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MASTER

LARGE RADIOISOTOPE HEAT SOURCE

CAPSULE PROGRAM

Topical Report No. 5

Compatibility Testing

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION

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By

E. BENN

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ABSTRACT

The Large Radioisotope Heat Source Capsule (LRHSC) Program supports the development of high temperature (2000°F) long life (5 years) radioisotope heat sources. The specific concern of the LRHSC program is to develop the tantalum alloy fuel containing capsule. The LRHSC test program is generating data on biaxial creep, impact deformation, burial temperature distributions and materials compatibility.

This Topical Report presents the progress made in the design, testing, and evaluation of the LRHSC materials compatibility samples during fiscal year 1968. Test data are presented on interface combinations involving Ta-10W, T-111 (Ta-8W-2Hf), Al_2O_3 , Pt-10Rh and iron titanate at temperatures in the range of 2000°F to 2500°F.

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I. INTRODUCTION

The Large Radioisotope Heat Source Capsule (LRHSC) is shown in Figure 1. The capsule consists of a radioisotope fuel annulus, tantalum base alloy structural members, and a platinum alloy outer clad. The tantalum alloys were selected because of their high temperature ($>2000^{\circ}\text{F}$) strength and their fabricability. The platinum clad protects the tantalum members from high temperature oxidation. It has been established that tantalum and platinum are incompatible at 2000°F ; therefore, an alumina coating is placed between the tantalum and the platinum to prevent interdiffusion and reaction of the two materials. The iron titanate high emissivity coating is required for heat transfer by radiation. The platinum has a low emissivity and would hamper the performance characteristics of the capsule. Thus, four distinct classes of material are required, each serving a specific function. It is necessary for all of these materials to be compatible for reasons of structural integrity in all phases of assembly, launch, operation, reentry and impact of the capsule. To investigate the compatibility of these materials a coupon compatibility task is included in the LRHSC program.

The specific objectives of the coupon compatibility task are to identify any interactions that occur between those capsule materials that will be in contact with each other, to measure the rates of interaction as a function of time and temperature and to determine the chemical and structural effects the interactions have on the base material properties.

The compatibility testing consisted of placing smooth surfaces of the materials in intimate contact and exposing them to high temperatures for fixed times in a vacuum. Details of the design of the coupon test stacks and test conditions are described in Section II.

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Figure 1. LRHS Capsule Conceptual Design

Eight coupon stacks were tested in GFY 1968; however, due to program funding limitations only preliminary evaluations of the test results were possible. The test data which have been accumulated to date and the preliminary conclusions from these data are discussed in Sections III and IV respectively. All of the test samples are currently being stored and it is anticipated that the more detailed analysis, such as those required to establish rates of reaction, will be accomplished when sufficient funding is available.

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II. COMPATIBILITY TESTING

A. TEST DESIGN

The compatibility test stacks were comprised of a series or sandwich of thin discs (coupons). This configuration allows a number of material interfaces to be investigated simultaneously after being exposed to the same test parameters.

The tests were conducted on coupons which were 1.00 inches in diameter and 20 to 50 mils thick. The tantalum alloys investigated were T-111 (Tantalum-8W-2.5Hf) and Ta-10W. The platinum alloy was Pt-10Rh. Two alumina coatings were investigated - chemically vapor deposited α alumina and arc plasma sprayed alumina. The iron titanate emissivity coating had the composition $\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$. The T-111 and Pt-10Rh discs which were coated by plasma spraying were first sand blasted. A view of the test coupons prior to coating is shown in Figure 2. All of the thick (0.050 inch) T-111 discs were fabricated from 1.00 inch diameter by 0.100 inch thick blanks which were full penetration electron beam welded on a 0.30 inch diameter circle as shown in Figure 3. The purpose of this weld was to allow observation of any differences in chemical stability between the as fabricated and the welded structure.

The coupon discs were machined with a slight edge taper beginning at the 0.70 inch diameter. The taper provided separation between the edges of the coupons in the test stack and, therefore, allowed identification of vapor phase transport reactions. The various test coupons were fabricated with the same materials and process specifications used for the LRHSC itself. The discs were arranged in a stack formation for testing. Figure 4 shows the location in the test stack of each of the Ta-10W, T-111 and Pt-10Rh coupons. Also illustrated in Figure 4 are the coatings applied to each coupon and the resultant interfaces on which compatibility information was derived.

Figure 2. Typical Compatibility Test Coupons -
All Uncoated

Figure 3. T-111 Blank with Full Penetration Electron Beam Weld

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Figure 4. Arrangement of Coupon Discs in Compatibility Test Stack

The coupon stack was installed into a tantalum holder as illustrated in Figure 5. The holder design assured that the coupon discs were in contact only with each other with no interference with foreign materials. Alumina discs were used to separate the coupons from the holder. The holder had two 1/16 inch diameter holes to allow evacuation. Figures 6, 7, and 8 show an array of coupons before and during loading into the coupon stack holder.

The chemically vapor deposited (CVD) α - alumina coatings were applied at San Fernando Laboratories and the arc plasma spray alumina coatings were applied at Microcraft Incorporated.¹ The tungsten flash applied to one T-111 coupon was applied by CVD at Atomics International. The iron titanate coatings were applied at Atomics International by conventional APS processes.

B. TESTING APPARATUS AND SET-UP

After the coupons were loaded into the holder, the top lid was inserted and torqued down to 25 inch pounds to insure good contact. The coupon test capsule was instrumented with three Pt-Rh thermocouples and loaded into the vacuum testing facility on a tantalum tripod as shown in Figure 9. The tripod is supported by the bottom shields of a tantalum radiation furnace which rests on a stainless steel platform inside of the vacuum chamber.² After a pyrometric black body was spot welded to the top of the stack, the heater and reflector shields were installed as shown in Figure 10. The entire vacuum furnace system prior to assembly and bolting down is shown in Figure 11.

¹ AI-AEC-12643, LRHSC Program Topical Report No. 3, "Ceramic Coating Process Development and Evaluation," by A. K. Smalley, March 1968.

² "Design, Fabrication and Operation of a High Temperature, High Vacuum Biaxial Creep System," by E. Benn, M. A. Fallandy, and P. B. Ferry, 1968 Vacuum Metallurgy Conference, June 10-13, 1968.

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Figure 5. LRHSC Coupon Compatibility Test Assembly

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Figure 6. Compatibility Test Holder
Surrounded by the 14 Coupon Discs
to be Tested

Figure 7. Coupon Test Holder and
Coupon Stack

Figure 8. Coupon Stack Inserted into
Loading Tool so that Coupon Test Holder
can be Placed Over the Tool

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Figure 9. Coupon Test Assembly on a Tripod Support which Rests on the Bottom of the Furnace

Figure 10. Coupon Test Assembly in the Heater

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Figure 11. Compatibility Test Facility

C. OPERATION AND SCHEDULE OF TESTS

Following bolt down of the vacuum system bell jar on a copper O-ring, liquid nitrogen sorption pumps were used to evacuate the chamber. During operation of the sorption pumps the bell jar and ion pump were baked out at 400°F. After the 10 micron pressure range was reached, the 200 l/sec ion pump was activated and evacuation continued to the 10^{-8} torr range. At that time the heater was slowly brought up to the testing temperature. The bell jar cooling water was turned on when the test capsule reached 1000°F.

Accurate long term temperature control was obtained by using a highly sensitive optical pyrometer which sighted down through the top window of the bell jar at the pyrometric black body welded to the coupon stack. The associated electronic readout system recorded on an instrument which displayed 3°F full scale. The central stack thermocouple reading was taken as the initial temperature standard upon which the optical pyrometer was calibrated. All subsequent thermocouple readings were subject to drift check by the calibrated pyrometer. Temperature control was maintained by using the central stack thermocouple and manual variable resistors connected to a silicon rectifier controller. For most of the tests performed, the furnace measured about 1 M Ω to ground and operated in the 90-120 volts and 11-15 amps range. The control accuracy of the furnaces was nominally $\pm 5^\circ\text{F}$. The complete test ensemble with all of the instrumentation used is shown in Figure 12.

The schedule of LRHSC compatibility tests is shown in Table I. The base operating temperature being considered for the heat source is 2000°F over a five year period. The 2250 and 2500°F tests were designed to accelerate exposure conditions and give indications of the effects of temperature spikes on the capsule components. As stated in Table I, coupon stacks 10, 11 and 12 are to be tested with creep test capsules in the LRHSC creep testing programs. These tests will allow comparison of compatibility at the various interfaces for stressed and unstressed conditions. The testing matrix (time vs. temperature) was designed to give data which would allow extrapolated estimates of the extent of any chemical attack observed between test coupons.

Figure 12. Coupon Test Assembly on a Tripod Support which rests on the Bottom of the Furnace

TABLE I
SCHEDULE OF COUPON COMPATIBILITY TESTING

Coupon Stack No.	Test Start Date	Temp. (°F)	Atm. Torr	Time of Test (Hour)
CS1	10/16/67	R. T.	760	O (Standard)
CS2	10/17/67	2000	10^{-8}	100
CS3	10/25/67	2000	10^{-8}	1000
CS4	10/20/67	2250	10^{-8}	10
CS5	10/27/67	2250	10^{-8}	100
CS6	11/15/67	2250	10^{-8}	1000
CS7	12/20/67	2500	10^{-8}	10
CS8	12/26/67	2500	10^{-8}	100
CS9	*	2000	10^{-8}	2200
CS10	*	2000	10^{-8}	1800
CS11	*	2000	10^{-8}	1000

* These stacks to be tested with creep capsules 25, 19 and 22 respectively.

III. TEST EVALUATION AND RESULTS

The LRHSC compatibility test evaluations are based on a systematic plan which was designed to reveal both gross reaction and traces of solid state diffusion. The major techniques used to date have been neutron radiography, metallography, and electron beam microprobe analysis. Future evaluations will include microhardness and X-ray diffraction analyses.

The initial plan of the evaluation program was to X-ray each coupon stack along two perpendicular diametral lines both before and after testing to show gross shifting and reaction of the coupons. It was found that X-rays were scattered so severely by the high density tantalum alloys that a clean sharp picture could not be obtained. Neutron radiographic results were somewhat better but not sufficiently good to justify use of the technique.

A. METALLOGRAPHIC EVALUATION

For metallographic examination, the coupon stack was vacuum impregnated with hysol metallographic mounting compound and cast into a cylindrical configuration with its axis perpendicular to the capsule axis. The casting was machined to a 1-1/2 inch diameter for automatic polishing purposes. The machined composite was then sectioned by diamond saw and polished. A polished section of stack CS2 is shown in Figure 13.

Photomicrographs of the coupon interfaces in both the unetched and etched condition were taken. Etching was accomplished using a solution of 3:1 nitric and hydrofluoric acid to reveal the T-111 and Ta-10W structures. To bring out the Pt-10Rh structure, an electrolytic technique using 5 amps in a salted hydrochloric acid solution was used.

The highlights of the metallographic investigations performed on CS2 (100 hour - 2000°F) are shown in Figure 14. Note the severe attack of the T-111 by iron titanate. Some reaction had been expected at the interface of

Figure 13. Macrograph (4X) of CS2 After Testing
(2000°F - 100 Hours)

T-111 and Pt-10Rh. It was discovered that in CVD coating one face of the coupon with Al_2O_3 , a very thin yet effective barrier was deposited on the black face. This layer was apparently sufficient to prevent the thermodynamically probable reaction. Note also the heat affected structure in the T-111 discs where a full penetration EB weld was performed. The welded regions showed no apparent difference in reaction characteristics from the parent metal. The photomicrographs show a significant depth of structureless area in the CVD coated T-111 coupons. However, previous analyses have shown that no major changes in alloying constituents have occurred in this region.

A closer look at the reacted areas (250X) is shown in Figure 15. Note the gross intergranular corrosion shown in the T-111 wafer. In this particular view it appears that Ta-10W does not suffer from this type of attack. However, when the half capsule was sectioned at 90°, it was observed that Ta-10W was indeed attacked (Figure 16). Similar results have been observed in CS4 (10 hour - 2250°F) as shown in Figure 17. After observation of the severe attack by the emittance coating on the tantalum alloys this interface was deleted from subsequent coupon stacks.

The 2500°F tests (CS7 and CS8) were conducted in the LRHS creep rig #1 which had a more efficient bell jar cooling system. Metallographic analysis of CS7 (10 hour - 2500°F) gave the first data on a reaction between Pt-10Rh and T-111. This reaction, a portion of which is shown in Figure 18, measured approximately 0.0015 inches wide. Upon examination at higher magnification (1000X), a distinct columnar region of the reaction zone was observed. This is shown in Figure 19. The nature of this reaction is being investigated.

B. ELECTRON BEAM MICROPROBE (EBM) EVALUATION

After complete metallographic characterization, stack CS2 was prepared for EBM analysis. A complete set of photomicrographs were sent with the sample to the probe to aid in finding areas of interest for examination.

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Figure 15. Detailed Coupon Stack Results

Figure 16. Apex View of a Quarter Section of CS2.
Note the gross attack of both T-111 and Ta-10W by Iron
titanate.

Iron Titanate
on Pt-10Rh

T-111

Ta-10W

Iron Titanate
on Pt-10Rh

Severe attack in
this entire region

Figure 17. Macrograph (4X) of CS4 (2250°F - 10 Hours) in Etched Condition

Figure 18. Photomicrograph of a Reaction
Occurring at an Interface of Pt-10Rh and
T-111 in CS7 (2500°F - 10 Hours)

Figure 19. High Magnification View of the Reaction Area Between Pt-10RH and T-111 in CS7

Only the one stack, CS2 (100 hour - 2000°F), has completely undergone post-test analysis through electron beam microprobe analysis. The coupon stack as it was examined was identical to the array shown in Figure 20. The stack interfaces were numbered 1 through 13, beginning with the interface between the Al_2O_3 spacer disc and the double coated T-111 coupon. The microprobe sample shown in Figure 21 shows a portion of the sectioned coupon stack which was prepared by core drilling a one inch sample from the original metallographic mount. Evaluation traces were made down the stack center line, the region through the welds in the T-111 coupons, and through the tapered edges (corresponding to the reference diameters A, B and C of Figure 20). Using these three traces, the effects of the weld structure and vapor transport on compatibility could be evaluated.

The results of this investigation³ enhanced the findings of the metallographic examinations. Again the dramatic interfaces were between iron titanate and both T-111 and Ta-10W. No other significant reactions were observed. Figure 22 shows the distribution of the elemental constituents present at interface 10 (Pt, Rh, Fe and Ti) where an iron titanate coated platinum rhodium disc is opposite a bare platinum disc. Note that the distribution is normal except for a slight diffusion of Fe into Pt. Figure 23 was observed at interface 5 where Pt-Rh coated with iron titanate was in contact with Ta-10W. This was one of the interfaces where significant reaction was noted metallographically. Here it may be observed that the iron, which was a constituent of the coating, has left the coating and diffused into the Pt coupon.

From these observations, it may be postulated that the iron oxide component of the coating ($\sim \text{Fe}_2\text{O}_3$) was reduced to Fe by the tantalum which oxidized, and the iron was removed to the Pt by diffusion. The photograph in Figure 23 indicates that this reaction went to near completion with no effect on the titanium distribution.

³ "Preliminary Electron Microprobe Examination of LRHSC Capsule CS2," by L. Cooper, Report No. TI-734-13-003, dated February 26, 1968

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Figure 20. Schematic Cross-Section of Capsule CS-2, Locating the Coupons and Coatings, and Defining the Interface Numbers and Reference Diameters

Figure 21. Electron Beam Microprobe Sample
cut from Compatibility Coupon Stack # 2

Fe X-rays

Pt-X-rays from Pt-10Rh Coupon

Ti X-rays

Gap between $\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$ coating and the Pt-10Rh coupon located between interfaces 9 and 10

Some Fe diffusion into the Pt-10Rh coupon

Approximate location of $\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$ coating

Figure 22. These three area scan photographs were taken at Interface 10 on diameter B and illustrate (1) the Fe and Ti to be "thoroughly mixed" in the coating, (2) a small amount of Fe diffusion into the Pt-10Rh coupon, (3) little or no Pt diffusion into the coating, and (4) a space between the coating and the Pt-10Rh coupon located between Interfaces 9 and 10

400X Photographs, $22\text{-}1/2 \mu$ per large division

Pt X-rays from Pt-10Rh Coupon

Fe X-rays showing that most of the Fe has left the coating and had diffused into the Pt-10Rh coupon

Ta X-rays from Ta-10W Coupon

Ti X-rays emanating from the region between the Ta-10W and Pt-10Rh coupons and, therefore, from the coating.

Figure 23. These four area scan photographs were taken at Interface 5 on diameter B and illustrate (1) the Ti remains in the coating, (2) the Fe separates from the coating and diffuses into the Pt-10Rh, and (3) there is little or no diffusion of Pt and Ta into the coating

400X photographs,

22-1/2 per large division

Other significant observations made by use of the microprobe were that there was no apparent difference in the compatibility behavior of the EB welded T-111 and the remaining wrought structure of the coupon disc. It was also revealed that the CVD alumina coating process deposited Al_2O_3 on both sides of the disc, as seen in the edge profiles of Figure 24. In the survey of the coupon edges, there was no evidence that vapor transport diffusion reactions had occurred between any coupons.

Figure 24. These area scan photographs, taken at the tapered ends of the T-111 coupons located between Interface Numbers 6 and 7, and 11 and 12, show Al(Al₂O₃) on both sides of each coupon, whereas (see Figure 1) only Interface Numbers 6 and 11 were expected to be coated with Al₂O₃. 200X Photographs
45 per large division

IV. CONCLUSIONS

Although only 20% of the scheduled tests have been evaluated, a significant amount of data important to the LRHSC program have been obtained. The original object of the experiments, as discussed earlier, was to yield sufficient time and temperature variation data such that reaction rates of interest could be evaluated. Preliminary evaluations of the test results have verified that sufficient data are available such that completion of the total evaluation program will allow a general plot of reaction rate at any interface against a time-temperature parameter. This data should then lend itself to basic thermodynamic and possibly synergistic computations.

The only violent reactions observed were those between the iron titanate coating and the tantalum alloys. The design of future systems must avoid this interface. Probably the most important piece of data obtained was that both CVD and arc plasma sprayed α alumina performed as effective diffusion barriers in preventing the Ta-Pt reaction from occurring. An interesting note on this reaction was that in the early stacks where reaction was expected, prevention was observed due to a less than one mil coating on the back side of CVD coated T-111 coupons. In later stacks such as CS7 (10 hour - 2500°F), where this thin film was ground off prior to testing, the Pt-Ta reaction occurred.

The early work in which a tungsten CVD flash was made on T-111 prior to CVD alumina coating was found to be unprofitable. It was initially thought that a W stop-off coating would protect the T-111 from air contamination during the alumina CVD application process. The results were that, in the case of the coupons, severe burn-up and contamination arose from the tungsten CVD process itself.

Complete compatibility was observed at interfaces of tantalum alloys with each other. The same was true for the Pt-10Rh alloy and the Pt with the emissivity coating. The electron beam microprobe was not capable of detecting

or measuring the concentration of interstitial contaminants such as C, O, H and N.

It was initially postulated that the hafnium in T-111 may be volatile in a 10^{-8} torr vacuum. However, the coupon edge analysis by EBM showed no traces of vapor phase transport due to exposure to the test conditions.

In summary, the LRHSC coupon compatibility stacks have been successfully exposed to the desired time/temperature/pressure conditions. The exhaustive evaluation program and subsequent generation of rate data have yet to be completed. However the preliminary evaluations have confirmed the expected compatibility of the basic capsule components, have indicated that APS alumina is as effective as CVD alumina as a compatibility barrier in this application, have indicated no vapor transport problems with these materials systems and have identified an iron titanate-Ta alloy interface as unacceptable.