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**Development Testing of the
Two-Watt RTG Heat Source and
Hastelloy-S/T-111 Alloy
Compatibility Studies**

Edwin I. Howell and Paul E. Teaney

September 29, 1993

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I. Development Testing of the Two-Watt RTG Heat Source

E. I. Howell

Abstract

The two-watt radioisotope thermoelectric generator heat source capsules were tested to determine their survivability under extreme environmental conditions: high external pressure, high impact, and high internal pressure. Test results showed that the capsules could withstand external pressures of 1000 bars and impacts at velocities near 150 meters per second. However, the results of the internal pressure tests (stress-rupture) were not so favorable, possibly because of copper contamination, leading to a recommendation for additional testing.

Introduction

The objective of the two-watt radioisotope thermoelectric generator (RTG) heat source capsule development testing was to select the most detrimental tests, based on historical data from previous heat source designs, to which the capsules could be environmentally subjected [1]. The tests selected were: 1) external hydrostatic pressure tests of 1000 bars (14,504 psi) to determine capsule survivability if subjected to deep sea pressure, 2) impact tests up to 150 meters per second to determine capsule survivability in the event of a transportation accident, and 3) internal pressure tests at various temperatures and pressures to determine capsule survivability in the event of a fire.

Heat Source Design Overview

The heat source for the two-watt RTG consists of a three-layered, nonvented capsule (Figure I-1) that triply encapsulates a thermal inventory of up to

75 watts produced from the decay of the 87.74-year half-life plutonium-238 fuel [2]. Yttrium, in the form of chips, is added to the plutonium dioxide ($^{238}\text{PuO}_2$) shards to getter oxygen. The gettering is accelerated by heating the capsules for one hour at 1623 K (1350°C), reducing the stoichiometric $^{238}\text{PuO}_2$ to substoichiometric $^{238}\text{PuO}_{2-x}$, and thus lowering the oxygen partial pressure. A lower oxygen partial pressure reduces the susceptibility of the T-111 refractory alloy inner layer, called the liner, to interaction with the fuel [3]. The T-111 refractory alloy middle layer, called the strength member, provides for internal pressure containment of the helium gas generated during the decay of the plutonium-238. The Hastelloy-S superalloy outer layer, called the clad, provides an environmental barrier between the strength member and terrestrial environments (that is, sea water or air). A list of the drawings of the components and assemblies is provided in Table I-1.

Table I-1 - A LISTING OF MOUND DRAWING NUMBERS FOR TWO-WATT HEAT SOURCE COMPONENTS AND ASSEMBLIES

<u>MD Drawing Number</u>	<u>Description</u>
AYD891409	Liner Shim
AYD891410	Liner Cap
AYD891411	Liner Body
AYD891412	Liner Assembly
AYD891413	Strength Member Body
AYD891414	Strength Member Cap
AYD891415	Strength Member Assembly
AYD891416	Clad Body
AYD891417	Clad Cap
AYD891418	Heat Source Assembly
(Terrestrial Three-Layer Capsule)

Figure I-1 - Two-watt RTG heat source. (Weld joint details shown in encircled enlargements.)

Capsule Testing

Hydrostatic Pressure

Two capsules, HYD-1 and HYD-2, were configured for this test. Both of these capsules were two-layered, consisting of a liner and strength member, with tungsten-molybdenum powder being used as a simulated fuel. After passing all nondestructive testing requirements throughout the manufacturing processes, both capsules were subjected to an external hydrostatic pressure of approximately 1000 bars (15,000 psig) for 30 minutes. The first capsule collapsed at an external pressure of approximately 960 bars (14,100 psig), and the second capsule collapsed at

approximately 885 bars (13,000 psig). The external appearance of the first test capsule is shown in Figure I-2. The second capsule appeared similar, but also collapsed from the opposite side. Subsequently, both capsules underwent postmortem testing and evaluation. Even though the capsules collapsed at a pressure lower than 1000 bars, the post-test leak rates for the first and second capsules were less than 1.9×10^{-9} atm-cc/s helium and less than 4.4×10^{-9} atm-cc/s helium, respectively.

Figure I-2 - The collapsed condition of capsule HYD-1 after exposure to an external hydrostatic pressure of 1000 bars.

Figure I-3 is a cross-sectional view of the weld zone of the second capsule showing the area of maximum distortion. The normal appearance of the weld zone is shown in Figure I-4 for comparison. No cracks were observed in the collapsed area of the capsule; however, folds in the material were evident on the cap and body sides of the weld underbead. It was concluded from these results that the capsules survived the hydrostatic pressure test.

Figure I-3 - Cross section showing the area of maximum distortion in capsule HYD-2 after exposure to external hydrostatic pressure of 1000 bars.

Figure I-4 - View of opposite side of capsule in Figure II-3 showing less distorted weld zone.

Impact

A gas gun was used to impact six capsules against a nonyielding steel plate at velocities ranging from 75 to 150 m/s. The capsules were preheated to 873 K (600°C) prior to impact and all but the initial capsule contained a clad. These impact capsules contained the same simulated fuel that was used in the hydrostatic test capsules. All but the last capsule was impacted at an attitude of 45° to the cap face. The last capsule was impacted at an attitude of 90° to the cap face (side-on).

Test conditions and results for all six capsules are summarized in Table I-2. Pre- and post-test views of the stress-rupture test assembly are shown in Figures I-5 and I-6, respectively. The clads were breached on the three capsules that were impacted at velocities above 144 m/s, and, of the two capsules that were impacted at approximately 150 m/s, one strength member had a small crack that went through the weld in the heat-affected zone (Figure I-7)

Figure I-5 - A side-on view of test capsule Impact-4 prior to impact.

Figure I-6 - A side-on view of the capsule in Figure II-5 after impact (45° to cap face at 149.4 m/s and 873 K).

Table I-2 - SUMMARY OF IMPACT TEST RESULTS FOR TWO-WATT DEVELOPMENT TESTS

Capsule Serial <u>Number</u>	Velocity <u>(m/s)</u>	Attitude <u>(degrees)</u>	Breached <u>Clad?</u>	Breached Strength <u>Member?</u>
Impact-1	75.5	45° to Cap Face	N/A	No
Impact-2	117.7	45° to Cap Face	No	No
Impact-3	144.0	45° to Cap Face	No	No
Impact-4	149.4	45° to Cap Face	Yes	Yes
Impact-6	149.9	45° to Cap Face	Yes	No
Impact-7	145.4	90° to Cap Face (side-on)	Yes	No

Figure I-7 - Crack in the heat-affected zone along the body side of the strength member weld.

Stress-Rupture

Six stress-rupture capsules were tested at 1444 K (1171°C), with internal pressures of helium ranging from approximately 24 MPa (3,500 psi) to 41 MPa (6,000 psi). All six capsules used a tantalum pellet to simulate the liner assembly. The pellet displaced the same volume that the fuel, getter, and liner components inside the strength member would displace, thus obtaining the same void volume within the strength member of the stress-rupture capsules. A heavy-walled tantalum tube was welded to a through hole in the strength member cap as a means for pressurizing and supporting the capsules in the furnace chamber.

The stress-rupture test matrix and test results are summarized in Tables I-3 and I-4, respectively. All the capsules failed earlier than predicted by previous heat source data [4]. Auger analysis of the fracture

Table I-3 - MATRIX FOR STRESS-RUPTURE TESTS

Test Pressure (MPa)	Equivalent Stress (MPa)	Temperature of Test (K)	Predicted Test Duration (hr)
69	486	1277	10.3
59	414	1277	24.2
49	345	1277	63.4
49	345	1444	3.1
44	310	1444	5.4
39	276	1444	10.1
29	207	1444	45.8
24	172	1444	119.9

Table I-4 - RESULTS OF STRESS-RUPTURE TESTS

Capsule Number	Test Pressure (MPa)	Temperature (K)	Duration of Test (hr)
PB-01	24	1444	2.87
PB-02	30	1444	0.61
PB-03 ^a	39	1444	1.09
PB-04 ^a	Unable to stabilize pressure		
PB-05 ^a	41	1555	0.19
PB-06	33	1444	0.42
PB-07	37	1444	0.15

^a Results invalid because of abnormalities in tests.

surfaces revealed the presence of copper, which may have contributed to the early failures. Pre- and post-test views of the stress-rupture test assembly are shown in Figures I-8 and I-9, respectively. All of the capsules failed in the cap-to-body weld (Figure I-9).

Figure I-8 - View of the stress-rupture capsule prior to test.

Figure I-9 - View of the failed stress-rupture capsule after test.

Metallographic and fractographic analysis of the capsules revealed failures on the cap side of the cap-to-body welds (Figure I-10). Scanning electron microscopic evaluation of the fracture surfaces indicated a primarily intergranular, ductile rupture, with some evidence of transgranular fracture during the final phase of the failure. Weld grains were elongated (Figure I-11). Fine dimples, indicative of ductile failure, existed along the grain boundary interfaces (Figure I-12). These dimples contained small precipitates, which facilitated the intergranular rupture (Figure I-13). Energy dispersive x-ray microanalysis indicated that the precipitates were predominately hafnium (presumably HfO_2).

Figure I-10 - Photomicrograph (25X) showing failure along cap side of weld.

Figure I-11 - SEM micrograph showing intergranular/ductile rupture, with evidence of transgranular failure near final phase of failure.

Figure I-12 - Fine dimples indicative of ductile failure along the grain boundary interfaces.

Figure I-13 - The fine dimples contained small hafnium-enriched precipitates (presumably HfO_2).

Conclusions

The results from the external hydrostatic tests and impact tests confirm that capsules with this design could survive impacts at velocities near 150 m/s and deep sea pressures of 1000 bars.

The results of the stress-rupture tests were not as favorable. All the stress-rupture test capsules failed earlier than predicted. It is believed that copper contamination from the wire used on the electrical discharge machining (EDM) to cut out blanks from T-111 plate metal weakened or attacked the grain boundaries during the closure weld and contributed to these early failures.

Recommendations for Further Safety/ Development Testing

Because of the large difference between the expected results and the results obtained on these test samples, some limited additional testing should be done on samples from blanks not contaminated by copper in order to determine whether the results are attributable to the capsule design or to a weakening of the grain boundaries of the weld by copper.

Acknowledgments

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II. Hastelloy-S/T-111 Alloy Compatibility Studies

P. E. Teaney

Abstract

A material compatibility study examined the use of Hastelloy-S as a material to clad the tantalum strength member of the two-watt radioisotopic heat source. Test capsules were subjected to high temperatures for various lengths of time, then cross sectioned and examined with a scanning electron microscope. Results of the study indicate that Hastelloy-S would be compatible with the underlying alloy, not only at the normal operating temperatures of the heat source, but also when exposed to the much higher temperatures of a credible accident scenario.

Introduction

The material that is used to clad the two-watt heat source must be compatible with the underlying T-111 alloy strength member. Hastelloy-S, the material of interest for this study, was therefore compared to two others whose compatibility with T-111 was already known, Hastelloy-C and Inconel-600.

Oxidation-resistant Hastelloy-C is the material used to clad the small radioisotopic heat source upon which the proposed design of the two-watt radioisotopic heat source is based. Previous compatibility studies [5] indicated that Inconel-600 alloy would also be a satisfactory compatible material for that application. However, because of potential special applications for the two-watt heat source, Hastelloy-S was considered to have the best material characteristics for the proposed operating conditions and was, therefore, selected to clad the two-watt heat source.

The constituents of Hastelloy-S are similar to those of Hastelloy-C, Inconel-600, and other super alloys for which limited diffusion data were available. These data indicated that the materials could be expected to be compatible at the proposed operating temperature of the heat source in the Radioisotopic Thermoelectric Generator (RTG). In order to verify the expected compatibility of the materials and obtain rates of reaction at various potential operating temperatures, a compatibility study was conducted.

Also, because the $^{238}\text{PuO}_2$ fuel within the heat source is highly toxic, safety considerations necessitate complete containment of the radioactive fuel in any credible accident environment. Therefore, it is important to know the reaction kinetics between the Hastelloy-S and the T-111 strength member in the event that the heat source were to experience an unplanned elevated temperature excursion.

This Hastelloy-S/T-111 alloy compatibility study was patterned after the Inconel-600/T-111 alloy compatibility study referenced above. The capsule design was the same except that in this study, so that the tests could be fabricated from materials on hand, a tantalum (10 wt% tungsten) alloy capsule was used instead of T-111 (90 wt% tantalum, 8 wt% tungsten, 2 wt% hafnium) to contain the compatibility disks. Because the reaction measurements were made at the interfaces of the compatibility disks near the middle of the cross-sectioned capsule, the substitution of this chemically similar container material would not be expected to influence the reaction. Also, the test matrix in this study was modified to include testing at 1173 K (900°C) in order to test closer to the proposed operating temperature of the RTG. Testing below 1173 K (900°C) was not considered practical because of the longer test time periods that would be required to obtain measurable reaction zones.

The nominal chemical compositions of Inconel-600, Hastelloy-C, and Hastelloy-S are compared in Table II-1. Molybdenum is contained in Hastelloy-S and Hastelloy-C, but is not present in Inconel-600. Hastelloy-S

**Table II-1 - COMPARISON OF CHEMICAL COMPOSITIONS
OF INCONEL-600, HASTELLOY-C AND HASTELLOY-S**

The alloys share most constituents in common,
with some differences in compositions.

<u>Element</u>	<u>Inconel 600^{ab}</u>	<u>Hastelloy C^{ab}</u>	<u>Hastelloy S^{bc}</u>
Ni	72.0 (min.)	50.0 (min.)	68.0 (min.)
Cr	14.0 - 17.0	14.5 - 16.5	14.5 - 17.0
Mo	-----	15.0 - 17.0	14.0 - 16.5
Fe	6.0 - 10.0	4.0 - 7.0	3.0 (max.)
Mn	1.0 (max.)	1.0 (max.)	0.3 - 1.0
Si	0.5 (max.)	1.0 (max.)	0.2 - 0.75
S	0.015 (max.)	0.03 (max.)	-----
C	0.15 (max.)	0.08 (max.)	0.02 (max.)
Cu	0.50 (max.)	-----	-----
W	-----	3.0 - 4.5	-----
Co	-----	2.5 (min.)	2.0 (max.)
V	-----	0.35 (max.)	-----
P	-----	0.04 (max.)	-----
Al	-----	-----	0.10 - 0.50
B	-----	-----	0.015 (max.)
La	-----	-----	0.01 - 0.10

^a Aerospace Structural Metals Handbook AFML-TR-68-115

^b In weight percent

^c Metals Handbook, Ninth Edition, Vol. 3

contains small amounts of cobalt, aluminum, boron, and lanthanum which are not present in Inconel-600, and Hastelloy-C contains tungsten, cobalt, vanadium, and phosphorus which are not present in Inconel-600.

Inconel-600 contains a small amount of copper, which is not present in Hastelloy-S or Hastelloy-C, and a minor amount of sulfur which is not present in Hastelloy-S. However, the alloys have most constituents in common, with some differences in composition. Because all three materials are nickel base alloys, similar compatibility behavior was expected.

However, the differences in composition were of sufficient magnitude to require verification of these expectations through compatibility testing in order to prove the safety of the heat source design.

Test Capsule Configuration

Differences in the thermal expansion coefficients of the two materials indicated that contact at elevated temperatures would be enhanced by enclosing the Hastelloy-S in the tantalum alloy. The configuration of the test capsule is shown in Figure II-1. The compatibility disks were lapped flat to a 5-7 RMS (R_a 3 μ in. or 0.15 μ m) finish, cleaned with solvent and acid following procedures appropriate for each material, and pressed in place with an arbor press. The top and bottom disks were then electron beam welded in place to ensure that intimate contact was maintained during testing. The specimens were subsequently radiographed to be certain that only specimens with good contact were used for testing. After fabrication, the specimens were tested according to the matrix in Table II-2.

Results

After testing, the samples were removed from the outer containment, cross sectioned, and examined metallographically and with the scanning electron microscope. Examination of the cross sections revealed reaction zones that appeared to be influenced by time and temperature. Observations were consistent with those in the previous study with Inconel-600. Although diffusion occurred in both directions, the diffusion of the Hastelloy-S

Figure II-1 - The T-111/Hastelloy-S test capsule was designed to ensure intimate contact throughout the test.

Table II-2 - COMPATIBILITY TEST MATRIX

<u>Temperature (°C)</u>		<u>Time (hr)</u>	
1225	1	4	16
1100	10	40	160
1000	100	400	1600
900	1000	4000	16000

components into the T-111 alloy predominated. Also, some Kirkendall-type voids were observed along the Hastelloy-S side of the reaction zone, but the voids were not as extensive as those observed in the Inconel-600/T-111 alloy study.

The reaction zone of the specimen that was tested at 1273 K (1000°C) for 400 hours is shown in Figure II-2, along with results obtained by energy dispersive x-ray analysis at various locations in the sample. The remainder of the specimens had similar reaction zones. However, either coalescence of voids or shrinkage upon cooling after testing, resulted in the formation of a gap along the reaction zone/Hastelloy-S interface in several of the longer term tests, making measurement of the reaction zone width in those specimens difficult.

Figure II-2 - Linear measurements of the reaction zones in all specimens were made from electron backscatter images from the scanning electron microscope (SEM).

① T-111 with trace of Ni	⑤ Ni and Mo, low in Ta
② T-111 with trace of Ni	⑥ Cr, Ni, Mo, Fe in Ta
③ High concentration of Ni in Ta	⑦ Mo, Cr, Ni, Fe low in Ta
④ Cr and Mo within Ta/Ni matrix	⑧ Mo, Cr, Ni Fe with trace of Ta

The widths of the reaction zones were determined using linear measurements of back scatter electron images from the scanning electron microscope. The results of these measurements are shown in Table II-3. These data were fitted to the parabolic rate equation $(\Delta X)^2 = 2kt + C$, where ΔX is the width of the reaction zone in μm , t is the test time in hours, and k and C are experimentally determined constants. The resulting rate curves for 1173 K (900°C), 1273 K (1000°C), 1373 K (1100°C), and 1498 K (1225°C) are shown in Figures II-3, II-4, II-5, and II-6, respectively.

Table II-3 - REACTION ZONE MEASUREMENTS FROM ELECTRON BACKSCATTER IMAGES FROM THE SEM			
<u>Temperature (°C)</u>	<u>Width of Reaction Zones in μm at Various Times</u>		
	<u>1 hr</u>	<u>4 hr</u>	<u>16 hr</u>
1225	23.2	38.5	65.9
	<u>10 hr</u>	<u>40 hr</u>	<u>160 hr</u>
1100	27.0	41.1	63.2
	<u>100 hr</u>	<u>400 hr</u>	<u>1600 hr</u>
1000	25.1	43.2	80.3
	<u>1000 hr</u>	<u>4000 hr</u>	<u>16000 hr</u>
900	27.0	48.0	96.9

The natural logs of the reaction rates for each temperature were then plotted as a function of $1/T$ (Figure II-7) for determination of the activation energy for the reaction. This energy was determined to be 65.7 kcal/mol (275 kJ/mol). This value is slightly lower than the activation energy of 72.6 kcal/mol determined for the Inconel-600/T-111 alloy reaction in the previous study. However, differences in the composition of the alloys make exact correlation of these data unlikely.

Figure II-3 - Reaction rate curve obtained for 900°C.

X-ray energy dispersive spectrometric analyses of the specimens in this study showed the preferential diffusion of nickel into the T-111 alloy, followed by molybdenum and chromium. Although iron was detected within the reaction zone, it appeared to be preceded by the three major constituents in the alloy. The previous study with Inconel-600 reported the preferential diffusion of nickel into the tantalum, followed by iron.

The activation energies for the diffusion of several major super-alloy constituents in tantalum and niobium are given in Table II-4 for comparison. The activation energy given for the diffusion of nickel in niobium is 63.1 kcal/mol. Because niobium and tantalum are chemically similar, and nickel was observed to be the primary diffusing constituent in the T-111 alloy/Hastelloy-S reaction, the value obtained from this study would be

Table II-4 - ACTIVATION ENERGIES OF SEVERAL MAJOR SUPERALLOY COMPONENTS IN TANTALUM AND NIOBIUM^a

<u>Component</u>	<u>Value for Diffusion in Ta^b</u>	<u>Value for Diffusion in Nb^b</u>
Fe	71.4	77.7±1.2/70.3±1.4
Cr	----	83.5±1.8
Ni	----	63.1±0.8
Mo	81.0	-----
Co	----	61.43±2.06/65.6±1.2

^a E. A. Brandes, Smithells Metals Reference Book, Sixth Edition.

^b in kcal/mol (1 kcal/mole $\approx$ 4.184 kJ/mol)

Figure II-4 - Reaction rate curve obtained for 1000°C.

Figure II-5 - Reaction rate curve obtained for 1100°C.

expected to be near that for the diffusion of nickel in niobium. These published data, as well as the results of the previous study, support the value for the activation energy determined from Figure II-7. Assuming that the same mechanism is the rate controlling one at temperatures below 1173 K (900°C), these data indicate that the width of the reaction zone would be 1.6 μm (0.06 mils) after ten years at 873 K (600°C).

Discussion

The reaction rate obtained in this study is for compatibility couples designed, pressed, and electron beam welded in place for maximum contact and reflects a worst-case condition that would not likely occur in a normal heat source design. In addition, the projected rate assumes conservatively that there is no change in the reaction mechanism below 1173 K (900°C).

A considerable number of thermally aged heat sources containing T-111 alloy in Hastelloy-C have been evaluated at Mound. Application of the

Figure II-6 - Reaction rate curve obtained for 1225°C.

results of this study to that design suggests that an interaction up to 101 μm (4 mils) would occur after 2 years at 1173 K (900°C). However, no Hastelloy-C/T-111 alloy reactions were observed for specimens tested for up to two years at 1173 K (900°C). There is no vapor phase reaction occurring at this temperature and there is uneven ("point") contact between the strength member and clad, thus inhibiting solid state diffusion.

The results of this study, experience with previous Hastelloy-C/T-111 alloy heat source designs, and reference data for similar materials, indicate that the proposed clad and strength member of the two-watt heat source would be compatible at its proposed operating temperature in the Radioisotopic

Figure II-7 - Rate projected to 600°C (1.6 μ m or 0.063 mils after 10 yr).

Thermoelectric Generator (RTG). In addition to this operating temperature requirement, one credible accident scenario requires that the heat source must survive a transportation accident in which the RTG is exposed to a 1073 K (800°C) fire for one-half hour. These data indicate that clad/strength-member compatibility exceeds this requirement up to the maximum temperature included in the test matrix.

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