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LARGE RADIOISOTOPE
HEAT SOURCE CAPSULE (LRHSC)
EIGHTH QUARTERLY PROGRESS REPORT
APRIL-JUNE 1969

AEC Research and Development Report

Atomics International
North American Rockwell

P2685

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ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION

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FOREWORD

This is the eighth quarterly progress report, describing the technical status of the Large Radioisotope Heat Source Capsule (LRHSC) Program. It covers the work accomplished by Atomics International during the period from April 1, 1969 through June 30, 1969 under contract AT(29-2)-2338 for the U. S. Atomic Energy Commission.

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I. INTRODUCTION

The Large Radioisotope Heat Source Capsule (LRHSC) Program has been established to develop the technology of an isotope fuel capsule capable of operation in a space environment at a surface temperature of about 2000°F for five years or more. The objectives of the present phase are the design, fabrication, test and evaluation of a test capsule whose design takes into account the operating requirements of a typical heat source. The work includes process, fabrication and test activities. The process and fabrication includes the work required to define an acceptable manufacturing specification for the reference design capsule and to manufacture the test items required for the program. The test program involves various coupons and capsules with evaluation of chemical compatibility, thermal cycling and adherence of coatings, capsule creep, capsule impact and capsule survival in potential abort environments. In addition to indicating the suitability of the reference capsule for ground test, the data generated in this program will be used to validate presently accepted design approaches and define design margins that would actually exist for high temperature capsules intended for flight systems.

The LRHSC Program is being accomplished under the following tasks:

- 1) Program Management
- 2) Capsule Design
- 3) Process Development
- 4) Fabrication and Assembly
- 5) Capsule Creep Testing
- 6) Impact Testing
- 7) Capsule Fire Test

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II. PROGRAM MANAGEMENT

A. TASK OBJECTIVES

The objectives of this task are to provide management, direction and planning for all aspects of the Large Radioisotope Heat Source Capsule Program. This includes technical direction, reporting, detailed plans and procedures.

B. SUMMARY OF TASK STATUS

The program milestones for GFY 1969 are shown in Table II-1. All milestones for GFY 1969 have been completed with the exception of the short term "C" capsule creep tests. Completion of these tests was delayed due to test capsule availability. The program completed the fiscal year on budget. The Program Plan for GFY 1970 has been completed and approved; and long lead time items for the GFY 1970 program have been ordered.

C. ACCOMPLISHMENTS DURING THE QUARTER

During the quarter discussions were continued with NASA-Lewis personnel on the application of the LRHSC technology to the large heat source system. Detailed design of capsules for the ground test system is expected in the first half of GFY 1970.

Negotiations with Mound Laboratories were completed and AI is supplying sub-assemblies and piece-parts to Mound for prototype fueling development. Completion of the hardware delivery will be in the first quarter of GFY 1970.

A major coordination meeting was held during the quarter on helium venting and the integration of vents into a LRHSC type capsule. Personnel from the AEC, industry and the using agencies participated in a review of the selective and non-selective venting programs, the LRHSC program and the fuels compatibility programs. The review was followed by a discussion of the details of integrating vents into LRHSC type refractory metal capsules and a

TABLE II-1
OFFICIAL MILESTONE SUMMARY

Official Milestones	Target Date	Status
1. Startup Creep Rig # 2	July 1968	Complete
2. Startup Creep Rig # 3	August 1968	Complete
3. Distribute Topical Report # 5	September 1968	Complete
4. Complete First "A" Capsule	October 1968	Complete
5. Startup Creep Rig # 4	October 1968	Complete
6. Complete First "C" Capsule	November 1968	Complete
7. Conduct Preliminary Impact Tests	November 1968	Complete
8. Complete First Creep Rupture Test	January 1969	Complete
9. Distribute Topical Report # 6	March 1969	Complete
10. Complete "C" Capsule Short Term Tests	May 1969	July 1969
11. Distribute Topical Report # 7	June 1969	Complete

preliminary discussion of GFY 1970 programs for accomplishing vent integration. Some effort in this area is anticipated in the GFY 1970 LRHSC Program.

The LRHSC Program Plan for GFY 1970 was completed and approved during the quarter. Next years program will be accomplished under the following tasks: Program Management; Analytical Evaluation, to include design of an optimized capsule for the large heat source ground test; Hardware Design, Fabrication and Assembly; Capsule Creep Testing, to complete those creep tests started this year and complete all post test evaluation; Capsule Impact Testing, to impact 17 capsules of various configurations; Materials Compatibility Testing, to include evaluation of alternate capsule materials and hold down materials; and Emittance Coatings, to evaluate alternates to iron titanate.

Long lead time materials for the GFY 1970 program were ordered during this quarter.

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III. CAPSULE DESIGN

A. TASK OBJECTIVES

The objectives of this task are to define the capsule and the test fixture requirements for the Large Radioisotope Heat Source Capsule Program. This encompasses capsule piece-part drawings, capsule assembly drawings, burial heater design and assembly drawings, and welding and test fixture design and drawings.

B. SUMMARY OF TASK STATUS

No design changes occurred during the reporting period.

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IV. PROCESS DEVELOPMENT

A. TASK OBJECTIVES

The objectives of the process development task are to provide the necessary technical guidance and control to all phases of the capsule fabrication in applications considered laboratory practice, to qualify fabrication, assembly and weld procedures, to perform the required experimental work in processes extending present technology, and to provide material and process specifications for the capsule fabrication program. The processes of interest include raw material and piece part fabrication, cleaning, annealing, joining, and non-destructive testing. Studies of alternate materials fabrication and assembly techniques will be performed as required.

B. SUMMARY OF TASK STATUS

The following is a summary of the met work on heat treating, creep rupture, coupons and miscellaneous samples:

- 1) Metallography and documentation of the microstructures of the tantalum base alloy components was accomplished.
- 2) Metallurgical analysis on all abort fire test capsule samples was completed and reported in Topical Report # 7.
- 3) Metallurgical analysis of creep capsule B5* (Burst Test Capsule) was initiated.
- 4) Metallurgical analysis of the impact capsules was completed.
- 5) Metallurgical analysis of the Pt-10Rh barrier shell deep drawing process was initiated.

C. ACCOMPLISHMENTS DURING THE QUARTER

1. Tantalum Base Alloy Components — Metallography and Documentation

The processes developed during the LRHSC Program have used interpretation of microstructures as a tool for establishing limits in forming, required heat treatments, and weldability. These metallographic studies were performed as required, but with limited documentation of the metallography. During this past quarter, documentation of the microstructures of the tantalum base alloy components has been completed. The following sections present examples of some of the microstructures and discuss the process development philosophy.

a. Potential Problem Areas in Tantalum Alloy Fabrication

The potentially serious problems which can be encountered in fabrication of tantalum base alloys involve critical grain size during forming and control of grain size during welding.

(1) Critical Grain Size

Recrystallization in metals is a nucleation controlled process. In general, the higher the amount of cold work the greater the number of nucleation sites. If the material is deformed uniformly, the nucleation sites are distributed uniformly throughout the metal; and, upon heat treatment for recrystallization, recrystallization occurs about these sites in a competitive process until equisized, equiaxed grains are produced. If the fabrication processes is properly designed, 100% recrystallization will produce this equilibrium structure. If the material is only slightly deformed, there will be some sites with much higher energy for recrystallization. Then, upon heat treatment, only selected sites will recrystallize. Furthermore, since there is not a competitive process, significant grain growth of selected areas will occur. This produces a duplex structure which is not amenable to further fabrication or welding. The specific problems associated with the fabrication heat treating process for the

T-111 structural shell have been detailed in a previous report from the program*. A typical example of a duplex structure from that report is shown in Figure IV-1. Further drawing of a material with this structure can be difficult because of non-uniform deformation under loading.

(2) Control of Welding HAZ

When materials are joined by welding, there is in fact a heat treatment to the base material. The volume over which this heat treatment occurs is normally termed the heat affected zone (HAZ). The temperatures reached in the HAZ are up to the melting point of the materials. Though the time at temperature is very short, the diffusion rates are very high. If the condition of the base material is not properly controlled, significant problems can occur. An example of the potential problem is shown in Figure IV-2. This figure shows the structure of a weld between two unalloyed tantalum sheets 0.005 and 0.010-in. thick. The thinner sheet was cold rolled from the thicker. The parts were not recrystallized prior to welding as is evident by the structure of the parent metals on the extreme right and left of the figure. During welding the driving force for grain growth is significant as evidenced by the very large grains (as much as three times the section thickness) in the HAZ. There are no interconnected zones in the weld nugget. Almost all of the deformation during straining will occur in only one or two of the grains (dependent upon orientation to stress axes). This will result in less than 1% total strain over a 1-in. gauge length, in a material which has a capability of over 100%. While this example is extreme, it indicates a boundary which can be encountered if care is not taken in the preweld treatment of this class of alloys. Care was taken to assure that this problem was not encountered on the LRHSC Program.

* LRHSC Program Topical Report No. 4, "T-111 Pressure Vessel Development and Fabrication," AI-AEC-12712, July 1968.

To prevent the high grain growth rate during welding, the base materials must be stable prior to welding. This is accomplished by recrystallizing the base metals. Since the driving force for grain growth is now small compared to the cold worked condition, little growth occurs during the short welding heat pulse, even though some of the base material reaches its incipient melting temperature.

Figure IV-1. Duplex Structure in T-111
Produced by Improper Forming and
Heat Treating Schedule

5334-4-1, 2, & 3

Figure IV-2. Weld Between Cold
Worked Unalloyed Tantalum
Sheet

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b. LRHSC Ta-10W Components

In the fabrication sequence for the Ta-10W components the heat treatment is designed to produce the optimum weld, high strain rate deformation, and stability properties. This requires the recrystallized condition. As will be shown there is some latitude on the degree of recrystallization; however, it should be in excess of 70%.

The tubing for the liners is procured in the recrystallized condition. After machining to final dimensions the components could be given a stress relief of 1 hour at 2000°F, but this has not proven to be necessary.

The liner end caps are coined from recrystallized stock. Figure IV-3 shows the structure of the cap near the weld lip of the formed parts. The figure also shows the recrystallization characteristics of the part. One hour at 2400°F is insufficient to impact significant recrystallization to the part. After one hour at 2800°F there is both recrystallization and significant grain growth. While this condition is acceptable for the application it is not optimum. After one hour at 2600°F the weld lip is about 85% recrystallized. The final heat treatment selected for these parts is one hour at 2700°F.

Figure IV-4 shows the structure of the welds made in the Ta-10W liners. These are typical of all the welds in the liner components. The grain growth in the HAZ, both in the end cap and in the tube wall, is minimal. There are also randomly oriented multiple zones in the weld nugget which is representative of a deformable weld. One relatively large grain (0.005-in.) in the fuel retainer ring suggests that the heat treatment temperature for this component could be increased if this is shown to be a problem. Impact tests to date have not indicated a problem in this area.

c. T-111 Components

Generically, two heat treatments are required for the T-111 semistock during fabrication of a capsule. One type is a process anneal between forming steps

a. Ta-10W End Cap After Forming

b. As Formed Microstructure

c. Microstructure After 1 Hour @ 2400°F

d. Microstructure After 1 Hour @ 2600°F

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Figure IV-3. Ta-10W End Caps Development

to render the semistock capable of further deformation; and, the final heat treatment is to develop the optimum of welding, creep, impact, and thermal stability properties in the component.

The development of the heat treating schedule for the T-111 structural components is contained in Topical Report No. 4 and will not be repeated here. Suffice to say that when a process anneal is required in the forming of the shell, one hour at 2700°F is used to produce about 90% recrystallization in the side walls of the drawn cup. This temperature is sufficiently high for the recrystallization, but low enough to prevent preferred grain growth. This is a very important consideration in development of the optimum forming properties by the heat treatment. In practice this process anneal occurs after the sixth stage of the drawing process prior to the two ironing operations which reduce the wall to the target 0.127-in. If the part is then to be further reduced to a thinner wall, for example 0.090-in., it is process annealed after this step and ironed directly to final size. The heat treatment to render the properties of the cold worked component optimum for assembly and/or service is performed after the final forming operation. This treatment is one hour at 3000°F. The final treatment is designed to produce a single phase structure and complete recrystallization in the walls of the tube. Comparison of the structure in the side wall before and after the heat treatment is shown in Figure IV-5. This structure represents the optimum condition for use. While the tensile strength of the material is higher in the cold worked condition than in the fully recrystallized condition (for instance 130 ksi vs 90 ksi), the creep properties of the recrystallized material are superior (by an order of magnitude in creep rate) to those of the cold worked material. While the impact characteristics have not been experimentally examined as a function of microstructure, one would expect that the material in the recrystallized condition can undergo a much more severe deformation than can the cold worked structure. The welding characteristics of the recrystallized material are far superior to the cold worked material. Figure IV-6 shows a weld made in 0.090-in. thick T-111

a. Inner Liner to End Cap Weld and
Inner Liner to Fuel Retainer Ring Weld

b. Outer Liner to End Cap Weld

Figure IV-4. Ta-10W Liner-End
Cap-Retainer Ring Welds

a. Prior to Heat Treatment

b. After 1 Hour @ 3000°F

Figure IV-5. Structure in Side Wall of T-111 Shell

Figure IV-6. Weld in 0.090-in. Thick Pressure Vessel

after the final heat treatment. Note the very even transition in the parent metal-heat affected zone. The driving force to induce grain growth during welding is so small as to make the existence of a classical HAZ virtually non-existent. Figure IV-7 shows the nugget-HAZ transition at the location of the root of the "V" in the weld lip. Note the oriented zones in the weld nugget produced by epitaxial nucleation from the grains in equilibrium in the HAZ. This condition results in a weld which contains the optimum in uniform deformation capability and strength.

There is one area in the structural shell which may not have the optimum of properties as a result of the heat treatment; that is the blind hemispherical end. Because of the nature of the forming process that portion has not received a significant amount of work from the wrought plate. As a result there is a tendency for some grain growth to occur during the final heat treatment. The grain size is about one or two ASTM micrograin size numbers smaller than the side wall. This condition might not be the optimum for impact deformation. Experiments have shown, however, that capsules impacted on that end up to 350 ft/sec undergo significant deformation without rupture.

2. Metallurgical Analysis of the Pt-10Rh Barrier Shell Deep Drawing Process

Metallurgical analysis of the Pt-10Rh barrier shell deep drawing process has been initiated. The drawing process is described in Topical Report No. 6. The Pt-10Rh material under investigation was taken from recently drawn shells, some of which failed during the drawing process. The examination of this material is, therefore, multipurposed.

- 1) To determine the cause of failure
- 2) To examine the microstructure of as-drawn Pt-10Rh

Figure IV-8 is a longitudinal cross section showing deformation of the material caused by partial drawing through the final sizing die. The thin section shown has passed into the land reducing its inside diameter to approximately $1.628 \begin{smallmatrix} + .010 \\ - .000 \end{smallmatrix}$ inches and thinning the wall to .020 inches, while

the tapered section next to it is being reduced to .020 inches by the female die. The thinning to .020 inches should take place at one major contact point with the land, at an angle of approximately 22° to the land of the die. As evidenced in this figure, there are at least two major points of contact with the land. The first is approximately .080 inches from the intended single contact point and the second is near the intended contact point. The material flows through the die with a moderate amount of deformation except at the outer surface where the material is highly cold worked. As the punch moves through the die the wall is thinned from approximately .038 inches to $.020 \pm .002$ inches. The material gathers above the land, as seen in Figure IV-8, causing preferred orientation of the grain structure.

Figure IV-7. Weld-HAZ Interface in T-111
Pressure Vessel Weld

Figure IV-8. Longitudinal Section
Showing Deformation of Partially
Drawn Pt-10Rh Barrier Shell

Etchant: HCl and H₂O Saturated
with NaCl
(100X Reduced to 55%)

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Figure IV-9 is a transverse cross section of the barrier shell prior to final sizing; that is, it has gone through five draws, been annealed at 1800°F for 1 hour and been drawn through the sixth draw. Figure IV-10 is a transverse cross section after final sizing. The Pt-10Rh in both figures appears sufficiently ductile to undergo further deformation and is therefore eliminated as a cause for failure.

Here then is the most probable cause of failure. The dies, after previous drawing runs, were cleaned several times. The method employed to clean the land was to hold the die in a lathe, spin it and apply emery cloth to the land. The very nature of this operation enhances the possibility of a higher metal removal rate at the edge of the land rather than at the surface away from the edge, and also yields a polished surface. An examination of the material flow, as seen in Figure IV-8, indicates that this is indeed the case. The ID of the land appears to be opened as much as .010 inches more at the edge than at the surface .080 inches into the land. This permits a forming angle of approximately 7° rather than 22° and introduces higher tensile loads. Also, the polished die causes the lubricant to break down allowing metal to metal contact which is conducive to galling. The resultant force required to push the shell through the die exceeded the tensile strength of the Pt-10Rh, resulting in failure. The dies used to draw the Pt-10Rh parts through final sizing will be corrected prior to final sizing of additional Pt-10Rh barrier shells.

100 X

Figure IV-9. Transverse crosssection of Pt-10Rh prior to
final sizing draw
Electrolytic Etchant - HCL and H₂O saturated
with NaCl

100 X

Figure IV-10. Transverse crosssection of Pt-10Rh after
final sizing draw
Electrolytic - HCL and H₂O saturated with NaCl

V. FABRICATION AND ASSEMBLY

A. TASK OBJECTIVES

The objectives of this task are to obtain all raw materials, to fabricate all capsule and coupon piece-parts, to build and check out all piece-part and assembly tooling, and to assemble all capsules and coupon stacks.

B. SUMMARY OF TASK STATUS

Six structure shells were successfully drawn through nine dies to a .090 wall. Assembly of creep capsule C-1a and impact capsule A-8a was completed.

C. ACCOMPLISHMENTS DURING THE QUARTER

Deep drawing of six structural shells to a .090 wall was completed; the shells were cleaned and annealed and delivered to final machining. Final machining of these six shells is in work and approximately 75% complete.

Assembly of creep capsule C-1a was completed (Figure V-1 and V-2).

Assembly of impact capsule A-8a was completed (Figure V-3).

Twenty fuel closure rings were formed and finish machined. Two each were used to complete the outer liner assemblies of creep capsule C-1a and impact capsule A-8a.

The tooling used to fabricate the Pt-Rh barrier shells and caps for a .090 wall structure has been designed and fabricated.

Purchase orders have been placed to procure GFY 1970 material.

Figure V-1. Creep
Capsule C-1a, Pt-10Rh
Assembly

Figure V-2. Creep
Capsule C-1a lead tube
Assembly

Figure V-3. Impact Capsule
A-8a

VI. CAPSULE CREEP TESTING

A. TASK OBJECTIVES

The LRHSC Program fuel capsule design utilizes a first generation tantalum alloy (Ta-10W) for primary containment of the fuel, a second generation tantalum alloy (T-111) for secondary containment, and Pt-10%Rh for oxidation protection of the reactive refractory metal containers. The objective of this task is to provide and test a design approach that accounts for creep deformation at elevated temperatures for this capsule design.

To achieve this end a test series has been designed to evaluate heat-to-heat variations on the creep of the T-111 structural shell, determine the effects of variable stress on creep of the shell, relate uniaxial and biaxial stress states to creep of T-111 and finally to determine the interacting effects of capsule components on the creep of the structural member.

During the first year of the program three biaxial creep rigs were operated to observe the creep of capsule components @ 2000°F in high vacuum. At the end of the first year preliminary creep data for test times less than 2000 hours had been obtained. During the second year additional creep rigs have been utilized and longer term creep data obtained.

B. SUMMARY OF TASK STATUS

1. Creep Capsule B-3a

Creep capsule B-3a is the first long term (.1% strain in 8800 Hours) creep test of a T-111 structural shell. The test was initiated during the last quarter and has continued throughout this reporting period to .097% strain in 1600 hours.

2. Creep Capsule B-6a

Creep capsule B-6a is also a long term test of the T-111 structural shell and has accumulated .095% strain in 1850 hours.

3. Creep Capsule A-2a

Creep capsule A-2a has been on test for 218 hours and has achieved approximately 0.07% diametral strain.

4. Equipment

The furnace for Rig #5 was completed and construction of the furnace for Rig #6 begun. Two vacuum chambers and the necessary materials to complete Rigs #6 and #7 were ordered.

C. ACCOMPLISHMENTS DURING THE QUARTER

1. Test Operation

Creep capsule B-3a is the first long term creep test of a T-111 structural shell that has accumulated 1600 hours at 16,500 psi hoop stress. The apparent diametral strain is approximately 0.097%. The capsule is scheduled to reach 0.1% diametral strain in 2800 hours while under a constant stress of 16,500 psi. Thereafter, it is to be increased at a rate to produce a total of 1% strain in the following 6000 hours. The behavior of this test is shown in Figure VI-1.

Creep capsule B6 is also a long term test of the T-111 structural shell and has accumulated 1850 hours at 16,500 psi hoop stress. It has an apparent diametral strain of approximately 0.095%. The creep schedule for this test is the same as that for B-3a except the stress rate will continue until the capsule has achieved 5% total strain. The behavior of this test is shown in Figure VI-2.

Creep capsule A-2a had been on test for 218 hours and had achieved approximately 0.07% diametral strain when the furnace for this system shorted causing the fuses in the SCR Unit to blow. The cause for the short was found, corrected, and the test was reinitiated. The behavior of this test is shown in Figure VI-3.

Figure VI-1. Creep Capsule B-3a Test Behavior

Figure VI-2. Creep Capsule B-6a Test Behavior

Figure VI-3. Creep Capsule A-2a Test Behavior

2. Creep Rig

The furnace for Rig #5 is complete and construction of the furnace for Rig #6 has begun. A T/C bundle was assembled and calibrated for Rig #5.

Two vacuum chambers for the construction of Rig's #6 and #7 were ordered and should be received within a week. The necessary materials to complete these systems were also ordered and have either been received or are in transit. The materials include Pt-Pt10%-Rh wire for thermocouple construction, Al_2O_3 standoffs and tantalum sheet for furnace construction, burst diaphragms, copper gaskets, and other assorted items which are necessary for the construction of and maintenance of creep systems. Rigs #5, 6, and 7 will be used in support of the GFY 1970 emittance and compatibility tasks. Rigs #2 and 4 will be used in GFY 1970 for aging the pressurized impact capsules and for long term compatibility tests.

VII. IMPACT TESTING

A. TASK OBJECTIVES

The objective of this task is to determine, by testing, the compatibility of the reference capsule design to survive impact. The developmental impact tests will also allow insight into the actual capsule deformation phenomena, determine the capsule failure modes and ascertain the relative influence of various capsule impact parameters on capsule failure. A total of 17 capsules will be impacted on a granite target at various velocities, impact angles, and temperatures.

B. TASK STATUS

During the reporting period a .090 wall capsule of the LRHSC "A" configuration was successfully impact tested at 1200°F at a velocity of 355 ft/sec.

C. ACCOMPLISHMENTS DURING THE QUARTER

Impact capsule A-8a, a 0.090 wall capsule, was completed, heated to 1200°F and impacted at 355 ft/sec. The capsule survived the test in a leak tight configuration. Figure VII-1 and Figure VII-2 show the capsule after testing. Figure VII-3 is a summary of the impact tests to date.

Figure VII-1. Impact Capsule A-8a After Testing

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Figure VII-2. Impact Capsule A-8a After Testing

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S/N	Impact Velocity ft/sec	Angle	Impact Temp. °F	Ini. Length (in)	Dia. (in)	Final Length (in)	Final Dia. (in)	Dia. Strain %	Axial Strain %	Mass LB	Impact Energy ft/lbs	Wall (in)	Post-Test Wall		With Pt Rh	Summary
													Max.	Min.		
A-1a	250	30°	1200	7.375	1.676	6.836	1.926	14.8%	21.2%	4.8	4,680	.127	.165	.127	Yes (Cap)	T-111 and Pt-Rh Leak Tight
A-2a	250	10°	1200	7.376	1.677	6.750	1.928	15.0%	28.7%	4.7	4,720	.127	.160	.127	Yes (Cap)	T-111 Leak Tight
A-3a	260	End On	1200	7.372	1.676	6.610	1.972	17.4%	26.4%	4.7	4,940	.127	--	--	Yes (Cap)	Leak Tight
A-4a	230	10°	1200	7.348	1.676	6.616	2.030	21.5%	26.8%	4.8	3,950	.127	--	--	Yes (Cap)	Leak Tight
A-5a	240	End On	1200	7.325	1.624	6.624	1.919	18.2%	29.0%	4.0	3,600	.100	--	--	No	Leak Tight
A-6a	260	43°	1200	7.323	1.625	6.525	1.932	18.9%	33.8%	4.0	4,200	.100	.140	.100	No	Leak Tight
A-7a	346	End On	1200	7.419	1.718	6.100	2.115	26.5%	42.2%	5.2	9,700	.127	--	--	Yes (Comp)	Leak Tight
A-8a	355	20°	1200	6.900	1.600	5.750	2.000	25.0%	40.0%	3.6	7,000	.090	--	--	No	Leak Tight

Figure VII-3. Mechanical Test Analysis of Impact Capsules A-1a Through A-7a

VIII. CAPSULE FIRE TEST

A. TASK OBJECTIVES

The objectives of this task are to subject the LRHS capsule to a launch pad accident fire environment and to determine the effect of the fire on the capsule. The intent of the test is to simulate an event where a fueled heat source capsule comes in contact with burning solid propellant. The desired result is that the fuel loading be contained within the fuel annulus of the capsule.

B. TASK STATUS

During this reporting period a complete post test analysis of the LRHSC Fire Test Capsule was completed and reported in LRHSC Topical Report No. 7 (AI-AEC-12841).

C. ACCOMPLISHMENTS DURING THE QUARTER

The LRHS Capsule Fire Test task was originated during the previous reporting period. The capsule was delivered to Isotopes, Inc., and subjected to a launch pad fire environment. The test took place on March 27, 1969. The burn time was 4-1/2 minutes with a heat flux of approximately 49 BTU/ft²/sec. The temperature in the fuel annulus reached 2800°F in 1 minute and 45 seconds. At this time the instrumentation lead tube burned off exposing the internal surfaces of the capsule to air during the rest of the test. As reported in the last quarterly the fuel simulant was contained within the fuel annulus, the fuel container was unaffected and the T-111 structural shell was not penetrated by the effect of the intense heat of the fire environment (up to 4870°F).

During this reporting period a complete post test analysis was conducted. Analyses included metallographic, X-ray diffraction, emission spectrography, and electron beam microprobe. The results of these tests follow.

Figure VIII-1. Post Test View of the LRHS Capsule

1. Post Test Examination

a. Visual Examination and Helium Leak Check

Following the test the capsule was visually examined for any external indications of failure. The capsule was covered with large deposits of residue generated from the propellant flame. The platinum shell appeared to be intact along the surface that was in contact with the fuel but, due to the deposits of residue surrounding the rest of the capsule, it could not be determined if the platinum shell was totally intact. Figure VIII-1 shows the bottom view of the capsule. Note the modular appearance of the propellant residue and the metallic substance covering the bottom of the capsule.

It was apparent from the radiographs that the capsule configuration was only changed by a minor reduction in wall thickness. To substantiate the integrity of the T-111 structural shell, a mass spectrometer helium leak test was performed. The end of the capsule which had been penetrated for instrumentation was machined flat to provide a vacuum tight sealing surface. The capsule was subjected to a vacuum of $< 1 \times 10^{-3}$ mm of hg using a Veeco Mass Spectrometer Helium Leak Detector. The sensitivity of the leak detector was determined to be $< 1 \times 10^{-10}$ std cc/sec of helium at 1 atmosphere of differential pressure. The capsule was surrounded by a plastic shroud which was purged with helium for 1 hour. There was no indication of a leak. The post-test sensitivity of the leak detector was confirmed to be 1×10^{-10} std cc/sec of helium.

The capsule was cut on a plane perpendicular to the tangent of the line of contact with the solid propellant. The Ta-10W fuel liner assembly was undamaged and the fuel simulant was totally contained. It was necessary to remove the fuel simulant in order to grind and polish the capsule to show the surface reaction. Therefore, Figure VIII-2 which shows the capsule interior does not show the fuel simulant. There are, however, isolated areas which can be seen in the photograph where the fuel simulant did adhere to the Ta-10W outer liner.

Figure VIII-2. Cross Sectional View of Capsule Interior

Figure VIII-3. Oxidation of T-111 in Oxy-Acetylene Flame

b. Analytical Analysis by X-Ray Diffraction, Fluorescence, and Emission Spectrography

Areas of interest along the capsule surface were selected and samples were taken for X-ray diffraction analysis. Samples representative of visual differences were taken first. Extensive metallographic and electron beam microprobe examinations were performed. Emission spectrography samples were also taken from these areas.

Analysis of the X-ray fluorescence data indicated that the silvery metallic substance removed from the capsule surface, at approximately 90° to the interface of the propellant, contained major amounts of aluminum, tantalum and platinum. The phases could not be identified by X-ray diffraction. Nodules near the ends of the capsule, containing gold and green colored platelets, were determined to be Al_4C_3 , $\alpha\text{-Al}_2\text{O}_3$ and aluminum metal. A black porous deposit from near the top was primarily AlTaO_4 with a minor amount of $\alpha\text{-Al}_2\text{O}_3$, while the white deposits from the top of the capsule were primarily $\alpha\text{-Al}_2\text{O}_3$. There was one isolated glassy appearing area approximately 1-in. in diameter on the top of the capsule that was amorphous SiO_2 . It is reasonable to assume that the source of SiO_2 was the foamed silica lead tube protection material.

An analysis of the propellant residue in fire tests performed prior to this one indicated the ashes to be 75% Al_2O_3 , 20% aluminum and 5% Al_4C_3 *. The presence of these materials on the surface or reacting with the capsule would be expected. An oxidation reduction test performed prior to the fire test showed that T-111 in an oxygen rich oxy-acetelene flame of 3350°F formed $\alpha\text{-Ta}_2\text{O}_5$. Comparison shows the grain boundary attack found in the oxidation test (Figure VIII-3) and the surface reaction found in the fire test (Figure VIII-4) to be very similar. After the integrity of the platinum oxidation barrier was penetrated, the formation of AlTaO_4 as a product of the propellant residue and the oxidized T-111 structural shell would be expected.

* Personal communication with Hal Nichol, Isotopes, Inc., March 15, 1969.

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Figure VIII-4. Cross Sections of Tested Capsules

Reduced to 75-1/2 %

(Sheet 1 of 2)

Figure VIII-4. Cross Sections of Tested Capsules

Reduced to 75-1/2%

(Sheet 2 of 2)

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Of the samples taken for analysis from the locations corresponding to metallographic and electron beam microprobe examination, only the primary phase of Al_2Pt could be positively identified by X-ray diffraction. This phase of Al_2Pt , although interspersed with tantalum, tungsten and rhodium, appears to be prevalent in all but the area immediately adjoining the structural shell. It was there that the elemental compounds were so intermixed that phase identification was unobtainable within a reasonable length of time.

Emission Spectrographic analysis of the above discussed areas is summarized in Table VIII-1 and indicates the presence of elements in minor and trace amounts which had not been identified by X-ray diffraction. The elements are chromium, copper, iron, nickel, titanium and tungsten. The emittance coating of $\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$ could account for the iron and titanium and the source of tungsten could be the T-111 structural shell. The chromium, copper and nickel are most likely from the solid propellant starter materials which consisted of electric matches, nails, wire and gun powder.

c. Metallographic Examination

Comparison of the radiographs and the sectioned capsule indicated that a reasonable estimate of uneffected wall thickness could be derived by examination of the radiographs. The radiographs were used to determine the area where the minimum wall existed. A section exhibiting one half the radial plane was removed at the minimum wall by an abrasive cutting wheel.

The sample was vacuum impregnated with hysol metallographic mounting compound and it was ground, polished and etched for metallographic examination. Figure VIII-4 shows a photomontage showing the microstructure of the Ta-10W inner and outer liners, the T-111 structural shell, and the intermetallic and ceramic formation surrounding the capsule. It also shows a photomacrograph of the sample in relation to its position on the solid propellant during the fire test.

TABLE VIII-1
EMISSION SPECTROGRAPHY OF REACTION PRODUCTS

Element	Sample Number				
	2608	2609	2610	2611	2612
Aluminum	M*	M	M	M	T
Chromium	S**	T	T	T	
Copper	S	T	S	T	T
Iron	S	T	S	S	T
Nickel	S	T	T	T	
Platinum	S	T	S	S	T
Silicon	T***	T	T	T	M
Titanium	T	T	S	S	
Tantalum	S	T	M	M	
Tungsten	T	T	T	T	

* "M" indicates a major constituent

** "S" indicates a minor constituent

*** "T" indicates a trace constituent

It is clear from the figure that an intergranular attack has taken place at all of the interfaces with the exception of the outer wall of the Ta-10W outer liner. Precipitation at the inner wall of the Ta-10W outer liner occurred to a depth of .008-in., while precipitation to the grain boundary at the surface of the outer wall of the Ta-10W inner liner was .006-in. Both the inner and outer liners were examined by an Electron Beam Microprobe (EBM) in an attempt to determine the composition of the precipitate. The liners were scanned for Fe, Pt, Al, W, Ta, Mo, Ti, Hf and Rh, and were void of all the elements with the exception of the normal expected levels of Ta and W.

There is a transgranular attack at both surfaces of the T-111 structural shell, primarily, however, at the interface with the Ta-10W outer liner. The depth of the attack was .006-in. and the measured minimum wall thickness of the T-111 was .041-in. These surfaces were scanned by the EBM at the same time as the Ta-10W liner, for the same elements, and with the same results. Only the normal expected levels of Ta, W and Hf were found. The only elemental identifications of tramp materials were in the reaction zone between the Ta-10W outer liner and the T-111 structural shell. The reaction zone is approximately .003-in. wide and consists of a phase identified by X-ray diffraction as a sigma tantalum-aluminum intermetallic somewhere between Ta_2Al and Ta_3Al . The EBM scans taken across this same reaction zone identified the presence of tantalum, platinum and aluminum, with the notation that the scans fluoresced, not only in the reaction zone but also in the region of grain structure attack in the T-111. This is usually an indication of the presence of an oxide. The annulus between the Ta-10W and the T-111 contained a platinum clad T/C sealed with alumina. The presence of these elements in the interface was therefore expected. Figure VIII-5 is a comparison of EBM area scans with a photomicrograph for visual reference.

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250X PHOTOMICROGRAPH ENLARGED 200%

Figure VIII-5. Electron Beam Microprobe (EBM) Area and Line Scans
(Sheet 1 of 2)

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5 LINE SCAN FOR Ta

E.B.M. LINE X-RAY SCANS
5 THRU 7

NOTE THAT THE CONCENTRATION OF
ALUMINUM CORRESPONDS TO THE
AREAS VOID OF TANTALUM

6 LINE SCAN FOR W

7 LINE SCAN FOR PLATINUM

2 Ta X-RAYS 800X

3 W X-RAYS 800X

4 Pt X-RAYS 800X

Figure VIII-5. Electron Beam Microprobe
(EBM) Area and Line Scans
(Sheet 2 of 2)

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Hardness of the T-111 structural shell falls within the range of full recrystallized T-111 ($20R_C$ to $25R_C$)*, except at the outer .010-in. of the T-111 structural shell where the hardness increases to as high as $55R_C$. Hardness of the Ta-10W inner and outer liners were higher at the surfaces in contact with the fuel simulant than that exhibited by fully recrystallized Ta-10W ($34R_C$ vs. $25R_C$). The lead tube burned off approximately 1 minute and 45 seconds into the test. Therefore, the increase in hardness was probably due to the 3 minute exposure to air at elevated temperature. The surfaces away from this interface were also slightly higher ($27R_C$ vs. $25R_C$). Microhardness of the intermetallic region formed from the interreaction of the solid propellant residue with molten platinum-10% rhodium and T-111, ranges from 308 to 1581 K.H.N. While the phase identified as α - Al_2O_3 has a hardness up to 3940 K.H.N. Figure VIII-4 has been detailed to locate areas representative of the phases identified by X-ray diffraction. These phases are Al_2Pt , Al_2O_3 , and the σ tantalum-aluminum intermetallic. Elements which were identified by the EBM are also called out on this figure wherever possible.

d. Electron Beam Microprobe Analysis (EBM)

In all of the scans through the intermetallic layer surrounding the T-111 structural shell, the following was found to be true. Where platinum was present there was a presence of aluminum, and when tantalum was present there was a presence of tungsten. There were isolated areas where the elements were separate of each other, as in the case of Ta-10W, Figure VIII-6, or when all was intermixed, Figure VIII-7. The needle like structure in Figure VIII-8a is a Ta-W rich in a primary matrix of Pt-Al with the presence of a darker structure identified to contain all four of the elements. The stringers shown in Figure VIII-8b are high in tantalum surrounded on one side by tantalum in a Pt-Al matrix, identified by X-ray diffraction to be Al_2Pt , and on the other side by

* R_C Hardness numbers were converted from 500 gram load Knoop Hardness Numbers.

B.S.E. Area Scan

800 X

Tantalum X-Ray Area Scan

800 X

Tungsten X-Ray Area Scan

Figure VIII-6. A Comparison of
Ta and W EBM Scans

800 X

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Tantalum X-Rays 800X

Tungsten X-Rays 800X

Platinum X-Rays 800 X

Aluminum X-Rays 800 X

Figure VIII-7. A Comparison of Ta, W, Pt and Al EBM Scans

Figure VIII-8. Photomicrographs of Intermetallic Region

primarily tantalum of a highly brittle phase. Figure VIII-9 illustrates the relative distribution of platinum and aluminum to rhodium. Platinum has separated from the rhodium and alloyed solely with the aluminum leaving two separate and distinct phases, Al_2Pt and platinum-rhodium with aluminum. Platinum and aluminum are thoroughly mixed through both phases, as evidenced in area scans 2 and 3 of Figure VIII-9. Area scan 4, of this figure, reflects the pattern formed by the matrix and by the back scattered electrons (area scan 1). Scan 4 shows that rhodium is found in only one of the two phases which would account for the visual difference. Area scans 5 and 6, for tantalum and tungsten, fill the voids in area scans 2, 3 and 4 (dark areas). Tantalum is more prevalent than tungsten and appears to surround or overshadow the tungsten as though they were separate of each other, although this had not been distinguished microscopically.

Of all the elements scanned for in the intermetallic region surrounding the T-111 structural shell, only iron had a concentration level high enough to be detected by the EBM and was detected only in isolated areas near the surface of the T-111.

2. Conclusion

The fire test has demonstrated that the LRHS capsule without reentry protection will withstand a solid propellant launch abort fire without having its structural shell penetrated.

3. Additional Data

LRHSC Topical Report Number 7 (AI-AEC-12841), summarizing the test and evaluation is available.

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Figure VIII-9. A Comparison of Pt, Al and Rh EBM Area Scans