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LARGE RADIOISOTOPE HEAT SOURCE
CAPSULE (LRHSC)
FIRST QUARTERLY PROGRESS REPORT
JULY-SEPTEMBER 1967

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

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FOREWORD

This is the first 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 July 1, 1967 through September 30, 1967 under Contract AT(29-2)-2338 for the U. S. Atomic Energy Commission.

SUMMARY

FY 1968 program planning has been completed. All materials specifications have been released and preparation of the process and testing specifications initiated. The Electronic Data Processing program has been completed and is being checked out.

Analyses have been accomplished on the shrink-fit of the capsule structural shell to the inner fuel assembly, the pressure restrictions on the helium under the Pt-10Rh clad and the pressure-electrical feedthru design.

The reference and creep test capsule designs have been completed and drawings released. Fabrication techniques have been specified for each of the capsule piece parts.

Screening tests have been completed for the braze alloys in the pressure-electrical feedthru assembly. Also preliminary results from feasibility tests indicate back extrusion may provide a suitable backup to deep drawing of the T-111 structural shell.

Fabrication and assembly tooling designs have been initiated and the preliminary weld development and quality assurance plans completed.

Preliminary results from the coating development tests show that α (alpha) phase Al_2O_3 can be laid down by CVD. Preliminary indications are that a repeatable Al_2O_3 coating process without undue contamination of the substrate is feasible.

The design of the coupon compatibility stacks started. The heater and vacuum systems for the coupon compatibility tests are being modified and checked out.

Modification designs for the creep test heater and vacuum systems are in progress. Design of the helium pressure system for creep testing is completed and fabrication of the system has started. Alternate methods of measuring in-test strain are being evaluated.

Approximately 5000 ft² of laboratory facility are being readied for the LRHSC program. Basic utilities and the bulk of the equipment required for FY'68 are installed.

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I. INTRODUCTION

The LRHSC Program has been established to develop the technology of an isotope fuel capsule capable of operating 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 preliminary 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 earth burial. In addition to indicating the suitability of the reference capsule for ground test, the data generated in this study 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) Planning
- 2) Analytical Evaluation
- 3) Capsule Design
- 4) Process Development
- 5) Fabrication and Assembly
- 6) Capsule Creep Testing
- 7) Coupon Compatibility and Coating Development
- 8) Impact Testing
- 9) Soil Burial
- 10) Test Capsule Evaluation

The sections of this report have been organized to correspond with the above task schedule. For each task an objective, summary of current status and discussion of accomplishments during the quarter are presented. In addition an appendix is included describing the status of the laboratory facilities and the equipment being used on the LRHSC Program.

II. PLANNING

The overall program schedule is shown in Figure II-1. The milestones completed during the first quarter consist of the following:

<u>Task No.</u>	<u>Milestone</u>
1a	Data Management Code - Code is complete through fabrication and assembly stations. Test and evaluation station code sheets remain to be accomplished. The remainder of the code is not required until approximately January 15, 1968.
1b	Quality Control Plan - Completed
2a	Design Analysis - Essentially Complete
2c	Coupon Test Support - Complete
3a	Capsule Piece Parts - Drawings Released
3b	Capsule Assembly Drawings - Drawings Released
3c	Assembly Fixtures - Drawings Complete - Not Released
4a	Reference Capsule Process Flow Sheets - Complete
4b	Creep Capsule Process Flow Sheets - Complete
4e	Material Specification - Complete - Released
4f	Test Specification - Coupon Compatibility - Complete

Formal program planning and control documents completed during the quarter are summarized below.

1) Weld Development: Weld development planning is complete. Because of the late arrival of the 6 kw Electron Beam welder, the T-111 weld for the structural capsules (a configuration) will be developed and pre-qualified under sub-contract. Final qualification will be accomplished

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Figure II-1. Large Radioisotope Heat Source Program Milestones
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Figure II-1. Large Radioisotope Heat Source Program Milestones
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at AI. All weld development is scheduled to be complete by January 15, 1968. Ten welds are to be developed, two on T-111 and eight on Ta-10W. EB welds and TIG welds are both used on the capsule.

2) Quality Assurance: The Quality Assurance plan has been completed and is in process of being implemented. Thirteen Quality Assurance Acceptance procedures have been written covering welding, coating, brazing, testing and evaluation.

3) Process Flow Sheets: Process flow sheets for the A, B, and C configuration capsules have been detailed and are available. These sheets form the basis for fabrication and assembly of the capsule as well as Quality Assurance planning.

4) EDP: The EDP (Electronic Data Program) Fortran input sheets, covering material physical and chemical certification, dimensional records on piece parts, configuration records and in-process data, have been completed. The initial print out format has been selected and a simulated capsule record input will be made to ascertain the appropriateness of the output. Other print out formats are to be derived later.

5) Specifications. A summary of the specification status in terms of a Specification Tree is shown in Figure II-2. Those specifications required for direct support of the program during GFY'68 are indicated with an asterisk (*).

The following is the list of LRHSC specifications written and formally released during this quarter.

<u>Material</u>	<u>Process</u>	<u>Test</u>
ST0170NA0048 Ta-10W Bar & Rod	ST0615NA0033 LRHSC In-Process Control Center	ST0408NA0023 LRHSC Coupon Compatibility Test
ST0170NB0049 Ta-10W Tubing	ST0610NA0005 Cleaning Ta & Ta Base Alloys	

<u>Material</u>	<u>Process</u>	<u>Test</u>
ST0170NB0050 Ta-10W Plate, Sheet Strip and Foil	ST0611NA0006 Cleaning Pt-10Rh	
ST0170NB0051 T-111 Plate, Sheet Strip and Rod		
ST0170NB0054 T-111 Tubing		
ST0170NB0058 Pt-10Rh Sheet & Strip		
ST0170NB0059 Pt-10Rh Tubing		
ST0115NB0018 Iron Titanate Powder		
ST0110NB0027 Ultra High Purity Helium Gas (99.9999)

Figure II-2. Specification Tree, LRHSC

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III. ANALYTICAL EVALUATION

A. TASK OBJECTIVES

The objectives of this task are to provide analytical support to the capsule design, fabrication, and test programs, and provide the analysis for verification of design approach, using the test data generated in the program. Further efforts will include definition of the design margins in the capsules by comparison of test program results with typical operational requirements.

B. SUMMARY OF TASK STATUS

To date analysis has been directed primarily to finalizing capsule design parameters with some analysis of the fabrication and assembly sequence. Analytical verification of the test capsule design has been completed with the exception of definition of the impact absorber parameters. Emphasis will now shift to the assembly and test operations.

C. ACCOMPLISHMENTS DURING THE QUARTER

Specific analysis have been accomplished in the past quarter on the shrink-fit of the capsule structural members, the platinum clad design and assembly criteria, and the pressure-electrical feedthru design.

1. Shrink-Fitting of Structural Components

The low emissivity of Ta-10W and T-111 could result in a large temperature difference between the Ta-10W fuel liner and the T-111 structural shell with resultant high capsule temperatures during assembly and operation. An interference fit has been adopted to reduce the temperature difference and lower the internal capsule temperatures. An investigation was made to determine the effect of contact pressure on ΔT between the two capsule components so that the required amount of interference could be determined.

A review of the existing literature in the area of thermal contact resistance has shown that as of 6 years ago most of the reported work has been done in the presence of air as an interstitial fluid. Today, there exists in the literature a large amount of data from investigations in vacuum environments. However, a large majority of these investigations were for specialized configurations, and little was obtained that could be used to predict the resistance of joints in other physical situations. A second shortcoming is the absence of adequate information on the surfaces, their properties, and other test parameters.

The most comprehensive bibliography on this subject was found in Ref. III-1. One of the most extensive contact resistance investigations in a vacuum is that reported by Clausing. Fried, in his summary report on contact resistance for NASA (Ref. III-7), makes special mention of Clausing's work (Ref. III-2, -3, -4, -5, and -6). However, Clausing's investigation did not attempt to perform tests of realistic engineering surfaces. It was designed with the primary objective of increasing understanding of the basic mechanism governing the thermal contact resistance.

Clausing presents a theoretical model which considers "microscopic" resistance and "macroscopic" resistance. The normal contact area is divided into contact and noncontact regions. The contact region, defined as the "macroscopic" contact area, is due to the large scale waviness, or non-flatness of the surfaces. The macroscopic contact area is populated by a large number of microscopic contact areas as the result of surface finish. By definition the noncontact region is that containing a negligible number of microscopic contacts. Clausing's model was shown to permit prediction of his experimental data and provided explanations of previously puzzling results. The value of his work lies in the proposed approach which recognizes the dominances of the macroscopic constriction effects.

Clausing was consulted about our particular configuration to determine if his work and/or any other documented information or information from current investigations could be used with confidence to predict the contact resistance for our geometry. His reply was that our problem is vastly different than that commonly studied. In his opinion, the use of published work would be meaningless for thin-walled concentric tubes. The literature would, of course, be of value in obtaining an understanding of the problem. He didn't know of any current investigations that would be applicable.

Clausing's educated estimate was that a contact pressure of 100 psi would provide a significant reduction in thermal contact resistance and that any contact pressure at all will produce a lower temperature difference than that expected by radiation between tantalum alloys. He suggested that the best way to determine the temperature difference was to measure it for the particular geometry and surface preparation.

In summary, any interference fit between the T-111 and Ta-10W will reduce the ΔT between the component. However, there is no data available upon which to predict ΔT as a function of contact pressure. It has, therefore, been decided to shrink fit the T-111 and measure the ΔT on several development capsules, using internal electrical heaters as a simulated fuel source.

Since shrink-fitting of the T-111 was selected, an analysis of the structural implications of the shrink fit was conducted. The contact pressures associated with a shrink-fit assembly of the T-111 structure capsule to the Ta-10W fuel liner were calculated. Definition of the contact pressure between the components as a function of interference is shown in Figure III-1. The total interference is composed of initial interference and that due to differential thermal expansion. The interference between the two components due to the latter is given in Figure III-2. The contact pressure due to differential temperature is plotted in Figure III-3. The tangential stresses in the components are plotted in Figure III-4 as a function of contact pressure. Since it

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Figure III-1. Contact Pressure vs Interference, T-111 Outer Shell Structure and Ta-10W Outer Liner Sleeve

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Figure III-2. Interference Due to ΔT vs ΔT -T-111 Outer Shell
Structure and Ta-10W Outer Liner Sleeve

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Figure III-3. Contact Pressure Due to ΔT Between T-111 Outer Shell Structure and Ta-10W Outer Liner Sleeve vs ΔT

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Figure III-4. Contact Pressure vs Interference, Outer Liner to Retaining Ring

has been proposed to also shrink-fit the outer fuel liner to the first retaining ring to reduce the gap for fuel leakage, the associated contact pressure for this case was considered and plotted in Figure III-5. The maximum allowable interference without retainer ring or liner buckling was analyzed and determined to be more than 4 mils radially at room temperature. From the pressure vs interference curves, the effect of temperature may be seen by comparing the room temperature curve with that of 2000°F. The curves show that the contact pressure for a constant interference is reduced by increased temperature. This effect is produced by the reduction of the elastic modulus. The effects of creep on the contact pressure were not considered in this analysis.

A preliminary thermal analysis has also been accomplished on the T-111 shrink fitting assembly sequence to define steady state cooling requirements and the transient response of the capsule and chill block assembly. Assembly will be accomplished by cooling the fuel liner assembly in a water cooled chill block and heating the T-111 in an electrically heated fixture. To assemble, the chill block is released from the inner capsule, and the inner capsule is then rapidly inserted into the structural shell. The heaters are turned off and the T-111 fixture is cooled by circulating water through the block. The chill block starts to cool and the inner capsule starts to heat up. The temperature response depends on the heat capacity of the components. Equilibrium is reached after thermal contact is obtained and the transients have died out. The equilibrium capsule temperatures depend on the contact resistance between the fuel outer liner and the T-111 and between the T-111 and the chill block.

The cooling requirements for both chill blocks are essentially the same. The chill blocks should have a uniform temperature to minimize temperature gradients around the capsule. The inside surfaces of the chill blocks must be carefully machined or honed so that a good mating surface with the capsule is provided and the temperature difference between the chill block and capsule is minimized in the vacuum.

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Figure III-5. Liner Tangential Stresses vs Contact Pressure

Temperature gradients in the chill block depend on the coolant tube spacing and the cooling flow rate. A water flow of 1 gpm will result in temperature gradients of less than 5°F in the proposed fixtures. The 1 gpm water flow will require less than 1 psi pressure drop in the fixture and results in a film temperature drop between the coolant and tube walls of only 3°F. The biggest uncertainty is the temperature difference between the capsule surface and the chill block during vacuum operation. The contact thermal resistance depends on the degree of mating between the two surfaces, the surface finishes, and the contact pressure. Because of the uncertainty in the degree of mating and lack of contact resistance data for vacuum operation of this geometry, the temperature difference could not be calculated. However, test measurements on the capsules with internal heaters will provide this needed information.

The maximum transient temperature response of the inner capsule assembly and the T-111 chill immediately after assembly were calculated using the data in Tables III-1 and III-2. The maximum rate of temperature rise for the inner shell assembly with isotope fuel is 126°F/min. With the electrical heater simulating fuel the maximum rate is 236°F/min. The maximum rate of decrease in the T-111 chill temperature is 13°F/min.

These rates of temperature change are not expected to cause problems during this sequence because the insertion and positioning of the inner capsule inside the structural shell will take only a few seconds. However, it is emphasized that this analysis does not include the potential effects of contact between the inner capsule and the structural shell due to cocking during the insertion and positioning operation.

The capsule design calls for line-to-line contact to 0.001 in. interference between the Ta-10W outer liner and the T-111 structural shell to assure contact between these two materials and reduce the temperature difference between them. The temperature difference depends on the contact pressure caused by the interference, the degree of mating, surface finish, etc. However, if the

TABLE III-1

INNER CAPSULE ASSEMBLY SUMMARY

Component	Weight - lb	C_p	MC_p - BTU/°F
Inner Liner	.22	.035	.0077
Outer Liner	.39	.035	.0137
Rings	.06	.035	.0021
Honeycomb (assumed 10% Theoretical Density)	.06	.035	.0021
Sub-Total	.73		.0256
Isotope Fuel	1.13	.06	.0678
Fueled Inner Capsule Total	<u>1.86</u>	-	<u>.0934</u>
Heater	(0.69)		(.0242)
Rods	.07	.035	.0025
Plates	.02	.035	.0007
Sheath (heated length)	.27	.035	.0095
Sheath (terminal end)	.08	.035	.0028
Wire (heater length)	.18	.035	.0064
Wire (terminal end)	.07	.035	.0025
Beryllia	.01	.2	.002
Inner Capsule with Heater Total	<u>1.42</u>	-	<u>.05</u>
Initial surface temperature, T_c	70° F		
Internal heat generation, Q_g	204 watts		
Effective surface area, A_c	0.172 ft ²		
Initial structural shell temperature, °F	500° F		
Configuration Factors, F_{c-s}	.0525		

TABLE III-2

T-111 SHELL CHILL BLOCK ASSEMBLY SUMMARY

Component	Weight- lb	C_p	MC_p - BTU/°F
Structural Shell	1.7	.035	.06
Chill Block	7.7	.10	.77
	<hr/>	<hr/>	<hr/>
TOTAL	9.4	-	.83
Initial temperature, T_s, T_b		500°F	
Effective surface area of structural shell, A_s		0.172 ft ²	
Effective surface area of copper block, A_b		0.566 ft ²	
Vacuum vessel wall temperature, T_w		70°F	
Configuration Factor, F_{b-w}		0.8	

coefficient of thermal expansion for T-111 is higher than that for Ta-10W, the T-111 will want to grow away from the Ta-10W, leaving a potential gap. The Ta-10W temperature will have to increase the required amount to close the gap. The temperature difference then becomes a function of the gap (200°F/mil) and the contact resistance.

Figure III-6 shows thermal expansion data for the two materials under consideration. The thermal expansion coefficients are very close and perhaps are the same; however, there is an indication that T-111 is slightly higher. If so, for line-to-line fit up, there would be the tendency for a gap and for the temperature difference between the T-111 and Ta-10W to adjust itself to just close the gap and then satisfy the contact resistance criterion.

2. Platinum Clad Design and Assembly Criteria

It is desired to have He in the gap between the Pt-10Rh oxidation clad and the T-111 shell to reduce the ΔT across this gap. However, it must be assured that the stresses induced in the clad by this He pressure do not lead to excessive creep of the clad over the capsule lifetime. A criterion of 1% creep in 7 years has been assumed. Creep data for Pt-10Rh is limited. However, some data was presented in Reference III-8. Reference 9 shows stress levels that will produce 1% permanent elongation for several temperatures and times up to 1000 hours. This data for 10, 100, and 1000 hours is plotted on Figure III-7. To obtain the 2000°F stress levels for 1% creep, the given data was extrapolated as shown. Then, to obtain a creep value for 2000°F and 61,350 hours, the values from Figure III-7 were replotted on Figure III-8 along with further extrapolation beyond 1000 hours. From this curve, a stress level of 75 psi is selected as the average working stress for 2000°F.

Due to the limitations of available data, it is recommended that additional creep data for stresses ranging from 50 to 150 psi at 2000°F and extended times be obtained. Note that the extrapolation over 2 decades (Figure III-8) to obtain the value of 75 psi leaves considerable doubt as to this value. The actual value could range between 50 and 150 psi.

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Figure III-6. Thermal Coefficient of Expansion for T-111 and Ta-10W

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Figure III-7. 1% Permanent Elongation Pt-10Rh Stress vs Temperature

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Figure III-8. Stress Time to Produce 1% Permanent Elongation, Pt-10Rh

Treating the Pt-10Rh clad as a thin walled cylinder the stress in the clad is equal to 42 times the internal He pressure. Therefore to meet the 1% creep criterion the He pressure at operating temperature must be 1-3 psi or lower. Various methods of reliably obtaining this pressure at temperatures are currently being evaluated. Candidate approaches include combinations of insertion pressure and temperature with volume control by dimensional tolerance, creep forming or shrink fitting.

3. Pressure-Electrical (P-E) Feedthru Design

The pressure-electrical feedthru tube will be welded to the outer liner and shell of the capsule and be subjected to full test pressure and temperatures. Since it is basically a test fixture, it is desirable to have its Margin of Safety high with respect to the tested component. In this case, a factor of safety of two compared to the structural shell should be adequate.

The tests for which the pressure-electrical feedthru tube functions will be the long time pressure-temperature creep tests. The Ta-10W and T-111 materials have very similar short time tensile and high temperature creep properties. In the range of 2000°F, the creep properties of the T-111 show about a 10% improvement over the Ta-10W. Accordingly, the Ta-10W tube and attachment will be based on a minimum strength of 2.20 times that of the T-111 shell.

Using the above criterion and a P-E feedthru I.D. of 0.215 inches (set by instrumentation requirements), a minimum wall thickness of 0.0625 inches has been established.

The attachment of the tube to the shell should be designed to minimize the complexity of stresses in the weld and to assure an adequate stress path around the tube. The integrally formed boss described in the capsule design section meets these criteria.

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IV. CAPSULE DESIGN

A. TASK OBJECTIVES

The objectives of this task are to design and prepare all the drawings necessary to completely define the engineering, fabrication, and testing requirements of the Large Radioisotope Heat Source Capsule program. This encompasses piece part drawings, assembly drawings, burial heater design, and assembly, welding, and test fixture design.

A major portion of this task is evaluation of the fabricability of the design and preparation of a complete tolerance study on all capsule configurations.

B. SUMMARY OF TASK STATUS

The first milestone of the LRHSC program was met when the reference capsule drawings were released on August 15, 1967. The test capsule drawings were also completed, approved and released in late September.

The coupon compatibility part, assembly and test fixture drawings are completed and fabrication has begun.

The annealing furnace design and fabrication is completed and the furnace has been delivered to test.

The creep capsule test fixture designs are about 25% complete.

C. ACCOMPLISHMENTS DURING THE QUARTER

The original LRHSC capsule design as shown in Figure IV-1 has undergone several significant design changes to enhance capsule performance, improve fabrication, or alleviate assembly problems. However, the basic capsule geometry, materials and materials thicknesses have not changed. The inner liner assembly is constructed of 0.020-in. thick tantalum-10% tungsten (Ta-10W) alloy and provides: (1) structural support for the isotope fuel and the honeycomb impact absorber and, (2) a selective leak to the center of the capsule for

Figure IV-1. Reference Capsule Design in RFP
(Request for Proposal)

Figure IV-2. Large Radioisotope Heat Source Capsule
Liner Assembly

helium buildup. The outer liner assembly is constructed of 0.020-in. Ta-10W and provides a leak tight structural container for the fuel and honeycomb impact structure. The tantalum-111 (T-111) shell assembly surrounds the inner capsule assembly and provides the primary strength for long-term He containment and for impact survival. The T-111 structure is coated with a 0.002-in. diffusion barrier of alumina. The structural shell is surrounded by a 0.020-in. oxidation barrier of platinum-10% rhodium (Pt-10Rh) coated with a 0.004-in. emittance coating of iron titanate ($\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$). Tungsten-molybdenum (W-Mo) is the fuel simulant specified for the tests in this program. The present reference capsule design is shown in Figures IV-2 and -3. Approximately 90% of all capsule drawings have been completed including the impact and burial designs which will not be released until GFY'69.

The following changes have been made to the original capsule design:

- 1) The inner liner and outer liner were changed from 2 piece construction to 3 piece construction. This will eliminate a deep draw or spinning operation. The stepped weld joints were incorporated to replace the butt joint. This will aid in the assembly and tooling operation.
- 2) The inner liner cap and ring have been made as an integral part which will allow for more accurate positioning and machining.
- 3) The Ta-10W Outer Liner Cap will be assembled to the Inner Liner Assembly with a surface/surface to .001 total indicator reading (TIR) interference fit on the diameter to reduce the possibility of fuel leakage through the stitch weld.
- 4) The Ta-10W Fuel Closure Ring will be assembled to the Outer Liner Sub-Assembly at 200° F with a surface/surface to .001 TIR interference fit on the inside diameter to the Inner Liner Capsule, and a surface/surface fit to .001 TIR interference fit on the outside diameter to the Outer Liner Sleeve. These changes reduce the possibility of fuel leakage and provide positive fitup for the outer fuel liner closure weld.

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Figure IV-3. Large Radioisotope Heat Source Capsule Assembly

- 5) The T-111 Structural Shell will be assembled to the Ta-10W Capsule at 200° F with a surface/surface to .001 TIR interference fit in order to reduce the ΔT between these two components in an internally heated (fueled) capsule.
- 6) A .002 to .004 inch trough was added to the outer flange of the fuel closure ring to provide a void for weld suck back. This should prevent a weld buildup on the outside diameter of the outer fuel line.
- 7) The Ta-10W feedthru tube dimensions have been changed to .215 I.D. x .340 O.D. This change was necessary to provide a greater stress margin between the T-111 structure and the Ta-10W feedthru tube.
- 8) The feedthru structure cap was changed to incorporate a formed boss which will be butt welded to the inner feedthru tube. This change was made to improve the feedthru weld joint design and to simplify fabrication.
- 9) The Pt-10Rh sleeve between the Barrier cap and feedthru tube was eliminated. Due to the changes in assembly procedures, the feedthru tube can be inserted directly into the Barrier cap and welded.
- 10) To reduce weld tooling costs, the 3 automatic welds on the platinum end cap to platinum sleeve weld and insert and the platinum tube to the stainless-platinum weld insert have been changed to hand welds. These welds are seal welds and do not affect the mechanical strength of the capsule.

V. PROCESS DEVELOPMENT

A. TASK OBJECTIVES

The objective of the process development task is 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 and joining. Development of coatings is being conducted separately and is reported in Section VIII. Studies of alternate materials, fabrication, and assembly techniques will be performed as required.

B. SUMMARY OF TASK STATUS

Specifications for semi-finished and finished tantalum and platinum alloy stock have been written and were reviewed with potential suppliers of material for the program. Plant visits were made to each of the potential tantalum alloy suppliers for this review and to evaluate their capabilities in meeting the requirements of the LRHS capsule program. The Metals Division, National Research Corporation of Newton, Massachusetts, was selected to provide the tantalum alloy semi-stock for the program. J. Bishop and Company, Platinum Works of Malvern, Pennsylvania, was chosen for Pt-10Rh semi-stock fabrication.

Annealing and cleaning specifications for the tantalum alloys have been prepared and the Pt-10Rh cleaning specification is being written.

A detailed plan for development of welding procedures and qualification of critical welds is being prepared. A quality assurance plan and non-destructive testing development plan are being prepared in conjunction with the weld plan.

Microbrazed alloys have been selected for joining Pt-10Rh to Ta-10W and Silcoro-60 or -75 selected for joining Ta-10W to Type 304 stainless steel.

The Metals Division of NRC is in the process of determining the feasibility of back extruding a T-111 cup with an L/D ratio of approximately 7:1. This extrusion work is being performed by Torrance Extrusion Corporation of Banning, California and is a backup to the presently planned deep drawing to form the T-111 structural shell.

To select the best energy absorbing material for the impact absorbers, both foamed and fibrous Ta-10W are being evaluated as alternates to the honeycomb construction.

C. ACCOMPLISHMENTS DURING THE QUARTER

1. Raw Materials

a. Tantalum Semi-Stock

The potential suppliers of Ta-10W and T-111 were visited in preparation for soliciting bids for semi stock for the LRHS capsule program. The requirements of the program were presented and the suppliers' capabilities were evaluated. Specifications were reviewed and the material quality now being supplied on a routine basis was factored into suggested revision of the documents. Those requested revisions which did not compromise the objective of the program or material quality were incorporated into the appropriate specifications. The contract for the tantalum alloys semi stock was awarded to National Research Corporation, Metals Division, Newton, Massachusetts.

As of September 21, NRC reports that all of the required tantalum alloy ingots have been fabricated and conditioned. This includes backup material in the event of unanticipated losses during fabrication. One of the T-111 ingots is shown in Figure V-1. It is currently being forged to sheet bar.

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Figure V-1. T-111 Billet (960 lb) Produced by NRC for LRHS Capsule
Fabrication

At the time the order was placed the dye penetrant and helium leak testing facilities at NRC had not been completed for this order. As of September 21 the leak checking facility was installed, and the dye penetrant facility was scheduled for completion on October 1. All testing will be done by NRC with the exception of the ultrasonic tests. These will be performed by Automation Industries of Columbus, Ohio.

b. Platinum Semi-Stock

Pt-10Rh semi stock specifications were reviewed with potential suppliers. Those requested changes which did not jeopardize the objectives of the program or material quality were incorporated into the purchasing documents. The contract for the stock was awarded to J. Bishop and Company.

c. Back Extrusion

The Metals Division of NRC placed an order with Torrance Extrusion Corporation (TEC) to determine the feasibility of back extruding the T-111 structural shell. The fabrication schedule would be to back extrude an over-size blank and machine the part to final size. For the actual shell the blank would have an L/D ratio of approximately 7:1. For the purpose of the feasibility work, available tooling was used where possible.

A part formed with an L/D of 4:1 in the initial trials is shown in Figures V-2 and V-3. TEC reported to NRC that the length of this part was limited by the die container which was too deep for the application. The greatest loads occur in the upsetting and then decrease while the cup is being formed. Therefore, TEC feels that the 4:1 ratio cup established the feasibility of the 7:1 ratio cup. One problem that did occur was excessive ram wear. This problem must be worked out before the process is considered a production procedure.

The part is now being examined by NRC. When the examination is completed, the part will be shipped to AI for further evaluation. Tests are planned to evaluate surface and internal contamination, microstructure and ductile-brittle transition temperature (DBTT) shift.

Figure V-2. T-111 Cup Formed by Warm Back Extrusion at Torrance Extrusion Corporation

Figure V-3. Results of Three Back Extrusion Trails to Form the Structural Shell. A. Approximately 1/2 in. Penetration, B. Approximately 2-1/2 in. Penetration. C. 3-1/3 in. Penetration. Dimensions of Part C are shown in Figure V-4.

d. Impact Absorbers

The impact absorbers placed in the capsule ends between the inner and outer liners are currently specified as a Ta-10W honeycomb material. Since the functional requirements for these parts is to be a crushable energy absorber, two other materials forms which are potentially less expensive and superior energy absorbers, are being evaluated. Astro Met Associates, Inc. of Cincinnati is supplying a 10% dense foamed Ta-10W structure and Hyck Metals Company of Milford, Connecticut is determining the feasibility of making a 10% dense fibrous Ta-10W body. Both material forms will be evaluated for crushing strength, dimensional control, interstitial content and cost. A decision on which of the three forms best meets the LRHSC program requirements will be made next quarter.

2. Joining

a. Welding

A test plan to define the necessary development of welding procedures and qualification of critical welds is being prepared.

Included in this test plan are six (6) major categories: 1) material requirements, 2) welding approach, 3) testing methods, 4) welding processes, 5) cost analysis, and 6) schedule. The test plan is intended primarily as a guide for weld development.

Equipment requirements necessary for the weld program, i. e., fixturing and welding apparatus, are either being designed or are presently on order. In addition, the materials required for development are on order, and are expected to be received early in the next quarter. As soon as development material arrives preliminary welding development will begin using the existing 3 kw electron beam welding machine. The new 6 kw electron beam welding machine has a scheduled shipping date of October 15. Developmental work on the new electron beam welder is scheduled to begin about November 15. This time lag is for setup and testing of the apparatus.

Quality assurance for the welding development has been established and preliminary testing methods have been selected. During the weld development all welds are accessible for any required inspection tests. However, not all welds are accessible during production. Thus, qualification standards will have to be set to insure reproducibility and reliability and non-destructive testing techniques will require development for some of the critical welds.

b. Brazing

The present design of the LRHSC capsule pressure-electrical feedthru (PEFT) requires brazing of a Pt-10Rh alloy circular sleeve to a stainless steel flange. After the Pt-10Rh sleeve has been TIG welded to another Pt-10Rh sleeve, a second brazing operation is required to join an inner Ta-10W alloy tube to the same stainless flange. Brazing alloys with low vapor pressures such as used for brazing vacuum tube components are desired.

Two brazing alloys were selected for the stainless steel to platinum alloy joint. Three brazing alloys were selected for the tantalum alloy to stainless joint. The trade names and constituents are listed in Table V-1.

TABLE V-1

BRAZING ALLOYS SELECTED FOR THE PEFT

Braze Alloy Trade Name	Constituents	Application
Nicoro-80	Gold-Copper-Nickel	Pt-10Rh to 304 SS
Nioro	Gold-Nickel (Eutectic)	Pt-10Rh to 304 SS
3% Ti Wire	Silver-Copper (Eutectic with Titanium Wire Core)	Type 304 SS to Ta-10W
Silcoro-60	Gold-Copper-Silver	Type 304 SS to Ta-10W
Silcoro-75	Gold-Copper-Silver	Type 304 SS to Ta-10W

Wettability brazing tests were run for each base metal binary system by placing small pieces of the appropriate, clean braze alloy on a clean specimen of the corresponding base metal alloy and then heating the assemblies to brazing temperature in a high vacuum furnace. Where appropriate a small coil of either Type 304 stainless steel wire or Pt-13Rh wire was placed on top of the brazing alloy foil to check the wetting characteristics of these binary systems. The stainless specimens with Nicro and Nicoro braze foils were held 10 minutes at 1850°F. The others were heated to 1700°F and held for 12 to 13 minutes at the brazing temperature. Approximately 100°F of superheat and longer than normal brazing periods were used intentionally to identify any undesirable features such as excessive braze alloy diffusion and/or the formation of hard phases at the base metal braze interface.

Simulated step brazing tests were conducted after the wettability test result had been evaluated. To simulate the first brazing operation small coils of 0.030 inch diameter Pt-13Rh wire were brazed near the edge of sheets of Type 304 stainless steel using in one case 2 mil thick foils of Nicro braze alloy, and in another case, 2 mil foils of Nicoro-80 braze alloy. These were vacuum brazed for 10 minutes at 1850°F. Then disks of the Ta-10W alloy were inserted between the stainless steel sheets with 2 mil thick Silcoro 60 brazing foils between the Ta-10W and the stainless steel surfaces. Finally, these assemblies were heated in vacuum for 13 minutes at 1730°F to braze the Ta-10W alloys to the stainless steel sheets.

These exploratory brazing tests were evaluated by metallographic inspection of sections of the brazes, by an estimate of the wetting contact angles, and by microhardness traverse tests across the joints. Also bend tests were performed with thin sections cut from specimens from the last two runs (Ta-10W to Type 304 SS with 4 mils of Silcoro-60, and Ta-10W to Type 304 SS with 4 mils of Nicro braze). The following conclusions were reached:

1) A 3% Ti cored-Pt brazing alloy is not suitable for brazing to stainless steel due to the formation of a hard reaction layer at the interface.

2) NIORO brazing alloy does not appear suitable for brazing to Ta-10W alloy due to the formation of a hard reaction phase at the Ta-10W surface as shown in Figure V-4.

3) A NIORO brazing alloy would be suitable for brazing Pt-10Rh alloys to Type 304 stainless steel. Excessive diffusion of the NIORO into the base metal alloys occurred under the conditions of these tests as seen in Figure V-5 but this can be controlled by reducing the brazing temperature and/or time at temperature. There is need to investigate further the hardening effect of the NIORO upon the platinum-rhodium alloy to insure that brittle phases are not formed.

4) Either Silcoro-60 or Silcoro-75 is suitable for brazing Ta-10W to Type 304 stainless steel. The microstructures of the braze to Type 304 stainless steel interface shows some intergranular penetration of braze alloy into the stainless as seen in Figure V-6 and V-7 but this did not appear to be objectionable. Both alloys appeared to wet the Ta-10W alloy very nicely, but the Silcoro-75 showed a lower angle of wetting. No hard phases were found in either alloy joint.

Bend tests of a Ta-10W to Type 304 stainless joint brazed with Silcoro-60 verified that a strong, ductile joint can be obtained with this brazing alloy.

5) Nicoro-80 brazing alloy would require testing at a lower brazing temperature and/or for a shorter period of time to evaluate its merits for brazing Type 304 stainless steel to platinum-rhodium. Excessive diffusion occurred at 1850°F with this brazing alloy.

304 SS

NIORO

Ta-10W

Figure V-4. Ta-10W Alloy Brazed to 304 Stainless Steel Sheet with NIORO Brazing Alloy. Brazed at 1850°F for 10 Minutes. Etched: Oxalic-Electrolytic. 500X

Met. 7992-2-1
7734-2517

Pt-13Rh Wire

NIORO

304 SS Sheet

Figure V-5. Type 304 Stainless Steel Sheet Brazed to a Pt-13Rh Wire with NIORO Brazing Alloy, 10 Minutes at 1850°F., then Reheated 13 Minutes at 1730°F. for a Silcoro-60 Step Braze Elsewhere. Etchant: 50% HNO₃; 50% HF 100X.

Met. 7986-9-1
7734-2518

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304 SS Wire

Silcoro-60

Ta-10W

Figure V-6. Ta-10W Alloy Brazed to a 304 SS Wire with Silcoro-75 Brazing Alloy
Met. 7986-4-1. Etched: Oxalic-Electrolytic. 500X.
7734-2519

304 SS Wire

Silcoro-75

Ta-10W

Figure V-7. Ta-10W Alloy Brazed to a 304 SS Wire with Silcoro-75 Brazing Alloy
Met. 798602-1. Etched: Oxalic-Electrolytic. 500X.
7734-2520

VI. 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

All material requirements for the LRHS capsule fabrication were established and the purchase order placed. All material required for the total program is being purchased at one time to provide control on the alloy heats and assure uniformity of properties between the test capsules. Delivery dates for the tantalum alloys is about November 15, 1967 and for the Pt-10Rh material about October 20, 1967. Early delivery of material of similar composition has been arranged so that the piece part fabrication development, weld development and the first compatibility test may be started while the actual test hardware material is being procured.

Piece part fabrication bid packages have been sent out for bid and the purchase order will be placed early in the next quarter.

The quality control requirements of the program have been established and a Quality Control Plan drafted.

Assembly and weld tooling requirements have been defined. Tooling design is about 75% complete. Bid packages have been prepared on completed tooling designs.

Four stainless steel capsule mockups of the reference design are being fabricated and are 50% complete. These will be used for tooling checkout.

C. ACCOMPLISHMENTS DURING THE QUARTER

The total capsule fabrication effort required for the LRHSC Program is outlined in Table VI-1.

TABLE VI-1

LRHS CAPSULE FABRICATION REQUIREMENTS

AI Drawing Number	Type of Capsule	No. Required FY'68	No. Required FY'69	Total Program Requirements
N73420042	Creep "A" with Feedthru	2	2	4
N73420043	Creep "A" without Feedthru	1	0	1
N73420044	Creep "B" with Feedthru	7	4	11
N73420045	Creep "B" without Feedthru	1	0	1
N73420046	Creep "C" with Feedthru	5	3	8
N7342247	Creep "C" without Feedthru	1	0	1
N73420048	Impact Capsules "A"	0	9	9
N73420049	Impact Capsules "B"	0	6	6
N73420050	Earth Burial "C"	0	2	2
TOTAL		17	26	43

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The "type of capsule" designations in Table VI-1 are explained in Table VI-2.

TABLE VI-2
LRHS CAPSULE TYPES

Type of Capsule	Description
Creep A, Impact A	T-111 Structural Shell with Inner Fuel Assembly
Creep B, Impact B	T-111 Structural Shell Only
Creep C, Burial C	Complete Capsule

The piece part requirements to fabricate the GFY'68 capsules are shown in Table VI-3. Based on these requirements, the material type, shape and quantity of raw materials and semi-stock were established. National Research Corporation received the order for all tantalum material and Bishop Company, Inc. the order for the Pt-10Rh material. Delivery for the tantalum alloys is 11 to 13 weeks and for the Pt-10Rh 6 to 8 weeks. Negotiations are being conducted to expedite these deliveries for "on schedule" availability of all material for GFY'68 piece part fabrication.

The T-111, Ta-10W, Pt-10Rh and tantalum material required for fabrication of coupons and coupon holders to put four (4) coupon stacks on test during October, was expedited for delivery. The material was received and the coupons and holders are presently being fabricated.

Quality Control requirements for the LRHSC program were established and the first draft of the Quality Control Plan completed.

Bid packages for all GFY'68 piece part fabrication have been prepared and distributed.

TABLE VI-3
LRHS CAPSULE PIECE PART FABRICATION EFFORT GFY'68

AI Part Number	Title	Mat'l Type	Number Required			Method of Fabrication
			Development	Test	Spares*	
N73420002	Barrier Shell	Pt-10Rh	2	6	2	Spinning or deep drawing
N73420003	Barrier Cap	Pt-10Rh	1	1	1	Coining
N73420005	Structure-Shell	T-111	3	17	5	Warm-back extrusion or deep drawing
N73420006	Structure-Cap	T-111	1	3	2	Coining
N73420008	Outer Liner Cap	Ta-10W	3	9	3	Coining
N73420009	Stepped Honeycomb	Ta-10W	3	9	3	Electron Beam Welding
N73420010	Fuel Closure Ring	Ta-10W	3	9	3	Spinning
N73420012	Outer Liner Sleeve	Ta-10W	3	9	3	Extrusion
N73420013	Honeycomb	Ta-10W	1	2	1	Electron Beam Welding
N73420014	Outer Liner Stepped Cap	Ta-10W	1	2	1	Coining
N73420016	Inner Liner Flanged Cap	Ta-10W	1	2	1	Coining
N73420018	Inner Liner Stepped Cap	Ta-10W	3	9	3	Coining
N73420019	Inner Liner Sleeve	Ta-10W	3	9	3	Extrusion
N73420021	Feedthru Barrier Cap	Pt-10Rh	1	5	1	Coining
N73420024	Feedthru Honeycomb	Ta-10W	2	7	2	Electron Beam Welding
N73420026	Feedthru Outer Liner Stepped Cap	Ta-10W	2	7	2	Coining
N73420027	Feedthru Structure Cap	T-111	2	14	3	Coining
N73420029	Feedthru Inner Liner Flanged Cap	Ta-10W	2	7	2	Coining

*Will be used for subsequent fabrication

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Three basic categories of tooling are necessary for capsule fabrication; these are assembly, electron beam weld, and TIG weld tooling. Secondary tooling requirements such as interim storage tools to keep the capsule cool during inspection are also being considered.

The assembly tooling will be a small hand press with various sized collets to permit axial alignment and location prior to the components installation in the electron beam weld tool.

The electron beam weld tool for the fueled subassembly is being designed to afford versatility in its use. The tool will be a belt driven through a collet fixture with changeable collets to permit all electron beam weldments prior to the fueling to be performed in this basic tool.

A fueling chill block is being designed to allow simulation of the fueling of the capsule, and press fitting of both the fuel closure ring and the outer liner end cap. This tool will have a mounting platform for the automatic TIG welding equipment which will be used to perform the stitch weld on the fuel closure ring and the seal weld of the outer liner cap. The tool will be machined from copper with integral cooling passages for water. To prevent contamination of the capsule, the chill block cavity will be covered with vapor deposited tungsten.

One difficult assembly operation is the press fit of the outer capsule assembly to the T-111 structural shell and electron beam welding of the cap to complete the structural capsule assembly. To accomplish this, a tool is being designed which will chill the fueled capsule, heat the structural shell, push the capsule into the shell by means of a pneumatic piston, and install the structural shell cap. The assembly is then cooled to room temperature and electron beam welded. This entire sequence is performed in the electron beam welding chamber. Analysis of the temperature difference required between the structural shell and the liner subassembly to permit the capsule to be inserted into the shell indicates that approximately 400° F will be sufficient.

VII. CAPSULE CREEP TESTING

A. TASK OBJECTIVES

The LRHSC Program fuel capsule utilizes a first generation tantalum alloy (Ta-10W) as a primary containment for the fuel, a second generation tantalum alloy (T-111) for secondary containment, and Pt-10Rh for oxidation protection of the reactive refractory metal containers.

During the first year of the program three biaxial creep rigs will be operated to test the T-111 containment vessel alone, the vessel in contact with Ta-10W, and the total capsule with oxidation barrier over the compatibility coatings. At the end of the first year preliminary creep data for test times less than 2000 hours will have been obtained.

During the second year additional creep rigs will be utilized and longer term creep data will be obtained on the three capsule configurations.

B. SUMMARY OF TASK STATUS

Three creep rigs will be required for the first year's test operation. Three 1200 liter/sec vacuum ion pumps and vacuum chambers with power supply and vacuum readout are in the LRHSC laboratory ready for assembly and checkout (See Appendix A). Redundant power and water supplies are installed along with liquid nitrogen source.

The design of the pressure control system is 98% complete. The three systems required for this year are under construction. All of the required components has been ordered and approximately 80% have been received.

The high pressure (6000 psi), high purity (99.9999%) helium gas supply requirements have been defined and supply sources located.

A review of the temperature measuring requirements is complete. Tests to determine the required power control system have been defined.

Strain measuring requirements and techniques for measuring creep are under study. Final design of the capsule fixturing and jigging is being held up pending furnace checkout and definition of the strain measuring technique to be used.

C. ACCOMPLISHMENTS DURING THE QUARTER

The creep capsules which are planned to be tested this year are indicated in Figure VII-1. Also shown is the sequence of testing and the utilization of three creep test rigs. The test conditions for the creep tests are shown in Table VII-1.

The major activity during this quarter has been obtaining components for test operation. Three 1200 liter/second ion pumps have been installed in the LRHSC laboratory for creep test operation during the first year. Presently mounted on the pumps are single wall metal vacuum chambers cooled by brazed copper tubing. These jars will be modified for the creep tests. Final design of the test setup, however, has not been completed. The components for the ion pump power, bakeout, sorption pumping, and vacuum instrumentation are in the laboratory but need to be assembled and checked out. Modification of the chambers will take place while the necessary repairs are being made on the pumps and instrumentation. The basic components are available but considerable work remains before they will be operational.

1. Temperature Instrumentation

A detailed review of the temperature measuring instrumentation for the creep tests to meet the requirements of $2000^{\circ}\text{F} \pm 5^{\circ}\text{F}$ over the entire creep capsule surface has been performed. (The results of this review also apply directly to the coupon compatibility tests). A summary of the recommendations of the study are:

- 1) Use bare Pt vs Pt-Rh or W vs W-Re thermocouples to instrument the capsule.

ALL TYPE-C CAPSULES ARE IN ONE RIG TO ISOLATE ANY IRON TITANATE VAPORIZATION PROBLEM THAT MAY DEVELOP. THIS RIG MUST BE AT LEAST 1200 LITERS.

THE B-TYPE HEAT-TO-HEAT STUDIES WILL ALL BE PERFORMED IN THE SAME RIG.
EACH TEST INCLUDES ONE WEEK FOR SETUP AND DISASSEMBLY.

○ FULL CAPSULES WITH Al_2O_3 AND Pt-10Rh SHELL.

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Figure VII-1. Creep Rig Utilization

TABLE VII-1
LRHSC CREEP CAPSULE TEST CONDITIONS

Test No.	Capsule No.	Nominal Test Time Hours	Helium Pressure		
			P ₀ (psi)	dp/dt (psi/hr)	P _{Final} (psi)
1	A-1a	158	3390	10.6	4450
2	A-2a	1,480	2440	.96	3400
3	A-3a	8,800	1750	.138	2578
4	A-8	10,000	0	.185	1850
5	A-Std	--	--	--	--
6	B-1a	158	2890	9.02	3792
7	B-1b	158	2890	9.02	3792
8	B-2a	1,480	2080	.82	2900
9	B-2b	1,480	2080	.82	2900
10	B-3a	8,800	1490	.118	2198
11	B-3b	8,800	1490	.118	2198
12	B-4	228	2890	9.02	4423
13	B-5	2,280	2080	.82	3556
14	B-6	13,800	1490	.118	2788
15	B-7	2,680	2080	.82	3884
16	B-8	10,000	0	.157	1570
17	B-Std	--	--	--	--
18	C-1a	158	3390	10.6	4450
19	C-2a	1,480	2440	.96	3400
20	C-3a	8,800	1750	.138	2578
21	C-4	228	3300	10.6	5192
22	C-5	2,280	2440	.96	4168
23	C-6	13,800	1750	.138	3268
24	C-7	2,680	2440	.96	4552
25	C-8	10,000	0	.185	1850
26	C-Std	--	--	--	--

* These times to test completion are approximations. The actual values are entirely dependent on strain measurements made during testing.

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2) Arrange a means for checking the calibration in-situ with a reference thermocouple with an accuracy of $\pm 0.6^{\circ}\text{F}$, which is not exposed to elevated temperatures other than during calibration.

3) Use an automatic optical pyrometer to cross check the calibration.

4) Use readout instrumentation of sufficient accuracy such that it will not significantly contribute to the $\pm 5^{\circ}\text{F}$ allowed.

The rig design will incorporate these recommendations.

2. Temperature Attainment and Control

A furnace has been designed for the creep tests. It is a tantalum resistance heater utilizing Al_2O_3 insulators. It is 8" in diameter and 16" long and is shown in Figure VII-2. (This design will also be used for the annealing furnace). The furnace has been built and is now being checked out. It is presently planned to control the furnace by constant thermocouple output rather than constant power. The power supply design under consideration is a semi-proportion Variac. Tests are in progress which will indicate if the design and control are adequate. A more detailed discussion of the heater work is presented in Appendix B.

3. Pressure Control

The three pressure control systems being built are shown in Figure VII-3. A pressure gauge will be mounted in the large hole in the top of each panel. In operation the pressure gauge will present the total system pressure. System pressure will also be monitored by a differential pressure transducer. This transducer will sense the pressure difference between a reference pressure and the capsule pressure. An electrical voltage proportional to the pressure difference is applied to the pressure controller. At the same time a slowly increasing voltage is being supplied to the pressure controller from the ramp generator. This signal will be adjustable to produce the desired

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Figure VII-2. LRHSC Creep Test Heater

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Figure VII-3. LRHSC Creep Test Pressure Control Instrumentation Systems

rate of pressure increase. The controller compares these two voltages and supplies power to a motor when the voltages are different. The motor controls a needle valve that controls the gas flow into the capsule. To obtain the required pressure resolution a low range pressure transducer is used. This method requires periodic resetting of the reference pressure. Each time the transducer voltage reaches the maximum value, a system will increase the reference pressure. At the same time a motor will adjust a pressure regulator to increase the supply pressure. The recorder presents a record of the transducer output.

4. Gas Supply

The pressure control system will be fed by a 6000 psi helium bottle. It is presently planned to use one bottle per rig to minimize contamination of more than one specimen due to a malfunction of any one of the rigs. As operational experience is gained this may be found unnecessary.

A study is in progress to determine the required instrumentation and sampling techniques for gas purity monitoring.

5. Strain Measuring

The presently planned method for strain measuring during test is to sight optically the diameter at the longitudinal mid plane of the specimen. It would be desirable to obtain measurements of strain in more than one plane. Effort has been expended and will continue to work out acceptable measuring techniques for additional planes on the capsule. The early coupon tests will be used to determine some of the limitations and advantages of different methods.

VIII. COUPON COMPATIBILITY AND COATING DEVELOPMENT

A. COMPATIBILITY

1. Task Objectives

The 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 functions of time and temperature and to determine the chemical and structural effects the interactions have on the base material properties.

The design of the tests consists of placing smooth surfaces of the materials in intimate contact with each other and exposing them to high temperatures for fixed times in a vacuum. In order to reduce the effects of uncontrolled variables, strict material, fabrication, cleaning and annealing specifications will be adhered to for all coupon components. These specifications are identical to those for the fuel capsules.

The test conditions for the compatibility stacks that will be tested this year are shown in Table VIII-1. Inferences will be made regarding the extent of material interactions after 7 years at 2000°F based on the results of these tests. Next year, longer duration tests up to 13,800 hours at 2000°F will be conducted in conjunction with the creep program to refine the predictions based on this year's data.

2. Summary of Task Status

The coupon compatibility test specification has been written. The drawings of the coupon stack components and assembly have been completed. The coupon stack materials have been received and the coupons are being fabricated.

Two high temperature vacuum systems have been redesigned to provide the necessary hot zone with the required temperature uniformity and visual access. The annealing heater presently undergoing check-out will be used for the first compatibility test.

TABLE VIII-1

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	0 (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*	3-2-68	2000	10 ⁻⁸	2200
CS10*	3-7-68	2000	10 ⁻⁸	1800
CS11*	5-15-68	2000	10 ⁻⁸	1000

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

3. Accomplishments During the Quarter

Since the LRHS capsule compatibility tests are scheduled to begin in mid - October, activity this quarter has been directed to all phases of test preparation: test specification, sample fabrication, test equipment preparation, instrumentation, and heater design and testing.

The compatibility test specification which defines the test program and operating procedures has been written. The test specification calls out the materials and fabrication specifications, instrumentation requirements, test conditions and sample configurations.

Coupon part and stack assembly drawings have been released and the components are being fabricated. The basic coupon stack showing the various material combinations and dimensions is shown in Figure VIII-1. The coupon-holder assembly complete with identification markings and thermocouple locations is shown in Figure VIII-2.

The coupon compatibility tests will be performed in ion pumped vacuum systems containing high temperature tantalum heaters. The coupon stack assembly will be supported in the furnace on an alumina pedestal. The attached Pt-Rh thermocouples will be fed to a millivolt recorder through a vacuum port. Calibration of the thermocouples (and hence, the temperature controller) during testing will be accomplished by periodic measurement of the temperature of an attached tantalum black body fixture using an optical pyrometer through the shielded top view port.

The heater and vacuum system, which will be used for the initial compatibility test is currently being checked out.

After testing, each compatibility coupon stack will be examined in the following manner:

- 1) Post-test X-ray of the stack
- 2) Longitudinal sectioning, polishing, and preliminary metallography
- 3) Preparation of the sample for electron beam microprobe analysis (EBMA)
- 4) EBMA of the test stack to obtain line profiles of elemental concentrations across coupon interfaces
- 5) Microhardness traverses across the coupon interfaces
- 6) Final polishing and metallography in the etched and unetched condition.

DETAIL A

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Figure VIII-1. LRHSC Coupon Compatibility Test Stack

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Figure VIII-2. LRHSC Coupon Compatibility Test Assembly

The extent of the examinations will vary from sample to sample as reaction phases are identified, unusual observations are explored in depth, and as an understanding of the interactions is obtained. Additional techniques such as X-ray diffraction and electron micrography will be employed where needed to gain a fundamental understanding of the test results.

B. COATING DEVELOPMENT

1. Task Objectives

Two ceramic coatings are required for the LRHS capsules: 1) a high emittance coating on the external surfaces of the Pt-10Rh cladding, and 2) a diffusion barrier between the Pt-10Rh and the T-111 structural shell. The materials tentatively selected for these applications are iron titanate ($\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$) and alumina (Al_2O_3), respectively.

The objective of the Coating Development Task is to prepare material and process specifications for both coatings. To accomplish this for iron titanate will require evaluation of arc-plasma sprayed coatings for use at 2000°F on Pt-10Rh; the technology for this coating material is well established for other substrate materials and at somewhat lower temperatures. The first objective of the alumina coating program is to evaluate the various coating processes for their ability to produce α - Al_2O_3 without contaminating the T-111 substrate. Once α - Al_2O_3 coatings are obtained, they will be evaluated for performance at the LRHSC operating conditions. Finally, one process will be selected, and the specifications written.

2. Summary of Task Status

a. Iron Titanate

Specifications for iron titanate powder and for arc-plasma spraying of the powder onto a Pt-10Rh substrate have been written. The material specification has been released, but the process specification is being withheld pending the results of trial runs.

The arc-plasma spraying facility has been set up and preliminary trials have been performed using a 10 mil thick sheet of Pt-10Rh as the substrate. Visual observation and gross evaluation indicate that an adherent coating has been obtained.

b. Alumina

Three coating processes have been selected for evaluation: chemical vapor deposition (CVD), arc-plasma spraying (APS), and sputtering. Initial test results indicate that CVD coatings of α - Al_2O_3 can be produced with only slight substrate contamination, and that the sputtered coating produced is amorphous Al_2O_3 . The sputtering work has been terminated, since α - Al_2O_3 coatings are not state-of-the-art for this process. The necessary equipment for APS in a high purity inert gas environment with the substrate at 1200°F has been built, and trial runs are now in progress.

3. Accomplishments During the Quarter

a. Iron Titanate

The successful use of iron titanate at 2000°F for long periods of time in air on a Pt-10% Rh substrate has not been demonstrated. A development contract from NASA now beginning may yield this information. However, based on data obtained by Pratt & Whitney with other substrates and lower operating temperatures, Atomics International has written both an $\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$ powder material specification and an arc-plasma spraying process specification. Material conforming to the material specifications has been obtained, and trial runs to apply this material to a 10 mil Pt-10Rh substrate are being made. The samples produced appear to have a uniform, adherent coating. Evaluations presently in progress will measure the properties of the as-applied coating, and the process specification will be revised if necessary before release. Long term evaluations are planned to measure coating compatibility, emittance, and adherence after exposure to creep and to stress cycling at 2000°F for long periods. Thermal cycling tests are also planned.

b. Alumina

(1) Chemical Vapor Deposition

Any of several reactions may be employed to deposit Al_2O_3 by CVD onto a substrate. In general, however, an aluminum halide vapor, usually AlCl_3 , is treated with an oxygen-bearing gas at high temperature in the presence of a substrate. The AlCl_3 is converted to Al_2O_3 plus usually gaseous by-products. Under suitable conditions the Al_2O_3 can be induced to deposit onto the substrate. These reactions are extremely sensitive to the processing variables. The rate of Al_2O_3 deposition and the coating quality may vary widely. Under certain conditions, nucleation of Al_2O_3 can occur in the vapor phase, which can lead to spectacularly unpredictable results.

Three CVD process variables, temperature, carrier gas flow rate, and reagent concentration, are being examined to select the optimum condition for producing α - Al_2O_3 coating. After the initial experiments have been completed and the specimens evaluated, at least six additional specimens will be coated under the apparent optimum conditions. This will permit verification of preliminary evaluations, and provide added confidence in the process.

The objective of these experiments is to develop a dense, continuous, crack-free coating of α - Al_2O_3 on T-111. In general, CVD Al_2O_3 has been observed to crystallize in the cubic γ form. This can lead to difficulties.

γ - Al_2O_3 has a density of about 3.5 g/cc as compared to 3.97 g/cc for α - Al_2O_3 . α - Al_2O_3 is the stable form at high temperature, and transformation from γ to α begins to occur rapidly at about 2200°F. Actually,

$\gamma \longrightarrow \alpha$ transformation has been observed at much lower temperatures. The transformation is, of course, accompanied by a volume decrease.

In the LRHSC configuration, a volume decrease of the Al_2O_3 standoff coating would put the coating into a stress state resembling pure tension, which is not acceptable for a ceramic. The consequences of this anticipated phenomenon have not yet been explored in detail, but cracking and spalling should occur. Hence, deposition of α - Al_2O_3 is considered mandatory.

For initial evaluation of the processes, small "bullets" of T-111 are being used. Chemical analysis of the T-111 bar stock used to make these samples is presented in Table VIII-2. An uncoated sample attached to a 304 stainless steel rod used to hold it in the CVD coating rig is shown in Figure VIII-3. Metallographic examination of the T-111 revealed numerous inclusions. The most prominent of these were relatively large spherical precipitates which occurred at grain boundary intersections, as shown in Figure VIII-4. The T-111 had an ASTM grain size of 9, which is equivalent to an average grain diameter of about 15μ . Its hardness was approximately 30 on the Rockwell C scale.

TABLE VIII-2

IMPURITY ANALYSIS OF T-111 COATING SPECIMENS

<u>Element</u>	<u>Amount, ppm</u>
Fe	100
Si	200
Zr	200
O ₂	35
N ₂	1
H ₂	1
C	< 50
Other Metallic Impurities	< 20 of any element

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Figure VIII-3. T-111 Coating Specimen (about $3/8$ inch dia x 1 inch) with 304 Stainless Steel Sample Holder

A contract was placed with San Fernando Laboratories (SFL) in San Fernando, California to perform the CVD coating effort. The first four samples received from San Fernando Laboratories were coated by the condition shown in Table VIII-3.

The hemispherical end of each coated specimen was light in color and had a grainy texture, as seen in Figure VIII-5. The cylindrical portions are dark gray, uniform, and reasonably smooth. The coatings did not contain the prominent protuberances seen in earlier coatings.

A Hi-Rez dye penetration test revealed that the specimens in general were crack-free; a single crack indication was seen in one specimen, and that could have resulted from handling damage. All of the specimens contained some fine,

Etchant: 50% HNO_3 50% HF 100X
Met No. 7985-1-1³

Etchant: 50% HNO_3 50% HF 500X
Met No. 7985-1-2³
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Figure VIII-4. Metallographic Structure of the T-111 Coating Samples

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TABLE VIII-3

CONDITIONS FOR CVD COATING Al_2O_3 ONTO T-111

Specimen No.	Deposition Temperature* (° F)	Reactant Concentration**	Oxidant	Coating Thickness (In.)	Finish (μ In RMS)	
					Pre- Coat	Post- Coat
C-1	2200	Standard	Wet air	0.0020	50	120
C-2	1950	Dilute	Room air	0.0021	50	90
C-3	1750	Standard	Room air	0.0006	50	60
C-4	1750	Dilute	Room air	0.0007	--	70
C-6	1900	Standard	Room air	0.0038	--	120
C-7	1900	Standard	Room air	0.0028	--	70
C-8	1900	Super Dilute	Room air	0.0020	--	50
C-9	1950	Super Dilute	Room air	0.0015	--	130
C-10	1950	Super Dilute	Room air	0.0025	--	160

* Coating time for all samples was 60 minutes.

** A relative value is used here to protect proprietary data of San Fernando Laboratories.

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Figure VIII-5. T-111 Samples Coated with Al_2O_3 by Chemical Vapor Deposition

generally distributed porosity; the level of porosity may have been as high as 10 volume percent.

X-ray diffraction patterns were run on the coated specimens to indicate the crystallographic composition of the Al_2O_3 . Specimen C-1, which was coated at the highest temperature of any in this series, consisted predominantly of α - Al_2O_3 . This was highly oriented, with the basal plane parallel to the substrate. The hemispherical end of the specimen, described earlier as light in color and grainy, consisted of a phase described as δ (delta) Al_2O_3 . This phase is the most stable of the metastable phases which exist between amorphous Al_2O_3 and α - Al_2O_3 . Specimen C-2 was similar in crystallographic composition to C-1. Specimens C-3 and C-4, both of whose coatings were deposited at 1750°F, also consisted primarily of α - Al_2O_3 . However, the peaks were not as sharply defined as those for C-1 and C-2. Some γ - Al_2O_3 was present in C-4.

Metallographic examinations of the coated specimens are in progress. The coating of specimen C-1 appeared to be dense and continuous, but it contained numerous radial cracks. It has a few inclusions which appeared to be pores, but other than that its structure was featureless. There appeared to be an interfacial phase between the Al_2O_3 and the T-111 substrate. This phase has not yet been identified. Specimen C-2 exhibited microstructure in its Al_2O_3 coating. It contained texture, grain boundaries, and inclusions, as well as radial cracks. There appeared to be some separation at the Al_2O_3 - T-111 interface, or possibly an interfacial phase. The coatings on specimens C-3 and C-4 were much thinner than those on C-1 and C-2. The former were both similar in features to the coating on C-2.

Bulk chemical analyses of interstitials in CVD - coated specimens C-1 through C-4 have been partially completed. These data shown in Table VIII-4 indicate that some minor contamination occurred during deposition of the

Al_2O_3 coatings. The level of contamination was remarkably uniform from specimen to specimen. These levels of C and H_2 impurities are not considered to be sufficient to have an appreciable effect on the ductility of the T-111. O_2 and N_2 analyses are yet to be completed.

An attempt was made to detect local concentrations of embrittling impurities. Microhardness traverses were made on the metallographic mounts of specimens C-1 through C-4. Starting in the immediate vicinity of the Al_2O_3 coatings, indentations were made at 2 mil intervals in the radial direction. The data were somewhat erratic, but they showed a distinct trend. Hardness in the first 2 to 4 mils from the coating was typically 55 Rockwell C. This decreased abruptly at about the 4th or 5th mil to 30 to 35 Rockwell C which is very similar to the as-received hardness. These data indicate that the T-111 specimens were indeed superficially contaminated with a thin skin of embrittling impurities during coating deposition.

TABLE VIII-4
BULK CHEMICAL ANALYSES OF CVD Al_2O_3 SAMPLES
C-1 THROUGH C-4

Specimen No.	Element, ppm	
	C	H_2
As Rec'd. T-111	50	1
C-1	57	11
C-2	50	12
C-3	57	9
C-4	56	13

Based on preliminary evaluation of samples C-1 through C-4, the most promising parameters were selected and a new set of samples coated. The conditions for these samples are also shown in Table VIII-3. The coating on sample C-5 was very irregular indicating a temporary malfunction of the coating equipment. No evaluation of this sample is planned. Specimens C-6 through C-10 have been received from San Fernando Laboratories, and have been shown to be α - Al_2O_3 . Dye penetration testing showed C-6 to contain cracks, but the others were crack-free. All contained some surface porosity.

A tungsten layer has been considered as a possible means of preventing surface contamination of the T-111 during coating. While the tungsten will react in a similar manner to T-111, the diffusion rates for interstitial impurities are much lower. Therefore, the tungsten may prevent the interstitials from reaching the T-111. To explore this approach, two T-111 specimens were CVD coated at 1650° F with about 0.0006 inch of tungsten. The specimens, W-1 and W-2, were subsequently coated with Al_2O_3 by CVD. X-ray diffraction patterns show the coatings to consist predominantly of α - Al_2O_3 . The traces have not yet been fully interpreted.

(2) Arc-Plasma Spraying

The arc-plasma spraying process is much simpler than the CVD process. Feed material in a carrier gas is fed into a spray gun, an integral part of which is a system of electrodes. An arc is struck across the electrodes. The carrier gas is ionized, and the feed material is melted, accelerated, and directed onto a substrate. Considerable operator skill is required to deposit controlled, uniform, and reproducible thicknesses of coating material, and there are several operating parameters which must be controlled. Although APS coatings are generally porous and of lower density than CVD coatings, the likelihood of producing γ Al_2O_3 is higher, of contaminating the substrate is lower, and the process is easier to control.

Two requirements of the LRHSC Program will make APS of Al_2O_3 coatings somewhat more complex than normal. One is that the T-111 must be protected from oxygen and nitrogen during the time that it is hot. The second is that the specimens must be preheated prior to coating to simulate the condition of a fueled LRHS capsule. These two requirements dictate that the coating operation be performed in an inert-atmosphere glove box. The specimens will be preheated to about 1200°F with a small removable furnace. Some design problems with the glove box are anticipated, since there will be large quantities of heat to control and dissipate.

A contract has been placed with Microcraft, Inc. of Inglewood, California to modify a glove box to provide a high purity inert gas atmosphere and a means of heating samples to 1200°F . Microcraft will coat ten T-111 specimens of the same configuration as the CVD specimens.

The glove box has been modified and trial runs using stainless steel samples are in progress. Coating of the T-111 samples should take place early next quarter.

(3) Sputtering

Sputtering is physical deposition accomplished by creating molecular vapors of a material by electron bombardment of a target in a vacuum system and causing a drift to a hot substrate by vacuum pumping. Since this process is carried out in a high vacuum, it has the greatest chance of depositing high density coatings with the least substrate contamination.

Specimens S-1 through S-5, coated by sputtering with about 0.5 mil of Al_2O_3 , have been received from Microcraft. The coatings were almost entirely amorphous, but contained 5 to 10 percent each of α - and γ - Al_2O_3 . It appears that considerable development effort would be required to produce α - Al_2O_3 by sputtering. Therefore, no further evaluation of this process is planned.

IX. IMPACT TESTING

A. TASK OBJECTIVE

The objective of this task is to determine, by testing, the capability 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 15 capsules will be impacted hot (~ 1150°F) on a granite target at various velocities and impact angles.

B. SUMMARY OF TASK STATUS

The impact testing will be accomplished in FY'69. No effort is planned for FY'68.

X. SOIL BURIAL

A. TASK OBJECTIVE

The objective of this task is to determine, by testing, the maximum surface temperature of a capsule buried in soil at a depth expected from earth impact of the capsule at terminal velocity. Additionally, a preliminary evaluation of the soil corrosion problem will be obtained under this task. Two internally heated, pressurized capsules will be used for the 1 year burial tests.

B. SUMMARY OF TASK STATUS

The burial tests will be accomplished in FY'69. No effort is planned for FY'68.

XI. CREEP CAPSULE EVALUATION

A. TASK OBJECTIVE

The objective of the Creep Capsule Evaluation Program is to obtain the maximum meaningful data from the creep testing program. To accomplish this, the creep capsules will be subjected to a wide range of pre- and post-test examinations and statistical correlation of the results of these examinations with the creep testing conditions will be attempted. The data gathered from this effort will be used for analytical evaluations of various creep models and if necessary development of a new varying-stress creep model. Precise measurements taken through the testing program and extreme care used in handling all capsules and test samples should greatly facilitate the correlation of the data.

B. SUMMARY OF TASK STATUS

A test plan has been prepared for creep capsule evaluation and the pre-test examination requirements have been defined.

No work is planned on this task until the first creep capsule has been tested, since pre-test examinations are made as part of the creep testing program.

APPENDIX A - FACILITIES

To conduct the LRHSC Program, new laboratories are being installed at the Canoga Park Facility of Atomics International. Approximately 5000 sq ft is being devoted to assembly, quality assurance, production control, and testing as shown in Figure A-1.

The fabrication area consists of approximately 2200 sq ft. In this area, equipment is being installed for assembly, cleaning, and welding the Ta-10W, T-111 and Pt-10Rh parts for the LRHS capsules. Since the presence of fuel is being simulated, all operations subsequent to the simulated fueling operation are conducted in inert gas atmospheres using artificially heated components. The atmospheres in the necessary glove boxes will be controlled and measured to less than one ppm of total active impurity content. The primary assembly area and the EB weld room will be connected with a dumb waiter so that partial assemblies can be readily transferred while contained in an inert gas filled box. All cleaning of parts will be performed in the cleaning equipment installed in the EB weld room.

Adjacent to the assembly area is the quality assurance room. In addition to the normal dimensional, dye penetrant, leak detection, and X-ray equipment, this room will contain ultrasonic and infra-red testing systems. Non-destructive testing procedures will be developed here and applied to the capsule being assembled.

To control the flow of material and parts and assure that the documentation requirements are being met, a production control area is being installed near the assembly and quality assurance rooms. In addition to records keeping, production control will assure conformance to applicable specifications and quality levels before release to the next operation.

NOTE:

AUXILIARY POWER SUPPLY PROPOSED
LOCATION AT NORTH END OF BUILDING 005.

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**PARTIAL FIRST
FLOOR PLAN**

Figure A-1. LRHSC Program Facility

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The test area will comprise approximately 2000 sq ft. Twelve high vacuum test systems are being installed in this area. Three of these are presently committed to biaxial creep testing at 2000°F and 10^{-8} torr, two are to be used for compatibility testing at 2000°F, 2250°F, and 2500°F in a vacuum of 10^{-8} torr and one will be used as an annealing furnace with a 3000°F, 10^{-6} torr capability. The remaining six units are available for additional work this year and will be used for creep testing next year.

To supplement the basic high vacuum systems, low pressure (2000 psi) and high pressure (6000 psi), high purity helium supplies are being installed for the biaxial creep tests; program controlled pressurization systems have been designed and are being built; an optical pyrometer and a cathetometer, both high precision, are being purchased; and a start-up cart which includes a sorption pump, bakeout furnace and supplementary power is being built.

The area shown in Figure A-1 as Receiving Inspection will be used as the Production Control Area and the former Production Control Area is being converted to an equipment renovation area. This area will be used to clean, recondition, and repair the test equipment and will contain a Molectric cleaner, an ultrasonic cleaner, gas hand welding equipment, a drill press, a sand blaster, a small lathe, and a sink.

To maintain long duration tests, the electrical power supply is obtained from one major power grid and backed up by a second independent power grid. In the event of an outage on the first grid, connection will be made to the second grid within 10 seconds.

All equipment for this facility is on hand except the EB weld unit (a 3 kw EB welder is presently available), two assembly glove boxes, the ultrasonic and infra-red testers, the pyrometer, the cathetometer, the pressure controllers and the test furnace heaters. All are presently being secured and will be operational before November 15, 1967.

FACILITY STATUS

a. Creep Testing

All secondary utilities to supply the creep and compatibility test systems have been installed and are operational. This includes power for the furnaces, ion pumps and instrumentation, and filtered recirculating water, air and liquid nitrogen for twelve complete systems. Nine of the twelve high vacuum systems have been installed in the LRHSC Laboratory. Their status is:

Three 1200 Liter Vacuum Systems (Creep Testing)

The bell jars on the three systems shown in Figure A-2 are presently being redesigned to accommodate the test capsule and heater. The consoles containing the vacuum and bake-out components are also nearing completion.

Two 400 Liter and One 150 Liter Systems (Compatibility Testing)

The three systems are intact and operable. The consoles have been rewired to Los Angeles Building Code and are currently being used for heater testing.

The 400 Liter Annealing Furnace

Modification of the vacuum chamber is in progress. There is a possibility of another pump being added to the system to increase the pumping capabilities. This can be accomplished in-house. The pumps must be reconditioned and pump and console power connected before start-up.

Three 400 Liter Vacuum Systems (Spares)

The three systems shown in Figure A-3 are intact with the exception of the bell jars. All three systems must be renovated before use.

b. Test Renovation Area

All utilities have been installed in this area and all equipment has been moved. Equipment hook-up and check-out is in progress.

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Figure A-2. LRHSC Laboratory Test Area Showing the Three Pump and Bell Jar Units to be Used for Biaxial Creep Testing

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Figure A-3. LRHSC Laboratory Test Area Showing the Three Units with Integral Chemical Sorption and Vacuum Ion Pumping

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c. Assembly and Quality Control Area

All utilities have been installed in the assembly and quality control rooms but have not been connected to the main supplies. This will be accomplished by October 15, 1967, as part of the E. B. weld area modification.

Two welding glove boxes, a programmed TIG welder (shown in Figure A-4) and a clean bench have been installed in the assembly area. Two assembly glove boxes are due to be received early in October, 1967.

The contract for modification of the E. B. Welding Room has been placed and will be completed except for installation of the E. B. Welder itself by October 15, 1967.

d. Production Control Area

All utilities have been installed and the equipment is being moved into the area.

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Figure A-4. LRHSC Laboratory Assembly Room Showing
the Two Welding Glove Boxes and the Programmed
TIG Welder

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APPENDIX B - HEATER DEVELOPMENT

A. OBJECTIVES

Heater development and evaluation are being performed to provide vacuum furnaces and an isotope heat source simulator. The vacuum furnaces are to be used for capsule creep testing, coupon compatibility development and hardware fabrication annealing processes. The isotope heat source simulator, or capsule heater, is needed to simulate the isotope thermal output during fabrication of development capsules in order to verify the suitability of the capsule tooling assembly under thermal conditions pertaining to fueled capsules. The capsule heater will also be used in a subsequent program phase: soil burial tests of development capsules.

Stringent heater requirements of temperature level and uniformity, high vacuum operation, long life, compatibility with capsule materials, and spatial constraints dictate the need for the heater development sub-task. However, extensive development efforts will not be needed since the LRHSC heater requirements taken individually fall within the state-of-the-art of heater technology. Evaluation of commercial high vacuum furnaces and of heaters developed at Atomics International for the SNAP 8 and 10A programs indicate that the LRHSC heater requirements can be met by modification or tailoring of existing heater designs with subsequent testing of prototypes to verify their design capability.

B. SUMMARY OF TASK STATUS

Heater requirements for the various LRHSC applications have been delineated. Heater designs have been prepared for the capsule creep tests, the coupon compatibility development, a tantalum annealing furnace, and the tooling assembly/burial capsule heaters. A prototype heater (Figure VII-2) based upon the SNAP 8 heater technology has been fabricated to verify the design capability of the creep test, coupon and annealing furnaces. Tests

of this prototype heater have demonstrated a 2000°F capability during the quarter. An alternate heater suitable for coupon compatibility testing has been ordered from Centorr Associates, Inc., Suncook, New Hampshire. There are currently several Centorr furnaces of similar design operating at AI under comparable temperature, vacuum and duty cycle conditions. Construction materials are being procured for additional furnaces similar to the prototype furnaces and for the tooling assembly heater. Power supplies and controllers are also being procured.

C. ACCOMPLISHMENTS DURING QUARTER

1. Heater Requirements

a. Capsule Creep Test Heater

- (1) Number and Availability: Three (3) by December 1, 1967
- (2) Temperature: 2000°F $\pm$ 5, in a uniform hot zone 2" dia.
x 12" long
- (3) Duty Cycle: 20,000 hours continuous operation
- (4) Environment: Vacuum, 10^{-8} torr or better
- (5) Penetrations: Access for the pressure electrical feedthru and thermocouple leads

Apertures to permit visibility across the mid-plane diameter of the capsule and in a longitudinal direction at three sites in regions of bottom, center, and top.
- (6) Electrical Rating: The power supply available is 208 volts, 30 amp, single phase

b. Coupon Compatibility Development Heater

- (1) Number and Availability: Two (2) by October 1, 1967
- (2) Temperature: 2500°F $\pm$ 5 maximum in a uniform hot zone of 2" diameter x 6" long
- (3) Duty Cycle: 10,000 hours continuously at 2000°F $\pm$ 5
500 hours continuously at 2500°F $\pm$ 5
- (4) Environment: Vacuum, 10^{-8} torr or better

- (5) Penetrations: Access for thermocouple leads to coupon holders and aperture at top surface to permit use of optical pyrometry.
- (6) Electrical Rating: The power supply available is 208 volts, 30 amp, single phase

c. Annealing Furnace

- (1) Number and Availability: One (1) by October 1, 1967
- (2) Temperature: $3000^{\circ}\text{F} \pm 30$ in an approximate hot zone, 10 inches diameter x 10 inches long
- (3) Duty Cycle: Two hours heat-up, five hours at 3500°F , and gradual cool-down
- (4) Environment: Vacuum, 10^{-5} torr or better
- (5) Penetration: Access through top for loading of work-piece and for passage of thermocouple leads
- (6) Electrical Rating: The power supply available is 208 volts, 50 amp, single phase

d. Tooling Assembly Heater

- (1) Number and Availability: Six (6) by November 15, 1967
- (2) Temperature: 2000°F maximum
- (3) Duty Cycle: 500 hours, 25 power cycles
- (4) Environment: Helium atmosphere or vacuum, 10^{-6} torr
- (5) Penetration: Electrical leads are restricted to a maximum diameter of 0.090 inches within the capsule pressure feedthru tube
- (6) Electrical: Heaters are to furnish a minimum of 204 watts in fuel zone; the available power supply is 110 volts, 20 amps, single phase

2. Heater Design Characteristics

a. Capsule Creep Test Heater

The design of a prototype heater suitable, with slight modification, for all three furnace applications was shown in Figure VII-2. The prototype heater was sized for application as the tantalum annealing furnace; smaller units can

be utilized for the creep test and coupon compatibility furnaces. This furnace design employs coiled tantalum wire supported on alumina ($99 + \% \text{Al}_2\text{O}_3$) insulators for the heating elements. This element design is based upon furnaces developed on the SNAP 8 and SRIP programs at AI for high temperature component testing. Many thousands of hours of furnace operation at temperatures up to 1500°F have been achieved with these furnaces with no perceptible change in the properties of the tantalum-alumina system.

The prototype furnace is of cylindrical design with the .066 inch tantalum wire wound into an approximate 0.3 inch diameter coil, which is supported on the inner tantalum cylinder by the alumina stand-offs. Three additional tantalum cylinders provide reflective insulation. The stand-offs and tantalum cylinders are bolted together with tantalum screws and stainless steel nuts. A horizontal slit ($1/8$ inch x 2.0 inch) through the side of all four Ta cylinders at approximately the mid-plane permits viewing of the test capsule for creep measurements. The top and bottom removable cover assemblies each contain four tantalum discs as reflective insulation. Holes in the top cover assembly permit the penetration of thermocouple leads attached to the test capsule.

The size of the prototype furnace is 8.08 inches O.D. x 16.5 inches long. The heat zone is 7.12 inches O.D. x 15.5 inches long. The length of uniform heat zone is anticipated to be about 12 inches. This size furnace can meet the annealing requirements (dependent upon tantalum drawing characteristics) but for the creep and coupon applications the heat zone is being scaled down to 4 inch diameter by 9 inches long to reduce power consumption.

The prototype heater consists electrically of two half heaters which are wired in series for creep testing (2000°F) and in parallel for annealing application ($\sim 3000^\circ\text{F}$). The heater-half resistances are 1.1 ohm at room temperature, ~ 4 ohms at 2000°F and ~ 5.5 ohms at 3000°F . The heaters

are designed to operate in the 100-150 volt range at power levels of 3-5 kw in the 2000-3000°F temperature ranges. The design voltage range is well below the arcing voltage for 10^{-6} torr or better vacuum operation. Deviations from these design levels can be compensated for by use of additional shielding. (The four shield design of the prototype heater was used to expedite construction and testing. Additional shields can be readily added as an auxiliary assembly if required.) The completed prototype heater is shown installed in the vacuum chamber preparatory to thermal testing in Figure VII-2. Thermal tests started September 26, 1967.

b. Coupon Compatibility Development Heater

The coupon compatibility test heater requirements are being met with a scale down of the prototype annealing furnace discussed in Section VII. However, a vendor fabricated furnace is also being procured for coupon testing; this furnace design which utilizes tantalum foil elements may also be applicable to creep tests of capsules if the need arises. The backup coupon test furnace is a Centorr series 60 vacuum furnace, a type which has been employed successfully at AI at temperatures in excess of 2500°F for several thousand hours. This furnace compares favorably in cost with the AI heater design for small heat zone requirements up to about 2 inch diameter by 6 inches long. Delivery of a Centorr furnace is anticipated about October 23, 1967. The scaled down prototype annealing furnace is being fabricated; completion of fabrication is scheduled for October 9, 1967.

Tantalum ribbon element heaters developed for SNAP 10A system acceptance tests were originally considered for coupon compatibility test application because of their availability and their demonstrated performance at ribbon temperatures of 3000°F. Their usability however was found to be restricted because of oxidation of the Ta ribbon which occurred during the SNAP 10A tests, which were performed at 10^{-2} torr. One heater assembly was tested for coupon heater application and it failed after reaching a temperature of 1600°F. No more attempts will be made to use these heaters.

c. Tooling Assembly Heater

The tooling assembly heater design is shown in Figure B-1. The heater consists of tantalum wire wound onto an alumina mandrel. The mandrel is designed to fit snugly over the inner liner and occupy part of the space allotted to the isotope fuel. The heater characteristics are as follows:

Wire diameter	.040 inches
Coil outer diameter	1.15 inches
Number of wire turns	38
Coil resistance	1.3 ohms at 1000° F 0.4 ohms at 70° F
Lead resistance	~ 0.1 ohms at 1000° F
Voltage/current	18 v/12 amps at 1000° F
Insulation	Plasma-sprayed alumina and alumina beads on the leads

The heater leads in the pressure feedthru tube are 0.060 inch in diameter. The seal at the feedthru exit is a conax type. The heater leads may be tantalum. The bare wire design allows accurate examination of the heater wire and the bonds between the heater element and leads, which is not the case in sheathed heater element designs.

This heater design also has potential use in the capsule burial test program since 2000° F operation is not beyond its design capability. Two heater units are being built, in addition to the six required units, for the purpose of qualifying the heater for both the tooling assembly and burial capsule applications.

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Figure B-1. Tooling Assembly Heater Design

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