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Impact Properties and Fabricability of Thin-Walled Capsules of Pt-30% Rh-8% W

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CAPSULES OF Pt-30% Rh-8% W

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ABSTRACT

Spherical shells of Pt3008 (Pt-30% Rh-8% W) with a wall thickness of 0.635 mm and a diameter of 40.7 mm were readily fabricated as capsules for 78% theoretically dense ThO₂ spheres. The spherical assemblies, encased in graphite, were impacted against granite at velocities of 76.2 to 89.5 m/sec. Because the ThO₂ was successfully contained by the Pt3008 at impact temperatures of 760, 1000, and 1250°C, the alloy can be seriously considered as the container for radioisotope oxide fuels in heat sources for space power applications.

Uniaxial impact tests of the alloy between 760 and 1350°C and at a comparable velocity show that the ductility decreases with increasing temperature to a value that establishes 1300°C as the permissible upper limit. An apparent impurity-dependent ductility minimum observed in the alloy around 1000 to 1100°C at slow strain rates is not observed in the higher velocity impact tests.

INTRODUCTION

The properties of a family of Pt-Rh-W alloys developed as a potential cladding for radioisotope oxide fuels for space power applications are described elsewhere.^{1,2} A Pt-30 wt % Rh-8 wt % W alloy, designated as Pt3008, was selected for further development because it represented the best compromise between adequate fabricability and high-temperature strength. Recent studies at Los Alamos Scientific Laboratories³ and Donald W. Douglas Laboratories⁴ confirmed that the alloy is compatible

¹C. T. Liu and H. Inouye, "Novel Platinum-Rhodium-Tungsten Alloy," U.S. Patent 3737309 (June 5, 1973).

²H. Inouye, C. T. Liu, and R. G. Donnelly, *New Pt-Rh-W Alloys for Space Isotopic Heat Sources*, ORNL-4813 (September 1972).

³R. D. Baker, Los Alamos Scientific Laboratory, private communication, May 19, 1975.

⁴J. D. Watrous, Donald W. Douglas Laboratories, Richland, Wash., private communication, July 1974.

with $^{238}\text{PuO}_2$ and graphite to at least 1100°C . A critical step in establishing the "technology readiness" of any alloy used as the container for isotopic fuels requires that the material is not ruptured when impacted under the maximum credible accident conditions for any phase of a space mission. The present task was therefore undertaken to demonstrate the impact capability of the alloy and to confirm its fabricability and weldability. Because of the broad spectrum of possible heat source service conditions, a sufficiently wide temperature range for the tests was chosen to establish temperature limits and performance trends. A further objective of these tests was to compare the fabrication of Pt3008 with the iridium cladding used in the Multi Hundred Watt (MHW) heat sources for current space power missions.⁵ And finally it is hoped that the uniaxial tensile impact test⁶ developed at ORNL could be used to predict the outcome of the more expensive and time-consuming systems impact test such as the fuel-sphere-assembly (FSA) test described below.

EXPERIMENTAL

Fabrication

The Pt3008 sheets used to fabricate the test components were prepared from nonconsumable arc melted and drop cast ingots $19 \times 19 \times 51$ mm from virgin melting stock with tungsten added as a rhodium-tungsten master alloy. The drop casting technique was similar to that previously used for the preparation of iridium ingots.⁵ The ingots were rolled initially at 1200°C with temperature decreasing to 950°C for the final hot passes at 0.91 mm thickness, then cold rolled approximately 30% to the final thickness of 0.64 mm. Table 1 lists the chemical analyses of the sheets used to fabricate the capsules for the FSAs and the tensile specimens for the uniaxial tests.

Hemishells 40.7 mm in diameter were formed at ambient temperature by use of a 1.27-mm-thick annealed copper waster sheet. The blanks were stress-relieved 1 hr at 870°C , which is approximately 30°C below the start of recrystallization for the cold-rolled sheet and is the preferred condition, as it allows the forming operation to be conducted in a single draw. Partially or fully recrystallized sheet requires many draws and invariably results in hemishells with cracks along grain boundaries.⁶ To meet the dimensional specifications, the hemishells were recrystallized 1 hr at 1000°C and coined into a copper waster sheet. Twenty one

⁵D. N. Braski and A. C. Schaffhauser, *Production of Ir-0.3% W Discs and Foil*, ORNL-TM-4865 (April 1975).

⁶R. G. Donnelly, *Isotopic Power Materials Annual Progress Report for Period Ending June 30, 1975*, ORNL-TM-5116 (November 1975).

Table 1. Chemical Analyses of Pt3008 Sheets^a
Used in the Impact Tests

Element	Content, wt ppm Except as Noted	
	LCPT-23	LCPT-24
Rh	31.25 wt %	29.76 wt %
W	8.04 wt %	8.06 wt %
Al	10	1
B	1	1
Cr	30	100
Cu	500	150
Fe	50	30
Ir	150	150
Mg	5	1
Mn	100	3
Ni	100	10
P	3	1
Pd	25	25
Si	10	10
Sn	20	20
Ta	20	20
Th	50	20

^aMelted from virgin stock.

hemishells were formed from six sheets, and only one failure resulted. Figure 1 illustrates an example of the preformed and coined hemishell and Fig. 2 the typical microstructure of the alloy recrystallized 1 hr at 1000°C.

We sent 14 hemishells to Mound Laboratory for weld development and assembly of three FSAs. At Mound the hemishells were reannealed 1 hr at 1050°C, coined again, and resized as required for pairing. Then vent holes were electrical-discharge machined into the pole of one hemishell of each pair. After a final cleaning the parts were annealed for 1 hr at 1200°C before welding. The welding parameters were essentially the same as those used for iridium except that the torch gas was 75% He-25% Ar instead of 100% He and the welding current was reduced from 70 to 51 A. Visual, dye penetrant, and radiographic examination of the welds detected no defects. Three as-welded capsules each containing a

Fig. 1. Preformed and Coined Pt3008 Hemishells. Scale is 25 mm.

Fig. 2. Edge Structure of Hemishell Annealed 1 hr at 1000°C and Coined. 150×. Electroetched with $\text{H}_2\text{O}-\text{HCl}-\text{NaCl}$.

78%-theoretically dense ThO_2 ball were shipped to General Electric Co., Space Division, King of Prussia, Pennsylvania, for impact testing.

Impact Testing

The capsules were encased by the General Electric Co. in a 10-mm-thick graphite yarn shell, heated to the impact temperature, and immediately impacted against granite at approximately 85 m/sec. At ORNL tensile specimens were prepared from the ends of the same sheets used to fabricate the capsules and were tested at velocities of 10^{-5} m/sec in an Instron machine and at 86.9 m/sec in an impact gun. The latter tests were conducted at the same temperatures and heating cycle as the FSA tests in order to compare and correlate the separate results. Figure 3 shows the schematic arrangements of the impact tests.

Fig. 3. Schematic Arrangement of Impact Tests.

RESULTS

Table 2 lists the impact parameters and the postimpact results for the capsules prepared from heats LCPT-23 and -24. The average deformation, which ranged between 4.4 and 10.7%, can be correlated with the rotational position of the FSA at the time of impact, the impact velocity, and the impact temperature. Figure 4 shows the shape of a capsule after impact at 760°C and 89.5 m/sec. Evidence of hoop strain and fingerprint-type failures was sought by visual and dye-penetrant inspection but not found. Examination of the capsules impacted at 1000 and 1250°C showed the same results. Figure 5 compares the shapes of the capsules after impact; they differ only in the degree of flattening. Figure 6 shows the sectioned capsules with the ThO₂ still in place. There are no differences in the fragmentation of the ThO₂ that can be attributed to the impact temperature. The appearance of the inside of the capsules is shown in Figs. 7, 8, and 9. The platinum foil weld shield used to facilitate assembly of the capsules appears as the diagonal strip across the sections. No defects

Table 2. Fuel Sphere Assembly Impact Data for Pt3008^a

Identification numbers			
Test	T-1	T-2	T-3
Graphite impact shell	898760-1	903748-2	899004-2
Postimpact containment shell			
Male	23-1	23-3	24-3
Female	23-2	23-4	24-2
Heat of Pt3008	LCPT-23	LCPT-23	LCPT-24
Impact data:			
Date	8/15/75	8/18/75	8/22/75
Temperature, °C	760	1000	1250
Rotation, deg from nominal	65	40	7
Velocity, m/sec	89.46	88.39	76.2
Postimpact results: ^b			
Average diameter, mm	44.86	44.22	42.29
Average height	30.07	29.97	32.59
Average deformation, %	10.7	9.1	4.4

^aData obtained by General Electric Co. Assemblies were not heat-treated before test.

^bCracks were absent by both visual and dye-penetrant examination.

(a)

(b)

Fig. 4. Profile and Face View of Impacted Pt3008 Capsule T-1. $\sim 2\times$. Impact parameters: temperature, 760°C ; rotation, 65° from nominal; velocity, 89.46 m/sec . (a) Side view. (b) Impact face. Photographs supplied by General Electric Company.

Fig. 5. Profiles of Impacted Pt3008 Capsules. $\sim 2\times$. (a) T-1 impacted at 760°C , 65° from nominal, 89.46 m/sec . (b) T-2 impacted at 1000°C , 40° from nominal, 88.39 m/sec . (c) T-3 impacted at 1250°C , 7° from nominal, 76.2 m/sec . Photographs supplied by General Electric Company.

Fig. 6. Impacted Pt3008 Capsules After Sectioning and with Fragmented ThO_2 in Place. $\sim 2\times$. Impacted at (a) 760°C , (b) 1000°C , and (c) 1250°C . Photographs supplied by General Electric Company.

Fig. 7. Hardness and Microstructures (50 $\times$) of Sectioned Pt3008 Capsule Impacted at 89.5 m/sec and 760°C. Numbers around photograph are DPH hardness numbers (1-kg load) at the indicated locations; average is 295.

Fig. 8. Hardness and Microstructures (50×) of Sectioned Pt3008 Capsule Impacted at 88.4 m/sec and 1000°C. Numbers around photograph are DPH hardness numbers (1-kg load) at the indicated locations; average is 289. H₂O-HCl-NaCl electroetch.

Fig. 9. Hardness and Microstructures (50 $\times$) of Sectioned Pt3008 Capsule Impacted at 76.2 m/sec and 1250°C. Numbers around photograph are DPH hardness numbers (1-kg load) at the indicated locations; average is 248. H₂O-HCl-NaCl electroetch.

were found on the inside surfaces. The average hardness around the capsule sections decreases with both the impact temperature and the amount of deformation; however, local hardness values are consistently higher near the hoop strain regions and the region where fingerprint-type failures occur in other materials than on the back and the center of the impact face. Microstructural examination showed no evidence of internal cracks or other defects in any part of the sectioned capsules. The microstructures at the locations indicated in Figs. 7, 8, and 9 were typical. All capsules did, however, show some roughness on the inside surface of the impact face due to a slight amount of penetration of the wall by ThO_2 fragments (see location B in Figs. 7, 8, and 9).

Table 3 lists the uniaxial tensile results, while Figs. 10 and 11 are scanning electron micrographs of the fracture surfaces. These data show that increasing the test velocity from 4×10^{-5} to 86.9 m/sec had no significant effect on the elongation, reduction of area, or fracture mode at 760°C. At 1000°C, however, the increase in test velocity almost doubled the elongation, about tripled the reduction of area, and promoted ductile rupture. The properties degrade with test velocity at 1250°C, as evidenced by a decrease in the elongation from 29% to about 14% and a fracture that is mainly intergranular. The impact properties degrade still further to only 9.9% elongation at 1350°C. In this case the fracture was mainly intergranular.

DISCUSSION

Fabricability

As noted above, our initial attempts to fabricate thin-walled hemishells from Pt3008 blanks produced only one failure out of 21 attempts. The defective hemishell was traceable to a blank known to contain an ultrasonically detected defect. Adequate formability is obtained when the alloy has a fibrous structure obtained by stress relieving. On the basis of the several heats evaluated, this formability appears to be insensitive to the trace impurities (see Table 1). Thus in regard to the microstructural requirements for fabricability, Pt3008 resembles iridium alloys. However, the Pt3008 alloy can be cold rolled to final gage and formed at ambient temperatures, not requiring the hot rolling, surface grinding, and hot forming operation required for iridium. Therefore, the fabrication cost of Pt3008 is expected to be significantly lower than that of iridium.

Impact Properties

The Pt3008 capsules obviously could have survived more severe impact conditions than those used in the current FSA tests. What these limits might be is estimated as follows: If the uniaxial impact data represents the failure curve for the alloy in a FSA impact test, the data for the

Table 3. Effect of Test Velocity and Temperature on the Elongation and Reduction of Area of Pt3008^a

Heat	Test Velocity (m/sec)	Elongation ^b (%)	Reduction of Area (%)
<u>760°C</u>			
LCPT-23	4×10^{-5}	17.0	86.8
LCPT-24	4×10^{-5}	20.0	~100
LCPT-23	86.9	26.0	c
LCPT-24	86.9	20.6	91.2
<u>1000°C</u>			
LCPT-23	4×10^{-5}	10.5	36.2
LCPT-24	4×10^{-5}	10.2	17.5
LCPT-23	86.9	18.8	88.9
<u>1250°C</u>			
LCPT-24	4×10^{-5}	29.2	55.5
LCPT-23	86.9	14.4	74.7
LCPT-24	86.9	14.2	68.9
<u>1350°C</u>			
d	4×10^{-5}	~50	~100
LCPT-23	86.9	9.9	38.8

^aAnnealed 1 hr in vacuum at 1200°C; gage dimensions: 0.635 by 3.18 by 12.70 mm

^bAverage strain between hardness impressions initially spaced 13 mm (0.5 in.) apart.

^cPull rod failed.

^dEstimated values based on other heats; no specimens available from heat LCPT-23 or -24.

Fig. 10. Scanning Electron Micrographs of Fractures of Pt3008 Tensile Specimens Tested at Velocities of (a), (c), and (e): 10^{-5} m/sec and (b), (d), and (f): 86.9 m/sec. 50 $\times$. Test temperatures are (a) and (b), 760°C; (c) and (d), 1000°C; and (e) and (f), 1250°C. Microstructures in (c) and (e) show recrystallization during test.

Fig. 11. Scanning Electron Micrographs of Fractures of Pt3008 Tensile Specimens Tested at Velocities of (a), (c), and (e): 10^{-5} m/sec and (b), (d), and (f): 89.6 m/sec. 300 $\times$. Test temperatures are (a) and (b), 760°C; (c) and (d), 1000°C; and (e) and (f), 1250°C. Microstructures in (c) and (e) show recrystallization during test. Dashed lines in (a), (b), and (d) outline boundaries of fracture surfaces.

amount of deformation sustained by the capsules must lie below the failure limit curve for the uniaxial tests, as shown in Fig. 12, because no capsules ruptured. Although Fig. 12 shows that the capsule deformation curve nearly parallels the uniaxial test curve, this is considered to be an uncorrelated result caused by the lack of control over the impact conditions. For example, numerous tests have clearly shown that the deformation of an iridium alloy capsule increases with both the rotation and the velocity of the FSA impact. (Therefore the effect of the impact temperature on deformation could not be uniquely determined by these tests.) Thus on the basis of an expected maximum deformation of about 12%, one obtains 1300°C as the estimated upper limit for impact survival at a velocity around 87 m/sec. The lower temperature limit for impact survival is further estimated to be considerably lower than 760°C because the uniaxial test data show that the ductility increases as the impact temperature decreases.

Fig. 12. Plot of Elongation of Pt3008 as a Function of Impact Temperature. Scatter band represents effect of rotation and impact velocity on deformation of FSA.

Further analyses of the FSA impact data show that (1) the strain distribution based on the hardness values around an impacted capsule is nonuniform and is in general highest in those regions where ruptures have occurred in iridium; (2) the similarity in the hardness of the center of the impact faces with the back suggests that little or no deformation occurs at the center of the impact face; (3) the average hardness of the capsules increases with the amount of deformation, but is also consistent with a recovery process that could have occurred on cooling from the impact temperatures; (4) although the welds in Figs. 7, 8, and 9 were located in the hoop strain region where the impact conditions are the most severe, the lack of any evidence of weakness indicates

that the impact properties of Pt3008 are not greatly influenced by the grain structure (i.e., the weld) or by the grain size (heat-affected zone).

An important characteristic of Pt3008 that is relevant to its impact properties at velocities under 89.5 m/sec is shown by the data in Table 3. At a rate of 4×10^{-5} m/sec the ductility degrades with temperature to a minimum value at 1000°C, but this ductility minimum is not observed at 89.5 m/sec. An increase in strain rate also increases the reduction of area at fracture and promotes ductile rupture (see Figs. 10 and 11). A prior study⁷ shows that the toughness of Pt3008 improves significantly upon increasing the strain rate from a value of $\sim 10^{-5}$ m/sec to about 2 m/sec in the temperature range 700 to 1000°C. This implies a strong dependence of the ductility (and toughness) on the strain rate between 4×10^{-5} and 2 m/sec but not between 2 and 89.5 m/sec. From a systems standpoint this means that the alloy could be used for containers over a large range of impact velocities. Furthermore if the slow strain rate ductility minimum and intergranular fracture are due to the segregation of impurities (such as boron⁸ and phosphorous⁹) at grain boundaries, it follows that these impurities have no effect on the impact properties of the alloy between 760 and 1250°C.

CONCLUSIONS AND RECOMMENDATIONS

(1) Alloy Pt3008 can be seriously considered as a fuel container in isotopic heat sources for impact temperatures up to 1300°C. Additional tests under more severe impact conditions and outside the temperature range presently investigated are recommended to establish the limits of capability for the material.

(2) Because no cladding deficiency or abnormal characteristics were noted in Pt3008 starting from a casting to the final impacted capsules, composition or fabrication modifications are not required for impact resistance.

(3) An FSA impact test provides a much lower return of information on metallurgical properties of the cladding than a less expensive and simpler uniaxial impact test; however, these tests could complement each other if the FSA test conditions could be more precisely controlled.

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⁷J. D. Watrous, Donald W. Douglas Laboratories, Richland, Wash., private communication, July 1974.

⁸R. G. Donnelly, *Isotopic Power Materials Annual Progress Report for Period Ending June 30, 1975*, ORNL-TM-5116 (November 1975).

⁹D. L. Rohr and S. S. Hecker, *Fracture Behavior of a Pt-30% Rh-8% W Alloy Between Room Temperature and 1300°C*, LA-UR-75-1189, July 1975.

the personnel at Mound Research Laboratories for conditioning the hemishells and welding the capsules, and the General Electric Co., Space Division, King of Prussia, Pa., for assembling, impacting, and recording the FSA test results.

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