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LARGE RADIOISOTOPE
HEAT SOURCE CAPSULE (LRHSC)
SECOND QUARTERLY PROGRESS REPORT
OCTOBER-DECEMBER 1967

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION

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Printed in the United States of America
Available from
Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U.S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

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A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION

CONTRACT: AT(29-2)-2338
ISSUED: JANUARY 15, 1968

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FOREWORD

This is the second 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 October 1, 1967 through December 31, 1967 under Contract AT(29-2)-2338 for the U. S. Atomic Energy Commission.

SUMMARY

The GFY 1968 program is currently on schedule and budget. All planning and control documentation has been released including the final weld development plan and the Quality Assurance Plan. Documentation methods for all phases of the program have been established and implemented.

During the quarter considerable analytical support to the test program was supplied. This work was mainly directed at thermal environments, chill block requirements and the interference fit tooling requirements.

All assembly and test tooling and fixture design were completed, released and fabrication was initiated.

In the process development area, the warm back extrusion process for producing deep T-111 cups was demonstrated; two types of impact absorbers were evaluated; TIG welding at 1 psia was done; and a heat treating study of Ta-10W and T-111 was completed. All of these processes are required in order to fabricate the first test capsule.

Coating development for application of the Al_2O_3 barrier coating continued during the quarter. A firm process for applying α phase Al_2O_3 is now in hand. A topical report was initiated.

During the October through December time period, four coupon compatibility stacks were tested. The 100 hour test at 2000°F , and the 10 hour test at 2250°F were completed. The 1000 hour test at 2000°F was initiated and will be completed in January. The 10 hour test at 2500°F was initiated in late December. The significant results found to date are a general incompatibility between iron titanate and both Ta-10W and T-111. Detailed evaluations are continuing.

A major effort has been expended on the creep test systems, test techniques and instrumentation. A final approach which meets both the temperature control requirements and the visual observation requirement has been verified.

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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) Creep 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.

II. PLANNING

A. TASK OBJECTIVES

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

B. SUMMARY OF TASK STATUS

During the reporting period the program has proceeded on schedule and within projected costs. The official milestones and the status of each are shown in Table II-1. All overall programmatic planning for GFY 1968 has been completed. GFY 1969 preliminary plans were completed and transmitted to the AEC during the reporting period. A number of program review meetings were held including two formal reviews with Sandia and AEC management.

C. ACCOMPLISHMENTS DURING QUARTER

Certain of the internal planning documents completed during the first quarter have been reviewed, updated and released. These revisions were based on further program progress. A final Quality Assurance Plan was prepared and should remain current during the remainder of the program. The weld development plan was revised to reflect the final test capsule design evolved during the end of the first quarter.

All key drawings and specifications have been "baselined" and are now under formal "change control" procedures.

During October a full day review was held at AI for the AEC, Sandia, NASA, and representatives of Mound and Battelle. This was a thorough technical review of all aspects of the LRHSC program. In early December an overall review was presented in Washington which included both technical and programmatic status.

An evaluation of manpower and cost control procedures used on the program revealed that manpower was within 0.6% of the original loading prediction during the first half of this year's effort. Further, the scope is being met within the allocated budget.

TABLE II-1
OFFICIAL MILESTONES SUMMARY

	<u>Official Milestones</u>	<u>Target Date</u>	<u>Status</u>
1.	Freeze design and release capsule piece-part drawings	August 1967	Complete
2.	Complete and release all material specifications	August 1967	Complete
3.	Complete and release all capsule assembly drawings	Sept. 1967	Complete
4.	Complete design and release of fixture drawings	Sept. 1967	Complete
5.	Complete Al ₂ O ₃ CVD vs plasma coating evaluation	Oct. 1967	Complete
6.	Complete all assembly specifications	Nov. 1967	Complete
7.	Complete all processing and test specifications	January 1968	OK*
8.	Initiate short term creep tests	January 1968	~1/30/68
9.	Complete coupon and coating development tests	March 1968	OK
10.	Complete GFY 1968 capsule fabrication and assembly	March 1968	OK
**11.	Complete Topical Report # 1, Capsule Design/Fabrication/Assembly	April 1968	OK
12.	Complete Topical Report # 2, Ceramic Coating and Coupon Compatibility Evaluation	May 1968	OK
*	Those required for GFY 1968		
**	New Topical Report, "LRHSC Program Material Specifications" has been completed and will be available in January.		

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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

During the first quarter, analysis was directed primarily to finalizing capsule design parameters. During this past quarter, analytical support has been provided primarily in the areas of capsule assembly and test. The tasks will continue to provide supporting analysis as required. The verification of design approaches and definition of design margin activities will not be initiated until later in the program when substantial test data are available.

C. ACCOMPLISHMENTS DURING THE QUARTER

Specific analyses have been accomplished in the past quarter on the platinum clad assembly criteria, the temperature drop between the structural shell and platinum clad, and the stresses in the coupon compatibility stacks.

1. Platinum Clad Assembly Criteria

Based on previously reported investigations the helium pressure in the gap between the Pt-10Rh and T-111 will be limited to 1.8 psia @ 2000°F. This pressure, which produces 75 psi stress in the Pt-10Rh, should result in $\leq 1\%$ creep of the Pt-10Rh in 7 years.

Alternate methods of assembly of the Pt-10Rh clad to limit the helium pressure have been investigated. The most straight forward approach is to make the clad closure weld in a low pressure helium atmosphere. During the past quarter initiation and maintenance of a TIG arc in 1 psi helium has been

demonstrated in a glove box in the LRHSC assembly area. Based on this capability the following criteria will be used for assembly of the Pt-10Rh clad.

Assembly Temperature	-	Chill Block
Radial clearance between T-111 and Pt-10Rh	-	0.0005 - 0.0015 in
Axial clearnace between T-111 and Pt-10Rh	-	line-to-line, both ends
Helium pressure at closure	-	1 ± 0.1 psia

Considering the changes in volume and temperature the above conditions will result in a helium pressure of 1.25 to 1.8 psi at 2000°F. The radial gap at 2000°F will be 3 to 4 mils.

2. Helium Gap ΔT

An analysis has been made on the effect of helium pressure on the temperature difference across the gap between the Pt-10Rh cladding and the Al_2O_3 coated T-111 structural shell.

The helium pressure in this gap at operating temperature will depend on the clearance and backfill pressure during assembly. It is desired to keep the helium pressure low enough to prevent significant creep of the Pt-10Rh but not so low as to introduce a large ΔT . Therefore, the effect of helium pressure on conductivity is desired.

At pressures near one atmosphere the intermolecular distances are much smaller than the body dimensions with which we are usually concerned in heat transfer calculations. The thermal conductivity of gases for pressures near one atmosphere has been found to be virtually independent of the pressure. However, at low pressures the molecular mean free path can become comparable with the body dimensions. The thermal conductivity is then proportional to the pressure and approaches zero with pressure because eventually no molecules are available for the transportation of heat energy.

The relative importance of the effects of pressure may be indicated by a comparison of the magnitude of the mean free path of a gas molecule with some significant body dimension, in this case the clearance between the Pt-10Rh cladding and the T-111 structural shell. This dimensionless ratio is referred to as the Knudsen number. For small values of Knudsen number the thermal conductivity is independent of pressure; for large values the thermal conductivity is proportional to pressure. The transition region from nondependence to dependence on pressure is gradual and is approximately .1 to 10.

The currently proposed assembly procedures uses a one mill gap and 1 psia at room temperature. At the operating condition of 2000°F the gap has increased to 3 mils and the helium pressure to 1.5 psia. The mean free path of the helium molecules is 0.45 mils at 2000°F and 1.5 psia. Therefore the Knudsen number is $0.45/3$ or 0.15. This value lies within the transition region as defined above. However, it is near the nondependence on pressure end of the region. The thermal conductivity is still insensitive to pressure and is not expected to be significantly different from the value at atmospheric pressure. The temperature drop across the helium gap is expected to be about 4°F based on a thermal conductivity of 0.2 Btu/hr-ft-°F and a 3 mil gap.

3. Coupon Stack Stresses

The coupon compatibility test stacks are contained in a Ta-10W holder which is torqued down during assembly to put on initial compressive stresses on the coupon stacks. An analysis has been made of the stresses induced by thermal expansion when the coupon test stacks are taken from room temperature to 2000°F. The initial test stack dimensions are as follows:

Component	Nominal Thickness (in)
Al ₂ O ₃ Spacer	.0622
T-111	.0195
Pt-10Rh with 6 mils of Iron Titanate	.0256
Ta-10W	.0221
Pt-10Rh with 4 mils of Iron Titanate	.0238
T-111 with 2 mils of CVD Al ₂ O ₃	.0454
Ta-10W	.0490
T-111 with 2 mils of Plasma Al ₂ O ₃	.0541
Pt-10Rh	.0214
P-10Rh with 4 mils of Iron Titanate	.0238
T-111 with 2 mils of CVD Al ₂ O ₃	.0545
Pt-10R	.0202
Al ₂ O ₃ Spacer	.0622
	<hr/>
	.5098

By grouping the materials the following table can be constructed:

Component	Thickness-t (in)	Expansion coef. (in/in°F from 85°F to 2000°F)	$\alpha t \Delta t$ (in)	E@2000°F (X10 ⁻⁶)	t/E ₆ (x10 ⁶)
Al ₂ O ₃	0.1324	4.6	0.00117	50	0.00265
T-111	0.1910	3.93	0.00144	10.3	0.185
Ta-10W	0.0711	3.19	0.00043	10.13	0.0069
Pt-10Rh	0.1008	5.6	0.00108	~10	0.0101
Iron Titanate	0.014	~1.2	0.00003	50	0.0003
W	0.0005	~3.1	0.000003	27	0.00002
Totals	<hr/> 0.5098		<hr/> 0.00416		<hr/> 0.0385
Ta-10W Holder	0.5098	3.3	0.00322	~10	0.051

From the table it can be seen that the coupon stack tends to expand more than the holder by 0.94 mils. The stresses induced by this differential thermal expansion can be calculated as follows:

$$\sum \frac{Pt}{AE} = e \quad \text{or} \quad \sigma \sum \frac{t}{E} = e$$

The strain in the coupon stack plus the strain in the holder must equal the differential thermal expansion

$$\sigma_s \cdot \sum_s \frac{t}{E} + \sigma_H \cdot \frac{t}{E_H} = 0.00094$$

The relationship of the stress in the stack and the stress in the holder can be derived from the relative areas since the total force in each must balance.

$$A_s = 0.385 \text{ in}^2$$

$$A_H = 0.543 \text{ in}^2$$

Therefore $\frac{\sigma_H}{\sigma_s} = \frac{.385}{.543} \quad \sigma_H = 0.69 \sigma_s$

Substituting in the previous equation and using the values of t/E from the table

$$\sigma_s = 13,500 \text{ psi}$$

$$e_s = 0.456 \text{ mils}$$

$$F_s = F_H = 5,100 \text{ lbs}$$

It should be noted that the above calculation assumes that all the coupons and coatings are perfectly flat and in uniform contact. Any lack of flatness would cause the coupons to act as springs and lead to considerable reductions in the compressive stress. Also local deformation of uneven coating surfaces could reduce the compressive stress substantially, particularly since the total differential thermal expansion is less than one mil.

Theoretically it would be possible to add a low coefficient of expansion material to the stack or to assemble the stack with a gap and thereby adjust the stress at temperatures to any desired value. However, the large effects on stress of very small uncertainties in coupon flatness or coating uniformity make such attempts unwarranted. Furthermore, attempts to adjust the stress in each stack would lead to less reproducibility between the coupon tests due to the additional uncertainty in the adjustment mechanism.

It should also be noted that, at 2000°F and above, creep and relaxation in the coupons and coupon holder will lead to fairly rapid reduction of any initial high stack stress.

The initial stress imposed on the coupon stack by the torquing operation on the Ta-10W cap has also been calculated. For dry threads and a torque of 60 in-lbs the resultant stress should be 500-700 psi.

In summary, it does not appear that the initial torque-down stresses or the thermally induced stress will be destructive. Greatest uniformity between coupons tests should result from selection of the lowest closing torque that is considered reproducible and use of this torque on all subsequent tests. Verification of this conclusion will be obtained from the initial short term, lower temperature (2000°F) coupon tests. In these tests any compressive stress induced failures will be easily separable from materials compatibility or inherent coating integrity problems.

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

All capsule piece-part, assembly, tooling and fixture design has been completed and the drawings released. All compatibility and creep test fixture design is complete.

No further design effort is required this year except minor modifications to the tooling that may become necessary when the tooling is checked out.

C. ACCOMPLISHMENTS DURING THE QUARTER

Capsule piece-part and assembly design was completed last quarter. The capsule tooling and fixture designs are discussed in Section VI.

The test fixtures are discussed in conjunction with the creep testing in Section VII and coupon compatibility testing in Section VIII.

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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, joining, and non-destructive testing. 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

Preliminary evaluation of both foamed and fibrous Ta-10W indicates that more development is needed to produce a cleaner foamed structure or a well-bonded fiber structure for the capsule impact absorber.

Annealing studies were performed on Ta-10W end caps and the in-process and final annealing conditions have been established for Ta-10W and T-111.

Welding development has begun at Electron Beam Welding, Inc. using a similar machine to the one purchased by AI for this program. The preliminary parameters have been established for the full penetration butt weld of the Ta-10W pressure feed through to the T-111 structural end cap of creep capsules. Development of the T-111 structural shell weld will begin as soon as suitable piece parts to mock up the T-111 joint and Ta-10W liner backing the joint are received. The new 6KW Hamilton Standard electron beam welder has been received and installed at AI, and is presently undergoing acceptance testing.

The High Resolution Ultrasonic Test System for inspecting the capsule welds has been received from D. Erdman Co., is installed and checked out, and is ready for test procedure development. The infrared test equipment for coating adherence testing has been received from Automation Industries, Inc. and installed. It is presently undergoing checkout.

All materials specifications for the LRHSC Program have been released. A topical report covering these specifications has been written and will be published next quarter. All process specifications except for welding and brazing have been released. A topical report covering these specifications will be written and published next quarter. The compatibility test specification has been released and the creep test specification is being prepared.

C. ACCOMPLISHMENTS DURING THE QUARTER

1. Impact Absorber

Swatches of foamed low density (7-9% theoretical density) Ta and Ta-10W, and felted but unsintered Ta and Ta-10W fibers have been received. Evaluation of these materials consists primarily of load deflection testing at 1150°F and chemical analysis for interstitial pickup. Vacuum sintering of the fibrous materials and vacuum outgassing of the foam is also being performed.

Initial visual examination of the foamed materials indicated that they were very brittle. Chemical analysis shows they contain up to 300 ppm of carbon and about 100 ppm oxygen. These contaminants probably came from the organic foaming agent used to produce low density material. The supplier, Astro Met Associates, has stated that a higher density (25-30% dense) material can be made without the foaming agent and this material should be clean and ductile. Three swatches and three hemispherical caps of this higher density material were ordered.

In an attempt to improve the quality of the as-received foams, vacuum outgassing at 3600°F is being evaluated. A disk of foamed Ta-10W which was vacuum outgassed is shown in Figure V-1.

The feltmetal supplier, Huyck Metals Company, does not have adequate sintering facilities for bonding their Ta or Ta-10W product. Therefore, it was agreed that they would felt the material and recommend a sintering cycle and Atomics International would do the sintering. The sintering parameters are critical to this process since too low a temperature fails to produce the required fiber-to-fiber bonds while too high a temperature causes a large density increase and loss of compressibility. A sample of the Ta-10W felt sintered at 3100°F is shown in Figure V-2. A significant number of fiber-to-fiber bonds was produced but as can be seen in the figure, all of the fibers are

Figure V-1. Ta-10W Foam metal vacuum outgassed at 3600°F for 30 minutes. Density approximately 7% of theoretical.

Figure V-2. Ta-10W Feltmetal sintered at 3100°F for 1 hr. at 10^{-6} torr. Density approximately 10% theoretical.

not bonded. The sample easily sheds fibers during handling. This material was resintered at 3400°F to improve its handling and machining characteristics. The amount of fiber-to-fiber bonding, although improved, was still not of good quality for further fabrication.

After cleaning and sintering, the samples to be load deflection tested were cut into uniform cylindrical discs and measured with a micrometer. The 7% dense foamed Ta sample (Figure V-3), was loaded into the load deflection testing apparatus between two press faces. The system was then pumped down to 10^{-5} torr and the sample was heated to approximately 1120°F. The results of the test are shown in Table V-1. The system was disassembled and the sample was removed. It appeared that more than 50% of the original disc had crumbled under the load as shown in Figure V-4.

Calculations show that at approximately 30% deflection the sample had absorbed a 60-psi compressive stress. The Ta-10W sample should be stronger as will be the Feltmetal sample. The high density foamed metal (25%) and the fiber metal should also show a significantly larger compressive strength.

2. Annealing

A series of Ta-10W end caps were coined and subsequently vacuum heat treated to establish the recrystallization and annealing range for the components. The parts are shown in Figure V-5. Post-annealing dimensional inspection of the caps indicated no detectable change in dimensions.

Table V-2 shows that a hardness test alone may not be sufficient to indicate microstructural changes prior to full recrystallization. The test has, however, established the heat treating range for the components.

To evaluate the effect of a diffusion pumped vacuum furnace on interstitial pickup of T-111 during annealing, a piece of T-111 scrap was heated to 2700°F for 3 hours and 3000°F for 1 hour at a vacuum of 5×10^{-6} torr in an oil diffusion pumped system. Chemical analysis is now in progress and preliminary results indicate less than 10 ppm of carbon pickup over the 30 ppm initially present.

a. Top View

b. Side View

Figure V-3. Foamed Ta Disc (7% Dense) Vacuum
Outgassed at 3500°F for 1 hr —
Before Load Deflection Test

Figure V-4. Foamed Ta Disc (7% Dense) Vacuum
Outgassed at 3500°F for 1 hr — After Load
Deflection Testing at 1120°F — 10^{-5} Torr

Figure V-5. Coined Ta-10W End Caps Used in Heat Treating Study

TABLE V-1
LOAD DEFLECTION TESTING OF FOAMED Ta (7% DENSE)
AT 1120°F - 10⁻⁵ TORR

Load (lb)	Stress (psi)	Deflection (in.)	Deflection (%)	Time at Load (sec)	Note
0	0	0.000	0.0		
5	10	0.001	0.46		
10	20	0.002	0.92		
15	30	0.014	6.56		
0	0	0.0125	5.86		Elastic Recovery
5	10	0.013	6.1		
10	20	0.014	6.56		
15	30	0.015	7.0		
20	40	0.051	23.9	90	
0	0	0.0485	22.7		Elastic Recovery
5	10	0.0495	23.2		
10	20	0.050	23.4		
15	30	0.0505	23.65		
20	40	0.051	23.7		
25	50	0.054	25.3	30	
25	50	0.0555	26.0	120	
25	50	0.056	26.2	240	
30	60	0.059	27.6	15	
30	60	0.061	28.6	30	
30	60	0.068	31.9	120	
0	0	0.066	30.95		Elastic Recovery

Original Sample Size: Diameter = 0.799 in.
Thickness = 0.213 in.
Area $\cong$ 0.5 in.²

Chemical Analysis of Sample (PPM): O₂ N₂ C
 33-102 4-6 141-241

TABLE V-2

Condition	Hardness (Ra)	Microstructure
As Fabricated	62, 62, 64	Cold Worked — Fibrous
1 Hour at 2400°F	64, 60, 60, 62	70-80% Recrystallized
1 Hour at 2600°F	59, 60, 60	90-100% Recrystallized
3 Hours at 2600°F	55, 54, 54	100% Recrystallized, Partial Grain Growth
1 Hour at 2800°F	21, 19, 22, 23	Full Annealed

3. Vacuum Brazing

An induction heated vacuum brazing rig has been installed in the assembly laboratory as shown in Figure V-6. The unit will be operational early in January. Final definition of the parameters for brazing the creep capsule feed through to the test chamber lid will be completed next quarter.

Figure V-6. The Vacuum Brazing Rig Installed in the LRHSC Assembly Laboratory. The pumping station is shown on the left, the induction unit on the right and the vacuum chamber in the center. A chamber extension which has been added to accommodate the capsule feed through is not visible.

4. Welding

Pending acceptance of a new welder by AI, electron beam weld development is being conducted at Electron Beam Welding, Inc. The welder being used is similar to the new one except for ultimate vacuum capability. Because of anticipated minor differences between machines, the welding procedures developed at Electron Beam Welding, Inc. will have to be verified and slightly modified at AI.

Thus far, effort has been concentrated on the Ta-10W to T-111 pressure feed through joint shown in Figure V-7. The optimum welding parameters, shown in Table V-3, meet the requirements of 100% penetration, 90% sound, defect-free metal and no cracks or lack of fusion. However, a considerable amount of drop through occurs. While drop through is not desirable, it does not affect the functioning of this test assembly weld and therefore, no major effort will be expended to eliminate drop through on this joint. The weld is made in two passes, the first for penetration and the second to smooth the weld surface. The second "cosmetic" pass is used to remove any ridges that would cause extraneous surface indications in a liquid penetrant test and to assure a smooth surface for subsequent assembly operations.

Development of the T-111 structural shell weld with a Ta-10W backing ring will begin early next quarter. The backing ring is used to simulate the outer liner of the fueled subassembly.

A 6-kw electron beam welder built to specifications based on the LRHSC welding requirements has been received from Hamilton Standard Division of United Aircraft Corp. It has been installed at Atomics International and is presently undergoing acceptance testing. This machine, shown in Figure V-8, is capable of an ultimate vacuum of less than 5×10^{-7} torr and can be pumped to 1×10^{-6} torr in 27 minutes.

5. Nondestructive Testing Development

For the purposes of this discussion, linear dimensioning involving special jigs and fixtures will not be considered as a part of the nondestructive test development effort. No development will be required for any of the raw stock inspection. This includes such materials as sheet, strip, plate, rod, bar, and tubing. However, for the raw stock ultrasonic tests, acceptance standards have

Figure V-7. Weld Development Mock-Up Design of the Ta-10W to T-111 Pressure Feed Through Joint

TABLE V-3
ELECTRON BEAM WELDING PARAMETERS FOR THE
Ta-10W TO T-111 PRESSURE FEED THROUGH JOINT

Parameter	Penetration Pass	Cosmetic Pass
Voltage	120 kv	120 kv
Beam Current	10 ma	5 ma
Weld Speed	20 rpm	50 rpm
Beam Diameter	0.016-0.018 in.	0.025 in.

NOTE: The vacuum during welding at Electron Beam Welding, Inc. was less than 10^{-4} torr. The electron beam was defocused during the cosmetic pass.

a. Control Console

b. Overall View Showing the Chamber on the Right
and the Pumping System on the Left

Figure V-8. Hamilton Standard 6-kw Electron
Beam Welder

to be specially prepared from some of the material received, since such standards are not commercially available. Insofar as nondestructive test methods are concerned, development work is not required for X-ray radiography, liquid penetrant, density, X-ray diffraction, normal helium leak testing (at one stage of capsule assembly an elevated temperature helium in-leakage test requires development) and emissivity testing.

All the points in the fabrication and testing sequence for the reference design where nondestructive testing will be employed, exclusive of raw stock are listed in Table V-4. This table is also applicable to the pressure feed through design. No need is anticipated for NDT development in the test capsule design for the pressure feed through tube modifications to the reference design. Normal high resolution X-ray radiography, liquid penetrant, and helium leak methods will apply. Modification of the ceramic NDT test developments from the reference design can be applied to the coated feed through tube components.

a. Ultrasonic Testing of Welds

With the exception of the structural shell weld, the various joints are in thin material, and require accurate and precise positioning relative to a test transducer. After certain stages of fabrication, inner liner weld examinations must be performed without contaminating the inside of the part with the ultrasound couplant. This places additional requirements on the test fixture. Precise location of any discontinuities across the thickness of the part requires special high resolution instrumentation. An ultrasonic test system capable of these stringent requirements has been purchased from D. Erdman Company of Pasadena, California. This equipment has been received and is ready for use as shown in Figure V-9. Operator training was provided by the supplier during the checkout period, and preliminary test techniques were developed. Final test techniques will be developed using weld qualification samples as they become available. It will now be necessary to establish the test procedures, standards and limits of sensitivity for all the welds in Table V-4 requiring ultrasonic test development. At all times during this work, prevention of contamination to the tantalum alloys will be of prime consideration. Also, the methods developed must be adaptable to thermally and radioactively hot parts in the future. Test capabilities and limitations will be factored into acceptance test requirements. A unique area of development is to measure for verification of the interrupted welds in the inner liner components.

TABLE V-4

SUMMARY OF REFERENCE DESIGN CAPSULE PARTS AND ASSEMBLIES
REQUIRING NONDESTRUCTIVE TESTING DURING PROCESSING
(Sheet 1 of 2)

Part No. and Name	Region for Nondestructive Test	Type of Tests	Development Required
N73420018 Inner Liner Stepped Cap	All	Liquid penetrant and He Leak	None
N73420019 Inner Liner Sleeve	All	Liquid penetrant, He Leak, and Ultrasonic	Ultrasonic
N73420017 Inner Liner Sleeve and Cap Assembly	Weld Joint	Liquid penetrant, He Lead, X-ray and Ultrasonic	Ultrasonic
N73420016 Inner Liner Flanged Cap	All	Liquid penetrant and He Leak	None
N73420015 Inner Liner Assembly	Interrupted Weld	X-ray and Ultrasonic	Ultrasonic
N73420009 N73420013 Honeycomb	All	Crush Strength	Crush Strength
N73420012 Outer Liner Sleeve	All	Liquid penetrant and He Leak	None
N73420014 Outer Liner Stepped Cap	All	Liquid penetrant and He Leak	None
N73420011 Outer Liner Subassembly	Interrupted Weld, Configuration, Weld Joint	X-ray and Ultrasonic, Liquid penetrant, He Leak	Ultrasonic
Tungsten and Molybdenum Powder	-	Density and Particle Size	None
N73420010 Fuel Closure Ring	All	Liquid penetrant	None

TABLE V-4
SUMMARY OF REFERENCE DESIGN CAPSULE PARTS AND ASSEMBLIES
REQUIRING NONDESTRUCTIVE TESTING DURING PROCESSING
(Sheet 2 of 2)

Part No. and Name	Region for Nondestructive Test	Type of Tests	Development Required
N73420007 Outer Liner Assembly (Before Final End Cap)	Retaining ring to inner liner in- terrupted weld	Ultrasonic and Elevated Temperature He In-Leak	Both
N73420007 Outer Liner Assembly	Closure weld and configuration	Ultrasonic, He Leak, and X-ray	Ultrasonic
N73420005 Structure Shell	All	Liquid penetrant, He Leak, and Ultrasonic	Ultrasonic of spherical end
N73420006 Structure Cap	All	Liquid penetrant, He Leak, and Ultrasonic	Ultrasonic of spherical end
N73420004 Structure Assembly	Weld Joint	Ultrasonic and He Leak	Ultrasonic
N73420004 Coated Structure Assembly	All	Eddy current, Beta Back Scatter, Infrared, and Electrified Particle	All
N73420002 Barrier Shell	All	Liquid penetrant and He Leak	None
N73420003 Barrier Cap	All	Liquid penetrant and He Leak	None
Fe ₂ O ₃ · TiO ₂ Powder	All	X-ray diffraction and particle size	None
N73420047 Capsule Assembly (Uncoated)	Closure Welds	He Leak, Liquid pene- trant, X-ray, and Ultrasonic	X-ray and Ultrasonic
N73420047 Capsule Assembly (Coated)	All	Eddy current, Beta Back Scatter, Infrared, and Electrified Particle	Eddy current, Beta Back Scat- ter, Infrared, and Electrified Particle

Figure V-9. The High Resolution Ultrasonic Test System. The precision locating fixture is shown in front of the couplant tank. The water level in the tank has been lowered to show the search tube that is used for X-Y scanning. The control and high speed CRT are above the tank and the X-Y recorder to the left.

b. Crush Strength Tests

This will be a test of the impact absorber. It is not expected that the development effort here will be extensive, being primarily a function of fixture design, proper loading, and detection of low strength. This test will be non-destructive only if the strength of parts is adequate.

c. Elevated Temperature Helium In-Leak

Certain welds in the inner capsule assembly are unique in that they are designed to leak helium. A test to verify this is required at some elevated temperature. It may not be necessary to develop this test for maximum capsule operating temperature (2000°F). Some lower temperature (approx. 1400°F) might be considered at this point. In expectation that the test to be developed will involve some type of functional simulation, a mass spectrometer leak

detector (helium and hydrogen), furnaces, controllers and vacuum pumps have been obtained for this work. A hydrogen detection capability at this point is desirable as a means of assuring that the ultrasonic test couplant (some hydrogenous liquid such as water, oil or alcohol) has not been entrapped in the assemblies.

d. Ceramic Coating NDT Developments

The Al_2O_3 coated T-111 alloy structural shell and $\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$ coated Pt-10Rh outer shell require the following nondestructive tests:

- 1) Integrity or soundness
- 2) Thickness
- 3) Adherence to metal substrate

Of these three areas, the one requiring the least effort will be determining coating integrity, i.e., freedom from cracks and gross voids. This will be performed by electrified particle testing similar to that developed for the SNAP fuel hydrogen retention barrier.

Thickness testing is being approached by two methods: (1) high frequency eddy current, and (2) beta back scatter. The eddy current test is the easier of the two to develop, but is only applicable to unfueled capsules. Beta back scatter will be developed as a back-up to the eddy current method as well as a method applicable to fueled assemblies.

Eddy current and beta back scatter equipment were obtained for measuring the coating thickness; electrified particle equipment was obtained for coating integrity tests. The feasibility of these tests (coating adherence, thickness, and integrity) was demonstrated to a limited degree on some small coated samples (3/4-in. diameter wafers). However, correlation studies using destructive tests to establish accuracy of these nondestructive tests will require additional samples. Also larger samples will be required for the infrared test development.

Adherence of the ceramic to the metal substrate will be determined by an infrared method. This method is based on a difference in transient heat transfer between areas of good adherence or contact of the ceramic with the substrate,

and areas of poor adherence or contact. The necessary instrumentation and fixtures have been received from Automation Industries Research Laboratories and are shown in Figure V-10. Operator training on this equipment has been provided at the suppliers facility.

Figure V-10. The Temp Test Infrared Test Equipment.
The test piece rotating fixture is shown under the control unit (left) and the strip chart recorder (right).
The infrared sensor is directly above the fixture (not visible) and the heater lamp is hanging to the left of the fixture.

6. Specifications and Documentation

The following specifications were written and formally released during the quarter:

ST0610NA0006, Cleaning, Platinum and Platinum Base Alloys, LRHSC
ST0620NA0053, Creep Standard Capsule "C," LRHSC, Assembly Of
ST0620NA0054, Creep Test Capsule "C," LRHSC, Assembly Of
ST0620NA0061, Creep Test Capsule "A," LRHSC, Assembly Of
ST0620NA0062, Creep Standard Capsule "A," LRHSC, Assembly Of

ST0620NA0063, Creep Test Capsule "B," LRHSC, Assembly Of
ST0620NA0064, Creep Standard Capsule "B," LRHSC, Assembly Of

Specification ST0609NA0031, Alumina, Application, LRHSC, has been written and reviewed. Formal release is being held up pending further technical investigation.

The following Baseline material specifications were revised and formally released during this quarter:

ST0170NB0048, Tantalum-10 Tungsten Alloy, Bar and Rod
ST0170NB0049, Tantalum-10 Tungsten Alloy, Seamless Tubing
ST0170NB0050, Tantalum-10 Tungsten Alloy, Plate, Sheet, Strip and Foil
ST0170NB0051, Tantalum-8 Tungsten-2 Hafnium Alloy, Plate, Sheet,
Strip and Rod
ST0170NB0054, Tantalum-8 Tungsten-2 Hafnium Alloy, Seamless Tubing
ST0170NB0058, Platinum-10 Rhodium Alloy, Sheet and Strip
ST0170NB0059, Platinum-10 Rhodium Alloy, Seamless Tubing

The revisions in the tantalum alloy specifications were primarily changes in the size of the tensile test specimens. Because of the high cost of these materials, the relatively large standard ASTM specimen will no longer be used. Rather, a minimum size specimen that will provide useful data for material properties control has been substituted. The ASTM testing procedures will still be used.

The revisions in the Pt-10Rh specifications increased the required tensile strength, changed the type of hardness tests from Vickers to Rockwell B, and clarified the microstructure and fabrication history requirements. These changes are based on consultation with the alloy supplier. The changes in no way compromise the quality of the product but rather bring the specification in line with industry practice.

The fabrication and assembly flow charts for Creep Test and Standard Capsules A, B, and C were extensively revised, redrawn, checked and approved during this quarter. Formal drawing numbers have been assigned to the flow charts — they are as follows:

N73420051, Creep Test Capsule "A"
N73420052, Creep Test Capsule "B"
N73420053, Creep Test Capsule "C"
N73420054, Creep Standard Capsule "A"
N73420055, Creep Standard Capsule "B"
N73420056, Creep Standard Capsule "C"

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 LRHSC Capsule Piece Part Fabrication and Assembly Program have been determined and the material ordered. Delivery of the tantalum alloys was divided into four shipments so that piece part fabrication development, weld development, and prototype parts fabrication could be started early while the material for test capsule fabrication was being produced and inspected.

Orders were placed for tool development and fabrication of three prototype parts of each capsule piece to certify the tooling.

Design of weld and assembly tooling has been completed and the tools ordered. As of this reporting data three of the six assembly tools have been completed with the remaining three 90% completed. "TIG" weld heads for the welding of the fuel retainer ring and the outer liner end cap were designed and fabricated.

The four stainless steel capsule mock-ups of the reference capsule design were fabricated. These mock-ups will be used for tooling checkout.

C. ACCOMPLISHMENTS DURING THE QUARTER

1. Tantalum Alloys

Delivery of the tantalum alloy material (Ta-10W; T-111, and unalloyed tantalum) from National Research Corp., has been divided into four categories: weld development tool development, prototype piece part and tooling certification, and test capsule piece parts. Material in the first three categories has been received. Delivery of the material for the test capsule piece parts is scheduled for January as shown in Table VI-1.

TABLE VI-1
LRHSC TEST CAPSULE MATERIAL DELIVERY SCHEDULE

<u>Item of Material</u>	<u>Shipping Date</u>	<u>Status</u>
T-111 Plate Heat # 1	12/01/67	In A. I. Receiving and Inspection
T-111 Plate Heat # 2	1/12/68	Material in NRC Final Inspection
Ta-10W Plate	12/29/67	In Transit
Ta-10W Tubing	12/29/67	In Transit

2. Pt-10Rh Alloys

All Pt-10Rh material for the LRHSC Program was delivered to the LRHSC in-process control center during this reporting period. Bishop Industries chemical analysis indicates that the rhodium content is 10.2% compared to the 9.9% to 10.1% requirement of the material specification. This material was accepted and released without restriction. All other discrepancies were mechanical in nature and dispositioned as "Use As Is w/o Restriction." AI chemical analyses has not been completed.

3. Capsule Piece Part Fabrication

a. Deep Drawing of T-111

All forming operations (i. e., coining and deep drawing) are being performed at Claiborne Metals. The tooling for deep drawing of the T-111 shell structure has been completed and two copper parts have been drawn to determine the blank size required for the draw and also to verify the dimensions of the tooling. Figure VI-1 shows the drawing tools for the five stages required to produce the shell structure.

After satisfactory deep drawing of the two copper shells, two T-111 blanks were drawn utilizing the tooling shown in Figure VI-1. The first blank was drawn through the first stage and stalled the press during the second stage draw. The part was then annealed at 2700°F for one hour at a pressure less than 1×10^{-5} torr. After annealing, the second draw was completed without any problems. The part was then drawn through the third and fourth stage without further annealing. The part cracked on the fourth draw.

The second blank was drawn through the first three stages without annealing. It was then annealed at 2700°F for one hour in vacuum and was subsequently drawn through the fourth and fifth (final) stages. Preliminary examination of the part revealed superficial galling. Figures VI-2 through VI-11 show the sequence of fabrication of the second T-111 blank by deep drawing. Figure VI-12 shows the T-111 shell after it has been sectioned to examine the uniformity of the wall thickness and to measure the hardness along the shell wall and at the spherical radius. Figure VI-13 shows the hardness readings along the inside and outside of the T-111 shell structure.

Figure VI-1. T-111 Structural Shell Drawing Tools

Figure VI-2. T-111 Blank Half Way Through First Draw

Figure VI-3. T-111 Blank Through First Draw

Figure VI-4. T-111 Blank During Second Draw

Figure VI-5. T-111 Blank Through Second Draw

Figure VI-6. T-111 Blank During Third Draw

Figure VI-7. T-111 Blank Completing Third Draw

Figure VI-8. T-111 Blank After Third Draw

Figure VI-9. T-111 Blank During Fourth Draw

Figure VI-10. T-111 Blank Through Fourth Draw
and Being Sized to Final Configuration
(Fifth Draw)

Figure VI-11. T-111 Blank Complete Through All Five Draws

Figure VI-12. Half Section of T-111 Shell Structure

Figure VI-13. Hardness of Structural Shell After
Final Drawing Operation in Rockwell A

Presently, three T-111 shell structures are being drawn as prototype units to certify the tooling prior to production forming of the test capsule shells.

The tooling for the T-111 end caps with the boss for welding of the Ta-10W feed through tube is now in work and approximately 80% completed.

b. Spinning of T-111

As a back up for forming of the T-111 structural shell, a contract has been placed for tool development and forming of three parts by spinning. The four stage spinning forms required for this part have been completed and copper parts have been spun to check out the tooling. Figures VI-14 through VI-17 show the four spinning forms in sequence. Figure VI-18 shows the first copper part spun to check out this tooling.

Figures VI-19 and VI-20 show the results of the first attempt to spin the T-111 shell structure. After first trying to form the metal at room temperature, the T-111 blank was heated to about 1500°F. The metal could still not be formed because it canted in the tooling rather than deforming. This is a tooling limitation that could probably be solved; however, the success of the deep drawing operation has led to the backup T-111 spinning development being terminated.

c. Machining

Final machining of all formed parts and capsule liner details will require very close tolerance tooling and fixturing. All tooling for the final machining operation is shown in Figure VI-21. This tooling includes the holding chucks for the T-111, and Ta-10W end caps plus the inner and outer liner tubes.

Fabrication of four stainless steel Creep "C" Standard Capsules was completed during the reporting period. Figures VI-22 and VI-23 show the fabrication of end caps for the Creep "C" design. The structural end cap with the feed through boss for welding is shown being machined. The radius cutter is shown taking an internal cut on the end cap.

4. Capsule Assembly Tooling Fabrication

Fabrication of all assembly tooling has been subcontracted. The tooling is presently in work with three tools having been completed and delivered to the LRHSC In-Process Control Center for inspection. The tabulated tooling status is presented in Table VI-2. A description of the assembly tooling fabrication status follows.

Figure VI-14. First Stage Spinning Chuck

Figure VI-15. Second Stage Spinning Chuck

Figure VI-16. Third Stage Spinning Chuck

Figure VI-17. Fourth Stage Spinning Chuck

Figure VI-18. Spun Copper Shell

Figure VI-19. T-111 After First Spinning Operation

Figure VI-20. T-111 After First Spinning Operation

Figure VI-21. Final Machining Holding Fixture

Figure VI-22. Radius Cutting of Stainless Steel
End Cap

Figure VI-23. Radius Cutting of Stainless Steel
End Cap

TABLE VI-2
LRHSC ASSEMBLY TOOLING STATUS

Tool No.	Description	Due Date	Expected Delivery
T-6001638	E.B. Weld Fixture, Radioisotope Liners	12/13/67	1/12/68
T-6001643	Press On Tool, Capsule Assembly, Radioisotope	12/01/67	Completed
T-6001645	Chill Block and Automatic Weld Head Locator Radioisotope	12/20/67	1/12/68
T-6001650	Plug Locator Inner to Outer	11/17/67	Completed
T-6001654	E.B. Weld and Assembly Fixture Large Radioisotope Element	12/27/67	1/12/68
T-6001656	E.B. Weld Fixture Structure Shell Feed Through Cap to Outer Liner Cap	12/04/67	Completed

a. E.B. Weld Fixture T-6001638

This assembly tool is used for all electron beam welding of the Ta-10W components up to the simulated fueling of the capsule, and including the welding of the feed through tube extension. Fabrication is 95% complete. All detail parts have been completed and the tool is in final assembly. Figure VI-24 shows the end cap locators for both the standard and creep capsule design. The three locators for the standard design are shown in the background while the locator for the creep capsule end cap with the feed through extension is shown in the foreground. The "lift" table for positioning of the E.B. weld fixture tool is also shown. This tool permits variable height adjustment of the E.B. weld fixture with a parallelism of 0.002 inch.

b. Press On Tool Assembly T-6001643

The function of this tool is to fit all components together prior to electron beam welding on E.B. Weld Tool T-6001638 previously described. The fixture has been completed and delivered to the LRHSC In-Process Control Center for

Figure VI-24. E.B. Weld Fixture T-6001638

final inspection. Figure VI-25 shows the tool with the press on bushing and guide plate in the "open" position. Figure VI-26 shows the press on bushing and guide plate in the "closed" position.

c. Chill Block and Automatic Weld Head Locator T-6001645

The function of this tool is to permit chilling of the capsule during welding. In addition, it serves as a locator base for the automatic "TIG" weld heads. An auxiliary part of this tool is used to press in the fuel closure ring. The tool is 90% complete. All detail parts required for this tool have been fabricated and the copper chill block is being tungsten coated. Figure VI-27 shows the stand and locating heads for the two "TIG" welds.

d. Plug Locator Inner to Outer Liner T-6001650

This tool is used to position the outer liner and inner liner assembly during the final "cold" E.B. weld performed on E.B. Assembly Tool T-6001638. The tool has been delivered to AI.

Figure VI-25. Capsule Press On Tool
(Open Position)

Figure VI-26. Capsule Press On Tool
(Closed Position)

Figure VI-27. Holding Fixture for Chill Block and TIG Welding Heads

e. E. B. Weld Fixture T-6001654

The function of this tool is to permit assembly of the capsule structural cap and shell while in the vacuum chamber. The tool is 95% completed.

f. E. B. Weld Fixture Structural Shell Feed Through Cap to Outer Liner Cap T-6001656

This tool is a locating fixture for the electron beam welding of the outer liner end cap to the T-111 structural shell. The tool is shown in Figure VI-28.

5. Automatic "TIG" Weld Heads

The two weld heads shown in Figures VI-29 and VI-30, are for the automatic "TIG" welding of the fuel retainer ring to the inner liner assembly and the final closure of the outer liner cap to the outer liner sleeve. The figures show the weld heads being hand held in the positions in which they will be held by the Weld Head Locator T-6001645.

Figure VI-28. Outer Liner End Cap to Structure
End Cap Positioning Fixture
for E.B. Welding

Figure VI-29. Interrupted TIG Weld Head Shown in Position for Welding

Figure VI-30. Final TIG Closure Head Shown in Position for Welding

VII. CAPSULE CREEP TESTING

A. TASK OBJECTIVES

The LRHSC Program fuel capsule design 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-10%Rh for oxidation protection of the reactive refractory metal containers. The objective of the task is to provide and test a design approach that accounts for creep deformation at elevated temperatures for this capsule design.

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

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

B. SUMMARY OF TASK STATUS

Three creep rigs will be required for the first year's test operation. Three 1200 l/sec vacuum ion pumps and vacuum chambers with power supplies and vacuum readouts have gone through initial checkout and repair. The design of the test rigs has been prepared. One of the assemblies has been modified and is undergoing final checkout. The remaining two are in the process of being modified.

The pressure control consoles have been designed and built. The three required initially are undergoing final checkout.

The high pressure (> 600 psi) high purity (99.9997%) helium gas supply has been located and three certified vessels delivered.

The coordinate cathetometer for strain measurement has been received. The assembly test fixture has been designed, built, and checked out.

A furnace design has undergone initial checkout been found adequate and is being modified to facilitate strain measurements. Final checkout of the modified furnace is in progress.

A scheme for measuring diametral strain of the capsule in an isothermal chamber during test has been tentatively worked out. The method is undergoing final checkout.

The test rigs will be available as scheduled for operation - the first in mid January, the remaining two the following month.

A direct reading optical pyrometer accurate to 1.8°F @ 2000°F has been obtained and is now being calibrated at the National Bureau of Standards.

A helium gas analyzer capable of measuring active impurities in the ppb range has been received and is undergoing final checkout.

C. PROGRESS DURING THE QUARTER

The major effort during the quarter was expended assembling components, checking out and repairing vacuum systems and power supplies, working out problems with pyrometry, building and checking out furnace designs, building and checking out the pressure control console, designing and building creep test assembly fixtures, working out strain measurement schemes, and designing and checking out the integral creep test assembly. The design of the systems proceeded to the point where it could go no further without experimentation.

As a result of the experiments the final design is essentially complete. The first creep test assembly should be constructed and ready for operation within the first month of the next quarter.

1. Design

- a. Chamber

The chambers which will be utilized for this year's program were initially designed and used for lower temperature and smaller furnace applications. These basic units are shown in Figure VII-1. The reference furnace design selected was a tantalum coil resistance heater utilizing tantalum reflectors and alumina insulators. To provide the thermally uniform volume required for the capsule without guard heaters, it was felt that the furnace might have to be as long as 16". The layout of a furnace this size is shown in a vacuum chamber in Figure VII-2. As can be seen from this figure, the chambers must be modified to locate the center of the capsule at the existing sight ports.

Four approaches to the modification were evaluated. The first two shown in the figure were to move the upper flange higher by about 3-1/2" to keep the feed throughs cool enough for proper operation. Both of these are a very expensive and time consuming operation. The third approach was to lower the sight ports. As can be seen from Figure VII-1 this required removing the brazed tubing, sealing the old ports, moving the flanges and rebrazing the assembly. The fourth approach was to add cooling facilities to the upper flange so that the furnace could be located higher with respect to the view ports.

A flange was instrumented, the feed-throughs that would be required were installed, and the flange was cooled utilizing copper tubing bonded by stake welding and a cement dispersed with graphite. A furnace was assembled and installed in the chamber with the top of the furnace 2" from the flange. With the furnace at 2000°F the flange and feed-throughs remained just above the

Figure VII-1. Three basic vacuum pumps and chambers to be modified for use as creep test systems. The third (farthest) chamber is covered with a bakeout oven.

cooling water temperature which was below ambient. The furnace was then moved up another 1-1/2 inches so that now the capsule mid plane is in the center of the existing sight ports. This second test is now in progress. No difficulties are expected even at temperatures higher than 2000°F. The remaining two flanges will be modified.

All seals in the chamber including the roughing valve are either brazed, welded, metallic gaskets, or a combination thereof. There are three ports on the sides of the chamber and six on the upper flange. Two of the side ports are for viewing the capsule mid plane at two radial locations. They are optically flat windows brazed to a pipe which is in turn welded to a flange. This flange is then bolted to a mating flange on the chamber; sealed by a copper gasket and knife edges on the mating flanges. The third port is for the ion gauge which is brazed much like a vacuum tube to the flange. The flange is then bolted onto the mating flange and sealed with copper gaskets in the same manner as the windows.

Two of the upper ports are power feed-throughs. They consist of copper rods brazed to an alumina insulator which is brazed to a flange. One upper port is used for the capsule pressure feed-through. The sealing of the capsule pressure feed-through to a flange will be accomplished by TIG welding. Another of the flanges is used to operate the shutter for the windows. Two types of shutter drives have been tested and found adequate. Both designs utilize a technique which does not incorporate any sliding seals. One uses magnets, the other an eccentric bellows as shown in Figure VII-3.

One upper feed-through is for thermocouples. Since Pt and Pt-10%RH high vacuum feed-throughs have not been found commercially available, iron base alloy feed-throughs had to be utilized. A schematic of two approaches for these seals is shown in Figure VII-4. Figure VII-4a shows the first approach that was tried. This arrangement allows brazing a series of feed-throughs at one time. A new harness can be made up simply by spot welding on the sensors when required. It was a simple matter to spot weld a new TC on the feed-through when a replacement was necessary. However, it was found when heat was applied to either the spot weld (Pt to Pt, Pt-10Rh to Pt-10Rh), or the braze of the feed wire to the through tube, a drift in apparent temperature

Figure VII-3. Bellows Rotary Seal

was noted. Because of this problem, the arrangement has been changed to that shown in Figure VII-4b. This design is more time consuming to replace, decreases pumping efficiency (because of trapped gases in the through tube), and is harder to make. The modified braze joint is difficult to fabricate since it is only 1/4" away from the glass to metal seal. However, this arrangement does not cause an uncompensated drift in the TC emf.

The sight windows are designed to rupture at 2 atm ΔP . To assure that they do not rupture in the event that the pressure system dumps the high pressure gas into the chamber, the sixth flange is a burst diaphragm. It is a thin member welded to a stainless steel pipe which when evacuated, merely deflects. However, when pressure is applied to the vacuum side it deflects against a knife edge and ruptures. It is designed to rupture at 1 atm ΔP inside to outside.

All of the flanges bolt and seal to mating flanges with copper gaskets. All of the systems except the burst diaphragm have been checked out and work adequately up to 500°F and a vacuum of 10^{-8} torr.

b. Furnace

The furnace and controller for the creep tests is required to provide an isothermal ($\pm 5^\circ\text{F}$) volume at 2000°F for a creep capsule and coupon stack together. In addition, there must be visual access to the diameter of the capsule at its axial mid plane in two radial locations. The purpose of the visual access is to measure the diameter of the capsule during tests. Access to the black body by the optical pyrometer must also be provided. In order to prevent a reduction in radiation to the pyrometer, no parts of the furnace may encroach upon a right cone 12" high (focal length of lens) by 1-1/2" base diameter (diameter of lens) with its apex at the black body. This is not necessary with the strain measuring access ports because of the nature of the telemicroscope of the cathetometer.

Based on initial trials a reference furnace design was selected and tests were performed to further evaluate the design. The test setup is shown in Figure VII-5. The furnace is 16" long and 8" in diameter. The temperature is supplied by resistance heating unalloyed tantalum coils. The heating element

Figure VII-4. Schematic Representation of T/C Vacuum Feed-Through

is insulated from the 7 tantalum radiation shields by tungsten coated alumina insulators. The tungsten coating is to prevent interaction of the heater and insulator in vacuum.

A capsule simulator (unalloyed tantalum cylinder) 1.7" diameter x 10" long was supported on an alumina pedestal. The cylinder was instrumented to determine the axial and radial temperature distribution of the assembly. For this basic test the access ports were not put into the assembly.

The temperature distribution in the test furnace at 2050°F was:

<u>T/C No.</u>	<u>Temp. (°F)</u>
Top of Capsule	2047
Capsule Midplane	2055
180° Capsule Midplane	On Controller
Capsule Bottom	2172*
Coupon Stack	2051
4th Shield Midplane	1631*
7th Shield Midplane	950
7th Shield Bottom	1495

* These T/C's were brazed by method in Figure VII-4a.

Adding the correction factor indicated by the calibration report of Figure VII-6, the variation in the furnace from top to bottom was only 5°F.

After the very promising results of the first test the furnace and chamber were disassembled and modified for the next test. The layout of this test is shown in Figure VII-7. The thermocouple harness put into the system to check the drift noted in the Standards Laboratory was removed and replaced.

Sight ports were put on the side of the chamber and the rotating feed-through moved to a flange where it could open both windows with one motion.

Holes were punched into the heater shields to allow visual access to the capsule edges and black body. To facilitate positioning and viewing the capsule, the following scheme was used. Early tests indicated that viewing the capsule in the isothermal furnace (effectively a black body) was virtually impossible.

ATOMICS INTERNATIONAL
A DIVISION OF NORTH AMERICAN AVIATION, INC.
8900 DESOTO AVE., CANOGA PARK, CALIF.

Standards Laboratory Certificate

THERMOCOUPLE CALIBRATION

4 Pt vs. Pt 10% Rh Thermocouples (.040" wire)
(Connected through vacuum seal;
extended with compensated leadwire)
Submitted by: Paul Ferry
Dept. 737

C E R T I F I C A T I O N

All measurements on which this report is based were made with reference to company primary standards that are calibrated at regular intervals by the National Bureau of Standards. Reports of calibration and test data documenting the traceability of these measurements to national standards in a known and valid manner are on file in the laboratory issuing this report.

The thermocouples described above have been compared to secondary standard Pt vs Pt 10% Rh thermocouples. The repeatability of the intercomparisons, based on several sets of measurements, appears to be no better than $\pm 2^\circ\text{F}$. It was found that heat, either conducted or radiated, to the silver-soldered seal region can cause significant error so that an effort should be made to minimize the temperature variations at that area. (for example: a solder gun not touching either wire, held about 30 seconds, can cause about a 10°F error in the plus direction.)

The thermocouple corrections are summarized below:

Tc #	CORRECTIONS @ $\approx 1920^\circ\text{F}$	
	mv	$^\circ\text{F}$
2	-0.0221	-3.4
3	-0.0104	-1.6
4	-0.0169	-2.6
7	-0.0306	-4.7

The cause of the relatively broad uncertainty in the repeatability reported above is being evaluated further. With Pt vs Pt 10% Rh thermocouples, the uncertainty of intercomparisons should be narrower than we are able to report for this case.

Temperature Standards
Dept. 737-744, Ext. 1617
Test No. T67332
Dates of Test: Dec. 4, 5, 6, 1967

FORM 734-G-1 NEW 7-67

Figure VII-6. Sample Certification of Calibration of each of the thermocouples which are used in the creep tests.

AI-AEC-12642

Test No. T67332

Dates of Test: Dec. 4, 5, 6, 1967

ACCURACY OF STANDARDS

The National Bureau of Standards Certificate of Calibration for our primary standard platinum vs platinum 10% rhodium thermocouples states that the uncertainty in the reported values is not more than 0.3 degree (on the International Temperature Scale of 1948) in the range 0° to 1100°C. The uncertainty increases from 0.3° to 2° from 1100° to 1450°C. In transferring these calibrations to our secondary standard working couples, the stated uncertainties are increased by not more than 0.2°C. These uncertainties represent about twice the standard deviation of observations so that any errors incurred will not exceed the stated uncertainties 95% of the time.

Marjorie D. Gerber
Temperature Standards

L. P. Inglis, Supervisor
Instrument Applications & Standards

Figure VII-6 (Continued)

A pair of alumina tubes were scalloped and drilled as shown in Figure VII-8a. The scallop radius is about 0.030" larger than the capsule radius. In practice these tubes will be assembled around the capsule supported only by the thin tantalum radiation shields. Figure VII-8b shows the sight tubes in contact with the capsule. As can be seen from the figure, the scallops serve two functions. They provide positioning of the capsule in two planes - one for viewing the capsule edges down the tubes, the other for viewing the edges through the lateral holes. This scheme also allows smaller holes to be punched into the heat shields, to allow for movement of the capsule away from the sight ports. For installation, holes are punched in the first three of seven radiation shields opposing the view ports so that the edge of the capsule can be seen against a cold background.

The sight tubes and mock-up capsule are shown in the furnace in Figure VII-8c. This also shows one of the advantages of the coil type vs the gauze furnaces. The access holes need be made in the heat shields only, the heating element need not be disturbed. In actual operation a series of heat shields will also be added to the sight tubes. A typical arrangement is shown in Figure VII-8d. These are added to reduce the radiation of the capsule to the sight holes, and hold the black body in position. The first two shields close to the capsule hold the black body, the remaining position it and provide a conical path from the target to the optical pyrometer. One can see the sight hole in the front shield in the figure. It is planned to add a very thin foil (0.0005") of tantalum around the tubes to increase their reflectivity and physically separate them from the capsule. It was not deemed necessary for this test.

The coordinate cathetometer that will be used for measuring the capsule diameter during test is shown in Figure VII-8e, in position to look down the sight tubes. The cathetometer is accurate to ± 0.00005 in. when viewing a sharp edge. If the curvature of the capsule causes the edge to be fuzzy, small bosses will be tacked to the capsule to facilitate measurement.

This second test with sight tubes and access ports has been assembled, evacuated, baked out, and is being brought up to 2000°F. At the present time, it is at 1600°F.

a. Scalloped Sight Tubes

b. Sight tubes in contact with mock capsule indicating location of sight holes

Figure VII-8. Scheme for measuring capsule strain in black cavity at 2000°F.

c. Installation of sight tubes in furnace with mock capsule. For clarity the black body and radiation shields are not shown in this view. Note that the sight tubes extend through all shields on the entrance side, and through only three on the opposite side.

d. Strain access subassembly showing the relationship of sight tubes, black body, and radiation shields with capsule.

Figure VII-8 (Cont.) Scheme for measuring capsule strain in black cavity at 2000°F.

e. Coordinate Cathetometer in position to measure capsule strain through the chamber view port.

Figure VIII-8 (Cont.) Scheme for measuring capsule strain in black cavity at 2000°F

The sight ports seem to work very well. Figure VII-9 is a composite photograph viewing down the sight ports past the edge of the capsule to the fourth opposing heat shield. The edge of the capsule is clearly defined in the sight tubes. In order to further test the requirement for penetrating the opposing heat shields, they were not removed from the sight line on the second view. It is virtually impossible to discern the edge of the capsule in this view. The shields will be punched for the next test.

The spot in the center of the photograph (Figure VII-9) is the sight port to the black body. The shields have shifted slightly and encroach on the visual cone. A fixture will be added to remedy this.

Figure VII-9 View of capsule edges down sight tubes at temperature In the blow-up of the capsule, the edges of the capsule and the black body can be seen The heater coil in the black body sight hole will be moved to prevent an indicated temperature error

This test is progressing sufficiently well to indicate that the furnace design and construction will be evolved to meet the technical and schedule requirements of the program. Once a proven design has been established it is only a matter of weeks before duplicate systems can be constructed and ready for operation.

2. Assembly Fixture

A fixture has been designed, assembled and checked out for assembling and installing the creep test. Because of the design of the chamber, the test is completely assembled to the upper support flange and then lowered into the chamber. The assembly fixture and sequence is best illustrated by the following figures. The assembly sequence figures were taken during the assembly of two different tests. One can note small differences such as window shutter, feed-through, chamber style, etc. However, the assembly fixture and sequence is compatible to all of the systems and is shown here.

The support flange is bolted to the basic cherry picker mounted on a table that is shown in Figure VII-10a. The cart is movable but is stabilized by counter weights and allows working at a convenient height. After the support plate is mounted, the vacuum feed-throughs are secured and tested for function (i.e. rotate, conduct, insulate, etc.). The furnace support rods are then screwed into the flange. A key way is provided in the threaded male section to provide a path for the trapped gases in the threads. The thermocouples for inside the furnace are fed through the upper shields. The upper shields of the furnace are then brought into place and held during primary assembly by a support wire secured from slipping by gloves in the support rods. The test piece (supported from the floor if not attached to the upper flange) is then brought into place and the thermocouples attached to appropriate locations, as shown in Figure VII-10b. Once the test piece is properly instrumented the furnace is then brought up around the test piece as shown in Figure VII-10c. The bottom support plate which is insulated from the heat shields by alumina tubes is then bolted to the support rods. A finished subassembly is shown in Figure VII-11a.

After the subassembly has been checked out, it is clamped to the forks of a portable hydraulic lift and unbolted from the table as shown in Figure VII-11b and Figure VII-11c.

a. Bare flange mounted to assembly support fixture

b. Detail of instrumentation assembly sequence. The support wire and capsule support tube are removed upon final assembly.

Figure VII-10. Creep Test Assembly Sequence

c. Final Assembly of the capsule subassembly.
Figure VII-10. Creep Test Assembly Sequence

Figure VII-11a. Completed Creep Capsule Subassembly and Fixture

Figure VII-11b. Completed creep capsule subassembly and assembly table.

Figure VII-11c. Assembly fixture is unbolted from table and clamped to tines of hydraulic lift.

The subassembly is then rolled over to the vacuum station, and lowered into the chamber (Figure VII-12a, b, and c). Here again the cantilever nature of the assembly fixture is required so that the feet to the lift do not interfere with the vacuum chamber. Once the upper flange is seated, the assembly fixture is removed. The fixture has been checked out during two tests and found to work quite well.

a. The Capsule Creep Test Furnace ready for insertion in the Test Chamber.

Figure VII-12. Sequence of inserting creep capsule subassembly into vacuum chamber.

b. The assembly is then lowered into chamber and
fixture removed

c. The assembly is then lowered into chamber and
fixture removed

Figure VII-12. Sequence of inserting creep capsule subassembly into vacuum chamber.

3. Pressure Attainment and Control

a. Attainment

A number of schemes for obtaining the highest purity helium at high pressures were evaluated. Evaluation indicated that evaporation of a liquid source could not achieve the purities claimed by the best chromatograph grades of the available at 2200 psi on a specialty basis (1 ppm active impurities). However, no vendor had supplied nor certified a source of chromatograph grade in excess of the standard 2200 psi. The major problem lies in the contamination of the gas during the next pressurization cycle from 2000 to 6000 psi. One supplier volunteered to supply helium in excess of 6000 psi and 99.9997% pure. Of those 5 ppm present, three would be neon. The active impurities would then be less than 2 ppm. Three of these sources were procured, are in the laboratory, and the supplier's certified analysis for one of the bottles (typical of all three) is shown in Figure VII-13.

The helium gas analyzer capable of measuring impurities in the ppb range has been procured and is now undergoing final checkout in the laboratory. This analyzer replaces the more antiquated analysis by concentration, by distillation and/or liquefaction which was previously employed. The older method was more expensive, time consuming, and not very accurate.

b. Control

Three pressure control systems have been constructed. They provide control of the gas from the sources to the capsule. The first is undergoing final checkout and should be ready within a week. The remaining two are scheduled for delivery to the laboratory the week thereafter.

4. Temperature Attainment and Control

The furnace has already been described. The control of the furnace power will be by a proportioning controller firing silicon controlled rectifier units. A few 20 amp units have been assembled and during initial tests have proven to be adequate. Once the final design has been tested, the console in Figure VII-14 which controls 50 amp SCR's, will be wired into the system. The final checkout will not be complete by mid January so the first tests will be run

THE LA FLEUR CORPORATION

CERTIFICATE OF COMPLIANCE

THE LA FLEUR CORPORATION, 2001 WEST ARTESIA BOULEVARD, TORRANCE,

CALIFORNIA, CERTIFIES THAT: Helium Gas - Grade 5.5 6000 PSI +

SUPPLIED IN ACCORDANCE WITH Atomics International

PURCHASE ORDER NUMBER: N-7270220 IS EQUAL TO OR

SURPASSES A PURITY OF: 99.9997%

IN ACCORDANCE WITH: P.O. # N-7270220

BY:

(SIGNATURE & TITLE)

DATE: October 13, 1967

Figure VII-13. Sample Manufacturer's Certification of 6500 psi helium supply bottle.

INDUSTRIAL GAS DIVISION
2001 W. ARTESIA BLVD,
TORRANCE, CALIFORNIA, 90504

PHONE : 770-0175

GAS ANALYSIS REPORT

CUSTOMER'S ADDRESS : _____ DATE : October 13, 1967

North American Aviation/Rockwell Corporation P.O. NO: N-7270220

Atomics International Division

8900 De Soto Street

Canoga Park, California
GAS SAMPLE TO BE ANALYZED : 6000 PSI Plus Helium - Grade 5.5

CONTAINER - CUSTOMER OWNED : _____ SERIAL NO: _____
LA FLEUR OWNED : X SERIAL NO: 1160

ANALYSIS REQUESTED : TOTAL IMPURITIES

DATE CYL. RECEIVED AT LAB: 10-2-67 DATE ANALYZED: 10-2-67

RESULTS :	Hydrogen	<u>0.1</u>
	Neon	<u>0.7</u>
	Oxygen	<u>0.25</u>
	Nitrogen	<u>0.26</u>
	Moisture	<u>1.3</u>
	Hydrocarbon	<u>0.04</u>
	ALL OTHER	<u>NIL</u>

ACCOUNTING DEPT. COPY :

FIELD TECHNICIAN'S TIME : _____ HOURS : _____ MIN : _____

MILEAGE : _____

MISC. EXPENSES CHARGEABLE TO CUSTOMER : _____

22 The
LABORATORY TECHNICIAN

FORM: IG-39

Figure VII-13 (Continued)

Figure VII-14. Power control console (left) and recording console (right) which will be wired into the creep systems as required.

from the experimentally assembled units. The console to the right in the figure was set up for temperature recording, however, because of the cost of installation, it will not be used until more of the systems are on the line.

While continuous control of the systems will be by the Pt-10% Rh T/C's triggering the controller, the drift of the T/C's will be monitored and compensated for by use of a direct reading optical pyrometer precise and accurate to 1.8°F at 2000°F. The procedure for this is best described by the following excerpts from the tentative creep testing specification.

3.7.3.1 Determining the Constant Temperature Factor When the creep capsule reaches test temperature (as indicated by the temperature controlling midpoint thermocouple), the indicated temperature of the black body positioned on the side of the capsule shall be determined with an optical pyrometer. The indicated temperature difference between the midpoint thermocouple and the black body shall then be used as a constant factor for determining the temperature drift during subsequent temperature checks.

3.7.3.2 Temperature Drift Using the following schedule, determine the indicated temperature difference between the controlling midpoint thermocouple and the black body. If the indicated temperature differs by more than 5°F from the constant temperature factor (refer to 3.7.3.1), the temperature drift shall be compensated for by adjusting the temperature controller. The indicated temperature of the black body shall be used as the standard for adjusting the controller.

Mode I	Midpoint and at the end of test.
Mode II	After 10, 25, 50, 100, 200, 400, 800, and 1200 hours of test and at the 8,000 and 10,000 end of test.
Mode III & IV	After 10, 25, 50, 100, 200, 400, 800, 1600, 3200, 4800, 6400, and at the end of test.

The thermocouples are calibrated in the Atomics International Standards Laboratory in which the precision of the thermocouples are traceable to the National Bureau of Standards.

The optical pyrometer has been fabricated with a special hand selected reference lamp and is now at the National Bureau of Standards for calibration. Until its delivery, an identical pyrometer without the selected lamp, precise and accurate to within $\pm 4^{\circ}\text{F}$ at 2000°F is being used. The black body is a standard 10:1 tantalum cavity to minimize changes in emissivity and reflectivity of the test piece.

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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 testing schedule has been revised from that reported last quarter. The new schedule shown in Table VIII-1 plans on the use of one compatibility testing rig and creep rig # 1 until it is needed for creep testing. The change in test schedule will have no effect on the task milestones.

2. Summary of Task Status

To date, two coupon stacks have been tested and one is in test. CS2 (2000°F - 100 hr.) was tested in November. The stack was gamma-graphed and neutron radiographed before and after test, prepared, examined and photographed in the metallographic laboratory, and is currently ready for electron beam microprobe analysis.

CS4 (2250°F - 10 hr.) was also tested in November. The stack was neutron radiographed before and after testing. It has been vacuum impregnated with mounting material and sectioned for metallographic inspection. Some preliminary results and photographs have been obtained.

CS3 (2000°F - 1000 hr.) is currently on test. The test began on December 14 and nearly 400 hours of test time had elapsed at the quarter's end. This test will continue until January 23, 1968.

TABLE VIII-1

SCHEDULE OF COUPON COMPATIBILITY TESTING

<u>Coupon Stack No.</u>	<u>Test Start Date</u>	<u>Temp. °F</u>	<u>Atm Torr</u>	<u>Time of Test (Hours)</u>	<u>Test Status</u>
CS2	11-06-67	2000	10^{-8}	100	Complete
CS4	11-21-67	2250	10^{-8}	10	Complete
CS3	12-14-67	2000	10^{-8}	1000	In Progress
CS7*	1-03-68	2500	10^{-8}	10	
CS8*	1-08-68	2500	10^{-8}	100	
CS5	1-25-68	2250	10^{-8}	100	
CS6	2-03-68	2250	10^{-8}	1000	
CS1	2-03-68	RT	760	0	Complete
CS9**	3-02-68	2000	10^{-8}	2200	
CS10**	3-07-68	2000	10^{-8}	1800	
CS11**	5-15-68	2000	10^{-8}	1000	

* To be tested in Creep Test Rig #1

** To be tested with creep capsule 25, 19 and 22 respectively

CS7 (2500°F - 10 hr.) is being loaded into creep test rig # 1 along with a creep test checkout assembly. This short test should begin in early January.

The capability of the compatibility test rig to perform within the original specifications ($\sim 10^{-8}$ torr and $\pm 5^\circ\text{F}$ at 2000°F and 2250°F) has been demonstrated. After two tests, the idiosyncrasies of the apparatus were sufficiently understood to attain test conditions from a cold atmospheric chamber in only 66 hours.

Preliminary observations from CS2 and CS4 indicate that:

- 1) T-111 and Ta-10W are severely attacked by iron titanate.
- 2) A very thin layer of Al_2O_3 is sufficient to prevent Pt-Rh vs T-111 reactions.
- 3) In spite of large differences in the mechanical and chemical properties of the stack constituents, polishing and etching for metallographic investigation can be accomplished.
- 4) Changes in the microstructure near the surface were observed in CVD coated T-111 specimens.
- 5) Welded areas in T-111 showed different metallographic structure than the parent T-111 but no apparent difference in reaction characteristics.

3. Accomplishments During the Quarter

The major activities during the quarter began with the check out of the testing facilities. During this period the compatibility test coupons, holders and loading tool were being fabricated and surface treated.

The basic stack contained 14 discs consisting of Pt-10Rh, T-111, Ta-10W and alumina. These are displayed surrounding the stack holder and end plug in Figure VIII-1. The sequence of loading the stack of coupon discs into the holder is shown in Figure VIII-2. The clean discs are loaded into the test holder by means of a loading tool. This tool is designed to load the stack into the holder such that the end discs are seated in the end plug recesses and the disc edges remain separated from the holder walls. The situation just prior to loading are shown in Figure VIII-3 and VIII-4.

Figure VIII-1. Compatibility Test Holder Surrounded by the 14 Coupon Discs to be Tested.

7-05-245-17

Figure VIII-2. Arrangement of Coupon Discs in Compatibility Test Stack Showing Thermocouple Locations.

Figure VIII-3. Coupon test holder and coupon stack. The stack is arranged so that the loading tool can be placed over it and then inverted.

Figure VIII-4. Coupon stack inserted into loading tool so that coupon test holder can be placed over the tool. The entire assembly is then inverted and the tool removed.

After loading the stack, the coupon test capsule is instrumented with three Pt-Rh thermocouples as shown in Figure VIII-5 and loaded into the testing facility on a tantalum tripod as shown in Figure VIII-6. The tripod rests on the tantalum heater bottom reflectors and shields. The heater and radial shields are assembled in the test facility and a pyrometric black body sighting block is placed on the top of the capsule as shown in Figure VIII-7. Finally, the top shield of the heater which has a central sighting hole is attached and a shield thermocouple is added. The assembly is then mounted in the bell jar as shown in Figure VIII-8. The bell jar seats on a copper O-ring and is equipped with two view ports. These view ports are protected when not in use by rotating shields which are controlled by a magnetic, vacuum-tight mechanism.

Operation of the system after bolting down the bell jar consists of pumping for 30 hours with liquid-nitrogen-driven sorption pumps while heating the bell jar and the ion pump to 450°F. This is followed by a 20 hour pumping with the ion pump while slowly bringing the heater up to test conditions. By these procedures, 2000°F at 2×10^{-7} torr can be attained in only 66 hours. The instrumentation used in operation of this test facility is shown in Figure VIII-9. In the figure are shown the test assembly, thermocouple temperature controller, temperature recorder, ion gage and ion pump controls, optical pyrometer and associated photometer, galvanometers, potentiometers and recorders.

The testing of CS2 (2000. F for 100 hr.) began with gamma-graphing the capsule prior to installation into the test rig. X-ray radiography was ruled out due to the heavy section of dense tantalum to be examined. It was hoped that pre-and post-test gamma-graphs across two diameters would show any gross shifting or growth of discs during exposure to test conditions. However, the resolution in these photos was of poor quality. The capsules were also neutron radiographed but with no better results. The possibility of using sophisticated screening techniques to obtain the desired resolution is being investigated.

After testing and removal from the test assembly, the capsule was vacuum impregnated with a mounting compound and diametrically sectioned with a diamond saw. This was followed by a series of careful grinding and polishing steps which revealed the structure shown in the 4X macrograph of Figure VIII-10. This figure shows the location of the three paths through

COUPON TEST ASSEMBLY

Figure VIII-5. LRHSC Coupon Compatibility Test Assembly.

Figure VIII-6. Coupon test assembly setting on a tripod support which rests on the bottom of the furnace.

Figure VIII-7. Coupon test assembly placed in the heater. Note the pyrometric black body on top of the stack.

Figure VIII-8. Compatibility test facility just prior to mounting of the bell jar.
The Ion pump is in the background behind the lower bell jar.

Figure VIII-9. Coupon compatibility testing facility complete with associated vacuum, power, thermocouple and optical pyrometer instrumentation.

Figure VIII-10. 4X Macrograph of CS2 After Testing
(2000°F - 100 Hr.)

the interfaces which are being investigated. The obvious curvature of one T-111 disc was due to an attempt to crack the alumina coating by centrally stressing the disc. The yield point of the T-111 was reached before the alumina cracked.

The highlights of the metallographic investigations performed on CS2 are shown in Figure VIII-11. The significant observation is the severe attack of the T-111 by iron titanate. Some reaction had been expected at the interface of T-111 and Pt-10Rh. It was discovered that in CVD coating one face of the coupon with Al_2O_3 , a very thin yet effective barrier was deposited on the black face. This layer was apparently sufficient to prevent a thermodynamically probable reaction. Note the heat affected structure in the T-111 discs where a full penetration EB weld was performed. The welded regions showed no apparent difference in reaction characteristics from the parent metal. Note also the depth of structureless area in the CVD coated T-111 coupons. Previous analyses have shown that no major changes in alloying constituents have occurred in this region.

A closer look at the reacted areas (100X) is shown in Figure VIII-12. Note the gross intergranular corrosion shown in the T-111 wafer. In this particular view it appears that Ta-10W does not suffer from this type of attack. However, when the half capsule was sectioned at 90°, it was observed that Ta-10W was indeed attacked (Figure VIII-13). The stack is now being examined by EBMA.

CS4 (2250°F - 10 hr.) is currently undergoing preliminary metallographic analysis. Some of the results of this test can be viewed in Figure VIII-14. Here again the attack of both T-111 and Ta-10W by iron titanate can be seen. The specimen has been completely etched thus giving a very decorated display. This sample will be thoroughly investigated in the near future.

The first 1000 hr. test at 2000°F (CS3) is running at this time with very good temperature control and at a vacuum of 1×10^{-8} torr. CS7 is being loaded into creep rig # 1 for testing at 2500°F.

Figure VIII-11. Results of 100 Hr., 2000°F Compatibility Stack Test

250X Reduced by 2.64
by Reproduction

7-D7-289-34

Figure VIII-12. Detailed Coupon Stack Results

Figure VIII-13. Apex view of a quarter section of CS2.
Note the gross attack of both T-111 and Ta-10W
by iron titanate.

Iron Titanate
on Pt-10Rh

Severe attack in
this entire region

T-111

Ta-10W

Iron Titanate
on Pt-10Rh

Figure VIII-14. Macrograph (4X) of CS4 (2250°F - 10 Hr.)
in etched condition.

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 for 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

Evaluations of the first arc-plasma sprayed coatings of iron titanate indicated that a chemical and crystallographic change occurred during spraying. Examination of the $\text{FeO}-\text{Fe}_2\text{O}_3-\text{TiO}_2$ phase diagram showed that this change could be expected and, further, that additional change could occur during exposure of the coating to vacuum at 2000°F .

Iron titanate powders from several suppliers were then examined and found to vary in chemical composition, particle size and crystal structure depending on where the material was mined and what, if any, further treatment or blending was performed.

Pratt & Whitney was contacted to resolve the questions of starting powder characteristics and spraying parameters. As a result of these discussions, Continental Coatings Corp. FCT-11H powder has been adopted as the reference starting material and the material and process specifications are being revised.

b. Alumina

Both the chemical vapor deposition (CVD) and arc plasma spraying (APS) processes for producing dense, adherent, crack-free α Al_2O_3 coatings have been evaluated. The CVD process has met all the requirements including lack of interstitial contamination of the T-111 substrate. This is now the reference alumina coating process. The APS process produced non-uniform, poorly adherent, porous, γ alumina coatings. Development of this process has been terminated.

Both the process reproducibility and the thermal cycling stability of the CVD alumina coatings has been demonstrated and a process specification has been written.

The use of a flash coating of tungsten on the T-111 substrate to prevent or minimize substrate contamination was evaluated. The data failed to show any clear evidence of additional protection by the tungsten flash.

3. Accomplishments During the Quarter

a. Iron Titanate

The initial iron titanate coatings arc plasma sprayed onto a Pt-10Rh substrate have been evaluated by X-ray diffraction and X-ray fluorescence. The starting material (Am PoW FeTiO), obtained from AstroMet Associates, Incorporated was predominantly pseudobrookite with a particle size range of 5-43 microns. The sprayed coating was ilmenite with an Fe/Ti ratio of 1.66.

A sample of a coarse grade of iron titanate sprayed onto a platinum substrate by Fansteel Metallurgical Corporation, Los Angeles, was analyzed and found to be totally pseudobrookite. Powder particle size appears to have a significant effect on the as-sprayed crystal form.

Two samples of iron titanate plasma spraying powder were received from Continental Coatings Corporation. These were a commercial grade (FCT-FS) and an extra fine cut of "100% pseudobrookite".

The analysis of the FCT-FS showed it to be mostly hematite (Fe_2O_3) and rutile (TiO_2), with a small amount of pseudobrookite. The "100% pseudobrookite" sample was representative of the naturally occurring material mined in Florida

up until three years ago. Analysis of this powder showed a mixture of rutile (TiO_2) and pseudobrookite ($\text{Fe}_2\text{O}_3 \cdot \text{TiO}_2$) with about three times as much rutile as pseudobrookite.

Obviously, the composition and crystal structure of commercial iron titanate powders is highly variable. Resultant coatings and, therefore, performance should also be highly variable. In investigating the question of what coating is needed and what starting powder should be used to obtain a high emittance coating for long term use in vacuum at 2000°F , the $\text{FeO-Fe}_2\text{O}_3\text{-TiO}_2$ phase diagram, Figure VIII-15, was studied. This ternary diagram shows that at temperatures in the range of $2000\text{-}2300^\circ\text{F}$, a sufficiently large oxygen underpressure (vacuum) will alter the equilibrium structure of compounds. In a vacuum of 10^{-8} torr and at 2000°F , the structure of pseudobrookite will change along the arrowed path to an ilmenite type structure. The rate of change is not presently known nor is the effect of this change on coating adherence and emittance. To properly evaluate the iron titanate system so that the best coating can be identified would require an effort beyond the scope of the current LRHSC contract. Therefore, the work on iron titanate for NASA by Pratt & Whitney Company will be used for selection of a reference iron titanate coating.

The "standard" coating material being used on the NASA Program at Pratt & Whitney is Continental Coatings FCT-11H grade iron titanate. A sample of the LRHSC Pt-10Rh was arc plasma sprayed with this material at Pratt & Whitney and returned to AI for analysis. The FCT-11H starting material is a mixture of rutile (TiO_2) and amorphous material. As sprayed, the coating was almost completely pseudobrookite (Fe_2TiO_5). This coating has now been adopted as the LRHSC Program reference iron titanate coating. The material and process specifications are being revised so the Pratt & Whitney coatings can be duplicated. Until sufficient data are available to warrant a change, all compatibility stack and creep "C" capsules will use this coating.

b. Alumina

(1) Chemical Vapor Deposition

Processing parameters were worked out on Specimens C-1 through C-10, as reported last quarter, and a reference process was established. The reference coating temperature is $1950 \pm 25^\circ\text{F}$ and the oxidant is room air.

THE SYSTEM FeO—Fe₂O₃—TiO₂ AT 2200°F

7-05-245-71

Figure VIII-15. Ternary diagram.

The reference reagent concentration was "superdilute", a term devised to protect the coating vendor's proprietary interest. Specimens C-11 through C-15 were coated by the reference process in separate coating runs in order to investigate process reproducibility.

In general, the coatings were quite reproducible. Also, they were not particularly sensitive to the processing variables, within the limits of this investigation. Coating thicknesses of the "C" series varied between slightly less than 1 and almost 4 mils as shown in Table VIII-2, but were quite uniform over each sample and for each set of process parameters. Surface finish was also reasonably uniform from specimen to specimen. Dye penetration testing showed the "C" series to be almost flaw-free; the few flaws were quite small. Without exception, the greatly predominant phase in all of the "C" series coatings was the desired $\alpha\text{-Al}_2\text{O}_3$.

Microhardness indentations made at about 0.002 in. intervals in the metallographic sections showed in most cases a hard layer in the T-111 immediately adjacent to the Al_2O_3 - T-111 interface as seen in Figure VIII-16. This, however, vanished within a few mils of the interface. The hardening was attributed to superficial contamination with interstitial impurities. Bulk chemical analyses of selected T-111 substrates of the C series showed them all to be slightly contaminated with C, N_2 , and H_2 during processing. The levels of contamination, however, are considered acceptable.

Four specimens of the C series were thermally cycled in a vacuum between room temperature, 2000°F, and room temperature in 4 hours. After 10 of these cycles, two of the specimens were dye penetration tested. There were indications of extremely fine cracks. However, the Al_2O_3 showed no tendency to spall from the specimens. Also, no substrate contamination resulted from thermal cycling and, as shown in Figure VIII-17, the surface hardening was removed by the thermal cycling.

In summary, these preliminary coating experiments have demonstrated that small T-111 specimens can readily be coated with high quality $\alpha\text{-Al}_2\text{O}_3$ by CVD. Potential difficulties associated with scaling-up to coat full-size LRHS capsules remain to be explored.

SPECIMEN NUMBER	SURFACE FINISH, μ -in. RMS	MICROHARDNESS R_c
C-1	120	52, 53, 36, 32, 38, 32, 30
C-2	90	56, 54, 58, 56, 58, 53, 50, 53, 43, 43, 42, 41, 39, 32
C-3	60	56, 42, 36, 31, 32, 26, 27
C-4	70	49, 31, 31, 27, 26, 26, 23

NOTE: THE HARDNESS VALUES ON THE LEFT WERE MADE IN THE IMMEDIATE VICINITY OF THE Al_2O_3 - T-111 INTERFACE. SUBSEQUENT VALUES WERE MEASURED AT INCREMENTS OF 2 mil INWARD FROM THE INTERFACE.

500X

MICROHARDNESS IDENTATIONS ON C-1

7-05-245-32

Figure VIII-16. Post-Coating Evaluations

250X

CVD Al_2O_3 AFTER 10 CYCLES FROM RT TO 2000°F TO RT
(4 hr PER CYCLES)

- NO LOSS OF ADHERENCE
- SOME FINE CRACKS FORMED
- NO CHANGE IN BULK SUBSTRATE COMPOSITION

7-D4-289-21

Figure VIII-17. Thermal Cycling of CVD Al_2O_3 Coatings

TABLE VIII-2

LRHSC Al_2O_3 COATING EVALUATIONS

Specimen Number	Coating Thickness in. (1)	Surface Finish, μ -in.	Dye Penetration Results	X-Ray Diffraction Principal Al_2O_3 Phase	T-111 Micro-hardness, R_c (2)	T-111 Bulk Chemical Analyses, ppm			
						O_2	N_2	H_2	C
C-1	0.002	120	OK	α	57	19	2	11	57
C-2	0.0021	90	OK	α	56	297	3	12	50
C-3	0.0006	60	OK	α	56	190	7	9	57
C-4	0.0007	70	Crack	α	49	20	1	13	56
C-5	PRACTICE SPECIMEN: NOT SUITED FOR ANALYSIS								
C-6	0.0038	120	Crack	-	-	-	-	-	-
C-7	0.0029	70	OK	α	46	41	9	6	104
C-8	-	50	OK	-	-	-	-	-	-
C-9	0.0015	130	OK	-	57	45	15	8	197
C-10	0.0025	160	OK	α	-	-	-	-	-
C-11	-	250	OK	α	27	30	51	16	150
C-12	0.0020	125	OK	α	36	53,31	20,76	7,5	135,150(3)
C-13	0.0014	100	OK	α	-	-	-	-	-
C-14	0.0014	90	OK	α	-	-	-	-	-
C-15	0.0007	80	OK	α	-	-	-	-	-
W-1	0.0018	100	-	α	27	50	20	10	86
W-2	0.0036	100	-	α	-	-	-	-	-
W-3	W FLASH COAT ONLY		-	-	-	37	5	13	393
W-4	0.0025	190	OK	α	53	54	18	11	54
W-5	0.0025	160	OK	θ	35	20	4	8	60
W-6	0.0022	110	OK	θ	-	-	-	-	-
W-7	0.0017	170	OK	θ	-	-	-	-	-
W-8	0.0028	210	OK	α	-	-	-	-	-
A-1	UNSUITED FOR ANALYSIS								
A-2	0.0047	320	Pores	α	R_b^{94}	24	8	3	69
A-3	0.006	230	-	α	-	-	-	-	-
A-4	UNSUITED FOR ANALYSIS								
A-5	0.0085	260	Pores	α	-	-	-	-	-
A-6	0.010	230	-	α	-	-	-	-	-
A-7	UNSUITED FOR ANALYSIS								
A-8	0.0077	325	Pores	α	-	-	-	-	-
A-9	0.015	330	-	α	-	-	-	-	-
A-10	0.0085	330	Pores	α	-	-	-	-	-
C-8	AFTER THERMAL CYCLING					35	3	3	66
C-10	AFTER THERMAL CYCLING					20	1	2	56

(1) By micrometer measurement

(2) In immediate vicinity of the Al_2O_3 coating. The hardness of the as-received T-111 and also of the bulk material after coating was about $R_c 30$.

(3) Specimen C-12 was sectioned longitudinally. One portion was further sectioned transversely, and bulk chemical analyses were made on the nose portion and the cylindrical portion. The first numbers in the table are the analyses of the nose portion.

(2) W-Flash Experiments

Concern was felt early in the program that a CVD process which involved admitting air to a tantalum alloy at 2000°F would result in massive contamination of the alloy. Consequently, it was suggested that a tungsten stop-off coating, or W flash, might effectively retard this potential contamination during application of the Al_2O_3 coating. Experiments were made to investigate the validity of the premise.

T-111 specimens identical to the C series specimens were coated at AI with about 0.5 mil of tungsten by CVD. These specimens then became known as the W series. Bulk chemical analyses of one of the W flash specimens indicated that it had picked up considerable carbon of unknown origin during the W deposition. Following the W flash, seven specimens were coated with Al_2O_3 by the reference CVD process, and were analyzed in a manner similar to the examinations of the C series. These data appear in Table VIII-2.

Neither bulk chemical analyses nor microhardness measurements showed conclusively that the W flash was effective in preventing embrittling contamination.

Some of the W series specimens differed from the C series in several important ways. For example, specimens W-5, W-6 and W-7 consisted predominantly of $\Theta\text{-Al}_2\text{O}_3$, while others in the series consisted predominantly of $\alpha\text{-Al}_2\text{O}_3$. The reason for this is not known. Metallographic examinations of specimens W-4 and W-5 showed evidence of separation between the W flash and the T-111 substrate. The Al_2O_3 coatings appeared normal. Moreover, thermal cycling (as described previously) of specimen W-7 caused some of its coating to spall off.

The observations that the W flash was not clearly effective in reducing contamination, and that the W flash may have caused deleterious side effects have led to termination of work on this progress.

The spalling of the W-7 coating provided an opportunity to analyze the Al_2O_3 . The specimen was probed vigorously with a scribe, resulting in additional spalled Al_2O_3 . This was analyzed by emission spectrography, with the results shown in Table VIII-3. When the coating flaked from the substrate, it apparently took a good portion of the W flash with it. Concern is felt for the

TABLE VIII-3
 CHEMICAL ANALYSIS OF CVD Al_2O_3 WHICH
 SPALLED FROM SPECIMEN W-7

<u>Element</u>	<u>Amount, ppm</u>
Ag	3
B	1
Ba	25
Be	1
Ca	500
Cd	1
Co	10
Cr	25
Cu	3
Fe	300
Mg	10
Mn	5
Mo	100
Ni	50
Pb	5
Si	250
Ti	250
W	1000

levels of Ca, Fe, Mo, Si, and Ti. The aggregate of these is 1300 ppm, or 0.13 percent. The coating vendor is being consulted on the probable sources of these contaminants, and means will be sought to eliminate them.

(3) Arc Plasma Spraying

Although chemical vapor deposition was considered the prime candidate process for applying the LRHSC Al_2O_3 coating, a backup process was also investigated. This was arc plasma spraying (APS) of Al_2O_3 onto T-111 substrates. As described in the previous quarterly report, these experiments were performed by a coating vendor in specially constructed equipment. This was a glove box equipped with a heat exchanger and a small furnace, as shown in Figure VIII-18.

The specimens used in the APS experiments, designated the "A" series, were identical to those used in the CVD studies. They were placed in the glove box, and the system was pumped out and backfilled several times with high-purity argon. The specimens were preheated to about 1700°F, removed from the furnace and mounted in a rotating chuck, and immediately arc plasma sprayed with Al_2O_3 with a special miniature plasma gun. The feed material was Norton Company grade 183-TR Al_2O_3 .

The coated specimens were returned to AI for evaluation, and the results are shown in Table VIII-2. Specimens A-1, A-4, and A-7 had badly spalled and had excessively thick coatings. They showed evidence of rework. No tests were run on them.

The remaining "A" series specimens had light gray coatings which were not of uniform thickness. The thicknesses given in Table VIII-2 are "average" values. The coating texture was somewhat rougher than that of the CVD coating, but the texture was relatively uniform from specimen to specimen. Dye penetration testing showed the coatings to contain considerable open porosity, which was expected. X-ray diffraction traces indicated that in all of the "A" series specimens tested $\gamma\text{-Al}_2\text{O}_3$ was the predominant phase. Some

indications of $\alpha\text{-Al}_2\text{O}_3$ were usually seen, but the amount of α was generally about 20 percent. Microhardness measurements as seen in Figure VIII-19, did not show a hard layer in the T-111 immediately adjacent to the Al_2O_3 coating in the only specimen so tested. Bulk chemical analysis of the same substrate showed that contamination during processing was extremely small.

The "A" series coatings were found not to meet two of the criteria of the LRHSC Al_2O_3 coating requirement. They contained appreciable open porosity, and they consisted primarily of the metastable $\gamma\text{-Al}_2\text{O}_3$. Consequently, and in view of the apparent success of the CVD process, arc plasma spraying is no longer a candidate coating process for the LRHSC Program.

GLOVE BOX SETUP

GLOVE BOX WITH HEAT EXCHANGER

Figure VIII-18. Gove Box equipped with a heat exchanger.

Figure VIII-19. Arc Plasma Sprayed Al_2O_3 Coating Results.

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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 ($\sim 1150^{\circ}\text{F}$) 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.

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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.

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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.