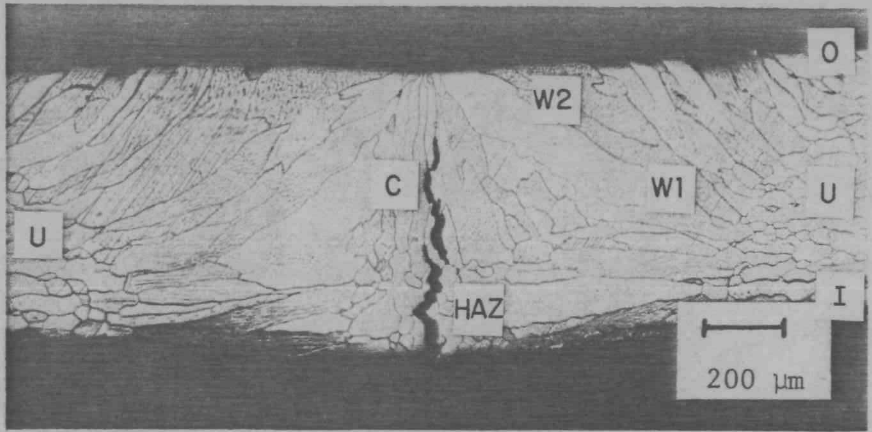

FIG. 3

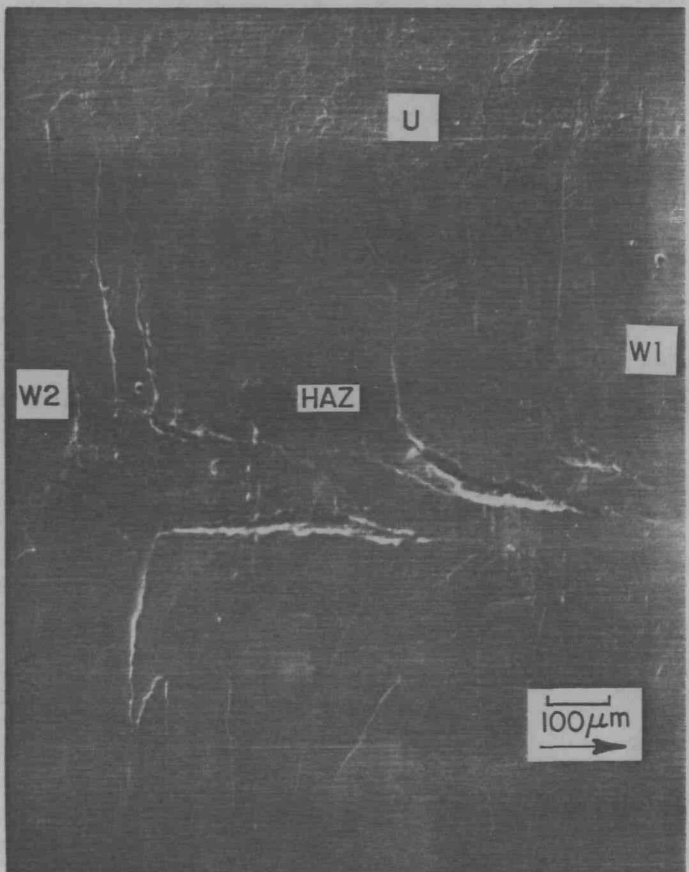

FIG. 2

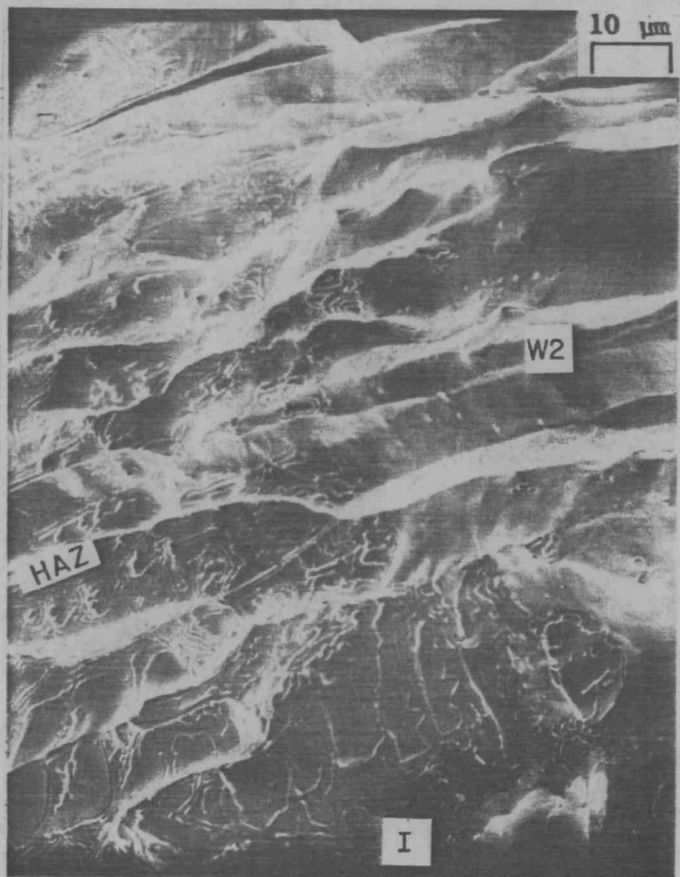

FIG. 4

LEGEND

I — Inner Surface
O — Outer Surface
U — Unwelded Alloy
W1 — Welded Once
W2 — Welded Twice
HAZ — Heat-Affected Zone in W1
C — Columnar Grains
← — Weld Direction

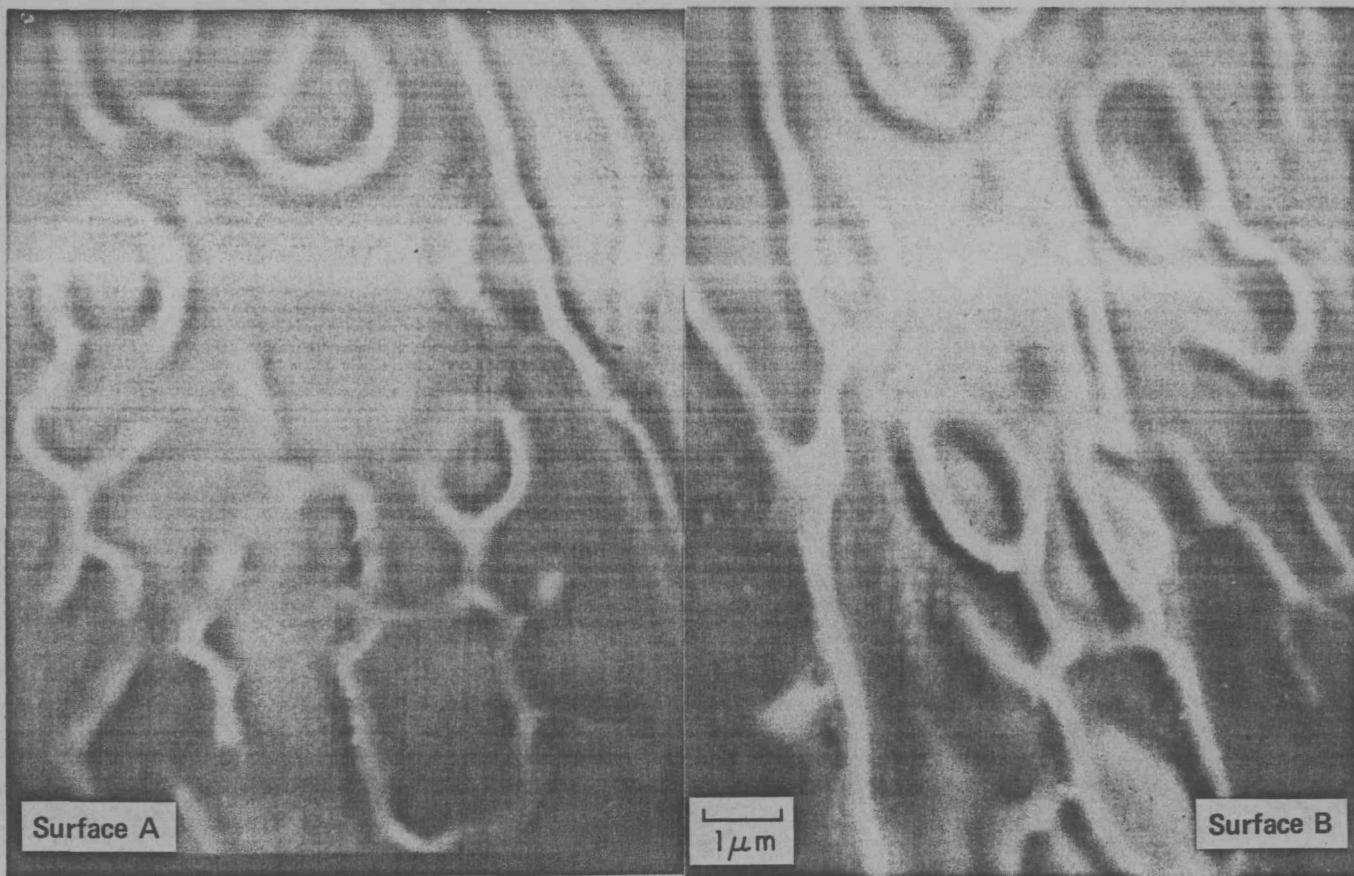

FIG. 5

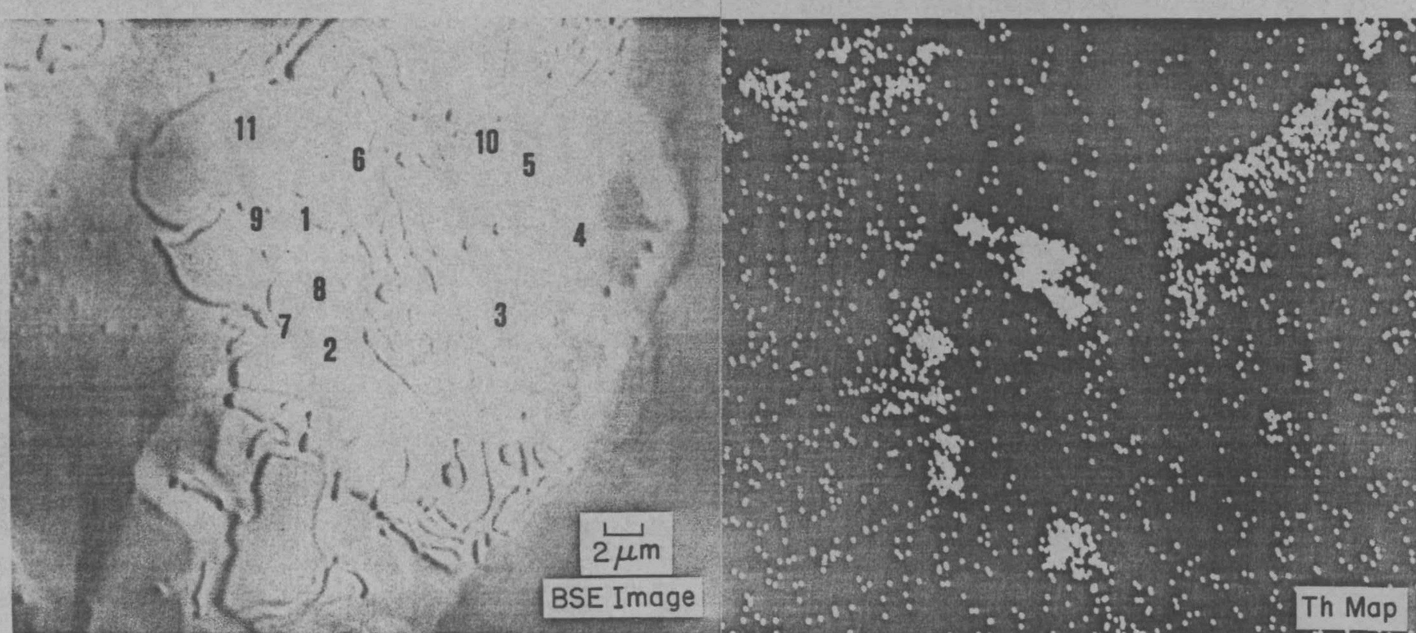

FIG. 6

FIG. 1. Welded DOP-26 Iridium Alloy Capsule.

FIG. 2. Weld-Quench Cracks on Capsule Inner Surface.

FIG. 3. Section Across Quench Showing Crack.

FIG. 4. Extended Ridge Networks on Grain Boundaries in Crack.

FIG. 5. Mating Grain Surfaces in Weld-Quench Crack Exhibit Mirror-Image Ridge Patterns. Depressions Between Ridges Constitute Grain-Boundary Cavities. Submicrometer Particles in Surface Contain Thorium.

FIG. 6. BSE Image and Corresponding Thorium Map Showing Thorium Deposits Outside of a Small Ridge Network. Point Analyses Detected Thorium (up to 5.9 weight %) at locations 1, 3, 5 and 9 Outside Ridge Network. Thorium Concentrations at Locations Within Ridge Network Were Below Detection Limit (~ 0.1 Weight %).