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LARGE RADIOISOTOPE
HEAT SOURCE CAPSULE (LRHSC)
THIRD QUARTERLY PROGRESS REPORT
JANUARY-MARCH 1968

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN ROCKWELL CORPORATION

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FOREWORD

This is the third 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 January 1, 1968 through March 31, 1968 under Contract AT(29-2)-2338 for the U. S. Atomic Energy Commission.

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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 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) Program Management
- 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

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II. PROGRAM MANAGEMENT

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

The official milestones for the program have been expanded. A list of the milestones and the status of each are shown in Table II-1. The program is essentially on schedule and is within projected costs. Detailed plans for the remainder of GFY 1968 and for GFY 1969 have been prepared and have been approved by Sandia and AEC management.

C. ACCOMPLISHMENTS DURING THE QUARTER

The primary planning effort during the quarter has been the preparation of the detailed program plans for the last quarter of GFY 1968 and for GFY 1969. The plans, including technical scope, schedules and manpower, have been completed and approved by AI, Sandia and AEC management.

The Quality Assurance Plan for the LRHSC Program was printed and distributed. This plan details the quality assurance procedures and techniques used in the program from materials receipt through capsule assembly, testing and post test evaluation.

Two topical reports were issued during the quarter.

Topical Report # 1 - LRHSC Program Materials Specifications

Topical Report # 3 - LRHSC Program Ceramic Coating Process
Development and Evaluation

TABLE II-1
OFFICIAL MILESTONE 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	September 1967	Complete
4. Complete and release fixture drawings	September 1967	Complete
5. Complete Al ₂ O ₃ CVD vs Plasma Coating Evaluations	October 1967	Complete
6. Complete all assembly specifications	November 1967	Complete
7. Complete all processing and test	January 1968	Complete
8. Complete Topical Report # 1, LRHSC Program Material Specifications	January 1968	Complete
9. Initiate short term creep tests	January 1968	Complete (March 1968)
10. Complete coupon (except coupon stacks associated with creep capsules) tests	March 1968	Complete
11. Complete Topical Report # 2, LRHSC Program Process Specifications	March 1968	April 1968
12. Complete Topical Report # 3, LRHSC Program Ceramic Coating Process Development and Evaluations	April 1968	Complete
13. Complete Topical Report # 4, LRHSC Program Capsule Fabrication Processes	May 1968	OK
14. Complete GFY 1968 Capsule Fabrication and Assembly	June 1968	OK

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

Analyses have been accomplished in support of capsule design, assembly and test design. The verification of design approach and definition of design margin activities will not be initiated until substantial test data are available. Until that time, the first quarter of GFY 1969, no specific efforts are planned under this task.

C. ACCOMPLISHMENTS DURING THE QUARTER

No activity under this task during the quarter.

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IV. CAPSULE DESIGN

A. TASK OBJECTIVES

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

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

B. SUMMARY OF TASK STATUS

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

No activity under this task during the quarter.

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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. Studies of alternate materials fabrication and assembly techniques will be performed as required.

B. SUMMARY OF TASK STATUS

A brazing procedure has been developed to join the Ta-10W pressure feed-through tube from the LRHS creep capsule to a stainless steel pressure tube. The parameters used have been refined and are being published as a specification

A study of the heat treating response of the T-111 structural shell was performed. The best heat treating cycle was identified as 2700°F for one hour after the sixth stage draw of the eight stage process.

Welding development at Electron Beam Welding Inc. has been completed. The required 100% penetration welds of the feed-through tube and the structural shell have been made and the parameters have been defined. The AI electron beam welder has been installed, checked out and has passed acceptance testing. Weld development at AI using the EBW parameters and the new 6 Kw Hamilton Standard Welder have been completed for the structural shell fabrication. The resulting welds have been examined by the use of ultrasonic and metallographic techniques and found to meet the requirements.

The High Resolution Ultrasonic Test System and the Infrared Test Equipment for LRHSC inspection and quality assurance, have been installed and checked out. Attempts to radiograph test welds in the structural shell failed to provide the desired sensitivity. The results showed that ultrasonic testing would best reveal the nature of any defects in these joints. Inspection techniques for examining the following capsule joints were developed:

- 1) The structural shell material.
- 2) The structural shell/end cap weld.
- 3) The T-111 end cap/Ta-10W pressure feed-through weld.
- 4) The Ta-10W feed-through/stainless steel braze joint.

C. ACCOMPLISHMENTS DURING THE QUARTER

1. Vacuum Brazing

During the past quarter, a technique was developed for brazing the Ta-10W capsule lead tube to a high pressure stainless steel tube. The technology and process parameters were evaluated in the LRHSC vacuum brazing rig shown in Figure V-1. The brazing alloy used was Au-30% Cu which melts at 1750°F. The remelt temperature of the braze appears to be some 150°F higher.

Tests in the creep rig indicate that the braze joint must be moved away from the upper furnace shields in order to keep the temperature of the joint to a safe operating level. When the center of the furnace is 2000°F, the temperature atop the furnace is approximately 1800°F. An inch above and away from the center the temperature was down to 800°F. The joint design selected for the creep capsules is shown in Figure V-2.

The design utilizes standard 1/4" OD high pressure stainless steel tubing compatible with available tube fittings. The nose of one end is pointed to 0.210" OD by swaging and then machined to match the ID of the Ta-10W tubing. The nose of the stainless steel tube is then wrapped with 0.002" thick braze foil and inserted into the 0.215" ID Ta-10W pressure tube. The high thermal expansion of the steel assures that the braze gap will be minimal at the braze temperature.

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Figure V-1. 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.

During the development stages of the brazing process, 12-inch lengths of stainless steel were swaged, machined to the specified dimensions, electro-etched and electroplated with nickel. The nickel was required to reduce oxidation of the stainless steel and enhance braze wetting. After wrapping the stainless tube with a one inch length by one circumference wide sheet of 0.002 inch thick braze alloy, the tube was fitted into a six inch length of Ta-10W capsule pressure tube. The assembly was then instrumented with a chromel-alumel thermocouple and centered in the brazing facility by the use of locator "spiders."

Brazing was accomplished after pumping the vacuum chamber down to about 5×10^{-5} torr. The 10 KC, 15 KW motor generator induction unit was used to bring the temperature to about 1830°F in a 10 minute period. This temperature represents an approximate 100°F superheat and was held for one minute. At that time the power was turned off and the piece was allowed to radiation cool.

Figure V-2. LRHSC Pressure Feed-Through Braze Joint Design

Evaluation of the braze joint consisted of a series of proof tests. The first test is helium leak checking. If the joint was impervious to helium by the standard spectrometric inspection, it was then sent to the nondestructive testing laboratory.

Prior to pressure testing and sectioning for metallographic examination, the joint was ultrasonically examined for braze distribution. The part was water coupled to a 15 MHz focused lithium sulfate transducer as shown in Figure V-3. The focal point, approximately 0.060" diameter was placed just above the braze joint. The instrument was then adjusted for the maximum signal response from the bond interface.

The signal was gated electronically and fed into a facsimile recorder. When the signal increased to above 30% of instrumentation saturation, the recorder would not print. The resulting "C scans" reveal the degree of bonding between the two tubes.

After a number of unsuccessful attempts (as judged by ultrasonic "C scans") due to either loose fits or tube eccentricity, some modifications were added to the procedure. First the gap between tubes was reduced from 5 mils to 3 mils and then the ID of the Ta-10W tube was reamed to a fixed dimension. These attempts met with sufficient success to warrant pressure testing and subsequent metallographic analysis. The pressure testing was done at 1000°F and 11,000 psi. After this the braze sample was sectioned, mounted and polished for metallographic inspection. A view of an acceptable braze joint section is shown in Figure V-4.

The braze joints for the first two creep capsules successfully passed all of the required leak, ultrasonic and pressure testing. The capsule was accommodated in the rig by the use of a second mounting "spider."

Figure V-3. Schematic representation of transducer and sound beam to part relationship for ultrasonic examination of the braze joint

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10X

Figure V-4. End View of Braze Joint

2. T-111 Deep Drawing and Heat Treating

A number of problems have occurred during the development of the process to fabricate the T-111 structural shell. The phenomenological aspects of the problems are detailed under Task 5. This section deals with the analytical metallurgy associated with solution of these problems.

a. Tensile Rupture of the Blank

After carefully analyzing those parts which failed by punching out the ends of the drawing blank, in a number of different drawing stages, it was concluded that the excessive loads required to deep draw the blank were associated in all cases with a breakdown of the lubricant and contact of the blank with the hardened steel die. This breakdown caused a cold welding of the part to the

die resulting in superficial tearing of both the die and blank. This phenomena (a common scourge of draw shops) is observed often in the early development of new drawing processes and is commonly referred to as galling. One approach to the solution of this problem is the selection of a lubricant which will withstand the high compressive stresses required by the process.

Initial experiments indicated that the lubricant search could require a considerable period of time. Some of the high compressive stress lubricants investigated included stearates, STP (a commercial extreme pressure lubricant, one of the better intermediate temperature films) and additions of these with solid lubricants such as hexagonal carbon and molybdenum disulfide mechanically mixed with the basic carriers. Each of the experiments indicated that while in the end the perfect lubricant could be mixed, a better, more positive solution would be to utilize a softer die material. This concept is utilized in most industrial wear applications.

Aluminum bronze was selected as a die insert material. To be on the safe side, the size of the reduction was reduced by the introduction of intermediate dies when the blank and, therefore the load on the part, was large. This solution entirely eliminated any tearing and galling problems.

b. Crazing of the Blank Surface

After solution of the galling/end punch out problem, the drawing process appeared in control. Four blanks were committed for tooling checkout. After drawing the blanks through the 0, 1, 1a, 2, 2a, and 3 (refer to Section VI) dies, the parts were heat treated (1 hr. at 2700°F) to recover the alloy's fabricability. (It is noted that this alloy had previously never been shaped to such an extreme blind L/D as through the No. 3 die). Upon subsequent reduction, all four parts exhibited multiple surface cracks which in the glass industry would be termed crazing. This condition is shown in Figure V-5. Examination of the blanks (now 3" dia. x 5" long) indicated that the cracking occurred whenever the bronze dies had been in contact with the alloy prior to the heat treatment. The die wash

(Approx. 8X)

Figure V-5. Multiple Cracks on the surface of the T-111 Shell drawn through bronze dies, heat treated and redrawn. Cracks appeared on the redraw

apparently had not been adequately cleansed from the part surface prior to heat treatment. If it alloyed with the part during the heat treatment and embrittled its surface, the die wash could be the source of the crazing. To test this hypothesis, the next blank was cleansed in caustic followed by a nitric acid soak between each reduction. This procedure was designed to remove all of the bronze die wash but not require special handling facilities nor technicians at a vendor's house.

Results of a test of a blank going through this process proved conclusively that this procedure (even in untrained hands) unequivocally eliminated the crazing problem.

c. Gathering and Nose Deformation

The part used to prove out the die wash postulate exhibited two additional phenomena either of which would render the part unacceptable for creep testing.

First a series of discontinuities (three or four observed) in the open end of the cup and two cracks at the nose were observed. Upon examination of the part the nose cracks apparently were associated with the deformation line of the No. 4 die, the first after the heat treatment (1 hr. at 2700°F).

The discontinuities near the mouth of the shell were machined out with a single point tool. The maximum indicated depth was 0.050" of the 0.215" gathered wall which left the wall very close to the initial thickness of the plate blank (0.160"). It would appear that these defects were a direct wall gathering defect - one which is very common in the early stages of developing a deep drawing process. Upon detailed examination of the fabricators process, it was noted that the concentricity fixture (described in Section VI) was compressively loading the blank during the ironing operations. While no detailed examination of this practice has been performed, parts have since been drawn through the exact process which produced the gathering defects but without the load; none show evidence of the defects.

d. Nose Cracks

The surface of the part which suffered nose cracks was examined. There was no previous evidence as to why this phenomena should occur. Since the part cracked on the first draw after the heat treatment, it (1 hr. at 2700°F) was suspect. In any of the early stages of drawing (up to die No. 3) the side walls of the cup received severe deformation while the nose or blind end received virtually none. It was expected that different areas of the part would react differently to heat treatment due to this work variation. A very critical area was at the radius of the nose of the part which had received a small amount of deformation prior to the heat treatment. This is the area that is first deformed

after the recrystallization treatment. If there were a variation in recrystallization, grain size or precipitation across the wall or around the radius, this poor response to the heat treatment could well render the part difficult to fabricate further.

A scrap part which had been previously drawn through dies 1, 2, and 3 was sectioned for a heat treating study. While the fabrication sequence of this part was not absolutely identical with the present process, it was an available part sufficiently close to warrant evaluation. To bracket the 1 hr. at 2700°F, samples were treated for 1 hr. at 2500, 2600, 2700, and 3000°F.

The as-fabricated part showed conclusively that there was very little work in the blind end of the cup as shown in Figure V-6. The grains are relatively equiaxed and uniform across the wall in the flat nose region, but into the radius there is a transition across the wall evident in the same figure. The structure on the outer surface is relatively highly worked as evidenced by the elongated grains, with a transition to the relatively (not completely) equiaxed grains at the inner region of the cup. In the side wall there is a relatively uniform region of deformed grains, completely across the wall. The continuous nature of the grain boundaries indicates that the part could possibly be deformed lightly without rupture. An example of the structure further deformed is shown in Figure V-7, typical of the side wall of a part fabricated to the finished shape (through die No. 5).

The structure of the heat treating study part (dies 1, 2, and 3) after 1 hr. at 2500°F is shown in Figure V-8. Except for partial recrystallization in some of the higher worked regions, there appears to be very little change from the as-fabricated condition. While recovery has occurred, sufficient recrystallization for further fabrication has not.

The same lack of recrystallization is true for the 1 hr. at 2600°F. There is still not enough recrystallization for the sizable deformation during the ironing that occurs in the No. 4 and 5 dies.

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GRAIN STRUCTURE THROUGH WALL OF T-111 CUP AFTER SIX STAGES
OF DEEP DRAWING AS FABRICATED PRIOR TO ANNEAL

Figure V-6.

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(Approx. 100X)

Figure V-7. Microstructure of T-111 shell after
drawing through die number 5

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GRAIN STRUCTURE THROUGH WALL OF T-111 CUP AFTER SIX STAGES
OF DEEP DRAWING WITH ANNEAL AT 2500°F FOR ONE HOUR

Figure V-8.

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The sample heated for 1 hr. at 3000°F is shown in Figure V-9 and exhibits what could be considered a poor response to the heat treatment. On the flat section of the blind dome the grains are completely equiaxed and there is some evidence of partial grain growth in the central regions of the plane. In the wall of the cup complete recrystallization has occurred and some partial grain growth. While in this region there is evidence of preferred growth in bands, this does not occur further up the wall. In the radius there has been complete recrystallization and major grain growth on the outer surface.

The location of the section in the figure was selected to show the transition across the wall. In the mid radius, the grain growth is complete across the wall, and the transition to the smaller grains in the side wall is not nearly as steep as in this section. The location of the section shown with the transition is approximately where the next die will deform first. This type of transition could well render the part incapable of further fabrication without cracking.

The 1 hr. at 2700°F sample is shown in Figure V-10. There is complete recrystallization throughout the part and the grains are relatively homogeneous throughout. There is evidence of grain growth in the radius, however, the variation cannot be considered severe.

On the basis of this study it is concluded that 1 hr. at 2700°F is the optimum heat treatment for the part and other causes for the nose cracks will have to be sought. Since this study was completed, four cups have been drawn without evidence of the nose crack. The source of the crack could well have been an inhomogeneity in the starting blank.

3. Welding Development

The 6 Kw electron beam welder which was built to specifications based on the LRHSC welding requirements, underwent acceptance testing during this reporting period. The acceptance test results are reported in Table V-1.

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GRAIN STRUCTURE THROUGH WALL OF T-111 CUP AFTER SIX STAGES
OF DEEP DRAWING WITH ANNEAL AT 3000°F FOR ONE HOUR

Figure V-9.

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Figure V-10.

AI-AEC-12676

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TABLE V-1
FINAL ACCEPTANCE TEST RESULTS

<u>Test</u>	<u>Specification</u>	<u>Results</u>
Ultimate Vacuum	1×10^{-6} torr, 45 min.	1.0×10^{-6} torr, 60 min.
Weld Penetration variation	$\pm 5\%$	$\pm 5\%$
Maximum Allowable Pressure during welding	5×10^{-6} torr	1.5×10^{-5} torr

The vacuum system discrepancies were judged to be acceptable for the LRHSC Welding Program. The machine was accepted on February 14, 1968.

During installation and acceptance testing of the AI electron beam welder, the development of procedures at Electron Beam Welding Inc. (EBW) was completed using equivalent equipment. The results achieved at EBW were satisfactory. Both Weld Procedure 1238 (T-111 Structural Shell), and Weld Procedure 1242 (Ta-10W/T-111 Pressure-Electrical Lead Tube) provided joints which met the quality requirements of 100% penetration, 90% sound, defect free weld metal and no cracks or lack of fusion. The work at EBW had been performed with short tubular sections for each material diameter and thickness to simulate the joint design of the actual hardware.

Weld Procedure 1238 is the structural shell weld. This joint consists of a 1.67 OD x 0.127 inch wall T-111 shell with a 0.020 inch Ta-10W liner beneath the T-111 weld. EBW refined their parameters to a point where the largest variable was the fitup between the Ta-10W liner and the T-111 structural shell. The variable fitup caused non-reproducible results when attempting to eliminate excessive weld penetration into the Ta-10W liner. Satisfactory results were not achieved with the quantity of material available.

Since the first test capsules to be fabricated included the structural shell without the Ta-10W liner, EBW was directed to develop parameters for this joint configuration with the remaining material. Satisfactory results for this weld were achieved. The underbead was uniform with complete penetration. Metallographic examination showed no significant internal weld defects.

Weld Procedure 1242 deals with the T-111 end cap to the Ta-10W pressure feed-through tube joint. As reported previously, the welding parameters used provided welded joints that were considered to be satisfactory for the required test purposes.

The final parameters developed at EBW are shown below in Table V-2.

TABLE V-2
FINAL WELD PARAMETERS
(EBW)

<u>Weld Procedure Number</u>	<u>Current (ma)</u>	<u>Voltage (KV)</u>	<u>Weld Speed (RPM)</u>	<u>Beam Diameter (inches)</u>
*1238	38	150	12	.040
*1238 cosmetic	38	150	12	.100
1242	10	120	25	.017
1242 cosmetic	10	120	50	.025

* Without liner beneath joint

The parameters reported for the weld procedures did not include the many sensitive parameters that required refinement because of the different EB welding machines, as well as the configuration and metallurgical characteristics of the final hardware. The work at EBW demonstrated that variations in fitup between the shell and liner (WP 1238) resulted in non-reproducible weld penetration. This situation must be corrected by adjusting dimensional tolerances for final hardware.

Material for the continued development of WP 1238 and WP 1242 at AI was received and prepared for welding. The weld parameters used at EBW were evaluated at AI using the 6 Kw electron beam welder. Using the parameters established at EBW for WP 1242 only 60 percent penetration was achieved in the weld joint. The penetration was increased by increasing the beam voltage and decreasing the beam diameter and weld speed. The welds were made by electron beam tacking a Ta-10W tube to a T-111 tube and then welding the interface. The resulting welds had a smooth surface with minor concavity, full penetration and a uniform weld drop-through on the inside diameter of the tube.

The weld required for the capsule hardware is a butt weld involving a Ta-10W tube welded to a boss on a T-111 hemispherical end cap. The end caps were received with the boss projecting about 0.030 inch, approximately 0.100 inch shorter than required. The boss height was limited by the thickness of the drawing blank. There was a question as to whether or not the weld could be satisfactorily made with these parts. An alternate method was used to achieve hardware with the final required boss height. This method involved building up the boss area with T-111 weld metal and subsequent machining to the drawing requirements. Welds using both types of hardware were attempted. A hemispherical end cap with a short boss was overlayed with T-111 filler wire by the TIG process. This part was then machined to the drawing dimensions except for the wall thickness which was 0.010 inch thinner than required because of a machining error. The assembly was then electron beam welded to a Ta-10W lead tube.

The end cap with the short boss was welded directly to a lead tube. The parameters previously developed were not satisfactory for this approach, because of the mass imbalance between components. As a result, additional passes were made to locate parameters which would provide full penetration. A second attempt with this type joint also resulted in an unsatisfactory weld

due to severe undercutting. It was apparent that this type weld is extremely sensitive to minor variations in the joint.

The trial electron beam welds with both the built-up boss and short boss proved to be metallurgically sound during metallographic examination. However, building up the boss was more satisfactory from the standpoint of ease of welding as well as being less sensitive to weld cycle parameter variations. Therefore, this method was selected for the "B" capsule fabrication effort.

Test T-111 end caps were built up with T-111 filler wire and machined to the configuration of the required capsule hardware. Trial welds were made joining the end cap to the Ta-10W lead-through tube. The parameters developed produced welds with a smooth uniform surface and minimal diametral buildup. Metallographic examination demonstrated that the desired weld quality of complete penetration with 90% T sound, defect-free weld metal with no lack of fusion was met. Consequently, the procedure was accepted for the "B" capsule fabrication.

The T-111 structural shell test welds (WP 1238) with actual components were made using the parameters developed at EBW. Full penetration was not achieved with these parameters. This condition was corrected by decreasing the travel speed of the work piece and keeping all other variables constant. However, difficulty in reproducing the weld was encountered. Defective transformers in the electron beam welder high voltage system were causing a variation in output voltage which resulted in an unregulated weld cycle. The transformers were replaced and the process was then found to be satisfactory.

Metallographic examination of the T-111 structural shell test welds demonstrated that the internal weld quality requirements of complete penetration with 90% T sound, defect-free weld metal with no lack of fusion were met. The weld surface and underbead were smooth and uniform and the drop-through was about 0.030 inch.

The procedure established for the T-111 structural shell-to-end cap weld was accepted for fabrication of the "B" type creep capsules. The final weld parameters are shown below in Table V-3.

TABLE V-3
FINAL WELD PARAMETERS (AI)

<u>Weld Procedure Number</u>	<u>Current (ma)</u>	<u>Voltage (KV)</u>	<u>Weld Speed (RPM)</u>	<u>Focus Current (dial setting)</u>
1242	10	130	12	326
1242 cosmetic	10	130	12	329
*1238	40	148	8	385
*1238 cosmetic	40	148	20	400

*without liner beneath joint; applicable only to "B" type creep capsules.

Each of the weld procedures consists of a two pass penetration weld with an additional smoothing pass.

Both the lead tube-to-end cap (WP 1242) and the structural shell-to-end cap (WP 1238) welds were made for the first two "B" type creep capsules. Non-destructive examination of the capsule welds showed no indication of flaws. One capsule is currently on test.

4. Non-destructive Test Development

During the quarter reduced funding resulted in termination of all non-destructive test development except as required to evaluate the structural shell capsules prior to creep testing.

The infrared test equipment for coating adherence testing was received and its operation checked out. Suitable size coated samples were not available for test development.

The scanning fixture for the high resolution ultrasonic test system developed some electromechanical problems. These problems were traced to a defective drive motor. The material used to waterproof the motor had contaminated the armature causing the brushes to stick. The drive motor was replaced by the supplier and a modification of the fixture was made to allow mounting the motor above water level thus eliminating the need to waterproof the motor. The scanning fixture was returned to AI and is now operating properly.

Attempts to use radiographic inspection to evaluate the quality of the weld test samples for the structural shell weld (T-111 alloy) were unsuccessful. The 300 KV industrial X-ray unit and the 3.5 currie Co^{60} source were both used for this evaluation. X-ray energies obtained at 250 KV, 10 MA for a six minute exposure were not sufficient to effectively penetrate the sample. Radiographs obtained using the Co^{60} source for a 15 minute exposure were of poor quality. The sensitivity of this radiograph was estimated at best to be $\sim 7\%$. Therefore, efforts to develop an ultrasonic test for this weld and for the structural shell material were resumed.

A test standard for ultrasonic inspection of the structural shell was prepared. Simulated crack type flaws were introduced into a T-111 shell with crazed surface by electrical discharge machining notches on the shell ID. These notches were placed in both longitudinal and transverse directions and were $\sim 1/4"$ in length and 0.005" and 0.010" in depth. These notches were easily detected using an ultrasonic shear wave technique. The C-scan recordings, Figures V-11 and V-12, show the good detail and sensitivity obtained by this test method. These tests were performed at maximum sensitivity of equipment.

Preliminary test techniques were developed to ultrasonically inspect the structural shell material, the structural shell-to-cap EB weld, the T-111 alloy-to-Ta/10W alloy feed-through tube EB weld, and the Ta-10W feed-through tube-to-stainless steel extension tube braze joint.

Figure V-11. Ultrasonic C-scan recording of ultrasonic test. Standard showing indications from longitudinal notches on ID of shell and from natural flaws on O.D. "A" 0.005" deep x 1/4" long notch on ID, "B" 0.010" deep x 1/4" long notch on ID, "C" numerous cracks (0.010" deep x 0.060" long) on OD.

Figure V-12. Ultrasonic C-scan recording of ultrasonic test Standard showing indications from transverse notches on inside surface of shell. Notch "A", 0.005" deep x 1/4" long
Notch "B", 0.010 deep x 1/4" long

Ultrasonic shear wave tests performed on two structural shell capsules B1a and B2a, prior to creep testing, showed no flaws in the shell material or in the shell-to-cap weld. During testing of the structural shell material, test sensitivity was set to give a saturation signal from the artificial flaws (notches approximately 0.005" and 0.010" deep x 1/4" long) on the inside surface of the structural shell ultrasonic test standard. Also detected at this sensitivity setting were the natural flaws (cracks approximately 0.010" deep x 0.060" long) present on the outside surface of the test standard. Figure V-11 is an ultrasonic C-scan recording showing both artificial and natural flaws in the test standard. Figures V-13 and V-14 are C-scan recordings of the structural shell material for creep test capsules B1a and B2a. The sensitivity of these tests, Figures V-13 and V-14, is identical to the test sensitivity used for the test standard (Figure V-11). The numerous small indications shown in Figure V-14 were caused by superficial scratches on the OD of capsule B2a.

C-scan recording of the shell-to-cap weld for creep test capsules B1a and B2a are shown in Figure V-15, along with a C-scan recording of the transverse notches in the test standard. These recordings show what appears to be weld spatter and weld drop through on inside surface of the structural shell. The exact nature of these indications could not be determined. Therefore, a more representative ultrasonic test standard for the shell-to-cap weld is being prepared from an actual EB weld in the structural shell material. Such a test standard is necessary to provide easier interpretation of the ultrasonic test results.

An ultrasonic shear wave test performed on a Ta-10W-to-T-111 alloy feed-through tube weld showed no indications of flaws. Since a test standard was not available for this weld, the test was set up using the test standard for the structural shell. With this setup, sensitivity of the test was estimated to be capable of detecting flaws (incomplete penetration and lack of fusion) which exceed 10% of the tubing wall thickness.

Figure V-13. Ultrasonic C-scan recording of structural shell for creep test capsule B-1a. Indication "A" along end of shell was caused by air bubbles adhering to rubber stopper use to keep water from entering shell

Figure V-14. Ultrasonic C-scan recording showing numerous small indications on structural shell of creep test capsule B-2a. Indications were caused by superficial surface scratches. Indications "A" and "B" are attributed to surface roughness on spherical ends of the capsule and on the cap-to-shell weld respectively

Test Standard

Figure V-15. Ultrasonic C-scan recordings of transverse notches "A" and "B" in test standard and cap-to-shell EB weld of creep test capsules B-1a and B-2a. Indications noted are: "A", 0.005" deep x 1/4" long notch on ID of shell; "B", 0.010" deep x 1/4" long notch on ID of shell; "C", weld spatter; "D", weld drop through; "E", surface roughness of weld

An ultrasonic compressional wave test was developed to evaluate the quality of the braze joint between the Ta-10W feed-through tube and the stainless steel extension tube. This test involves monitoring the ultrasonic signal reflected from the inside surface of the Ta-10W feed-through tube. The absence of this signal indicates a good braze. Very good correlation was obtained between the ultrasonic test results and metallographic examination of several sample braze joints. This test was applied to the brazed joint of creep capsule Bla and a large unbond area was detected. This area extended for 90% of the joint's length and 20% of its circumference. This joint therefore, was rebrazed before preceding with the creep testing, however test scheduling did not allow for an ultrasonic inspection of the rebrazed joint. The rebrazed joint was subjected to and passed a 10,000 psi pressure test prior to creep testing.

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VI. FABRICATION AND ASSEMBLY

A. TASK OBJECTIVES

The objectives of this task are to obtain all tantalum and platinum alloy semistock required for the LRHSC Development Program, to fabricate all capsule and coupon piece parts, to develop, fabricate, and check out all capsule piece part and assembly tooling, and to assemble all capsules and compatibility coupon stacks.

B. SUMMARY OF TASK STATUS

All tantalum and platinum alloy material required for the LRHSC Development Program was received from National Research Corporation (tantalum alloys) and Bishop Industries (platinum alloys) during the reporting period. Delivery and receipt of the tantalum alloys was divided into four separate shipments so that piece part fabrication development, weld development, prototype fabrication, and test capsule fabrication could proceed on schedule.

Development of the T-111 structural shell deep drawing processes was completed and the tooling certified with the successful fabrication of three structural shells.

The tooling for the forming of T-111 structural end caps with the feed-through boss for attachment of the Ta-10W pressure feed-through tube was developed during the reporting period. Structural end caps were formed for use on "Creep B" capsules and for weld development of the Ta-10W tube-to-cap electron beam (EB) weld and for the end cap to structural shell EB weld.

Tooling for the Ta-10W inner liner flanged cap was fabricated and is being checked out using copper and Ta-10W blanks. The Ta-10W blanks are being used to certify the tooling.

Fabrication of all weld and assembly tooling was completed, inspected, and accepted. This tooling is presently being checked out in the assembly area by LRHSC assembly personnel.

Creep capsules B1a and B2a were fabricated, assembled, inspected and delivered to creep testing during the third month of the reporting period.

The remaining compatibility coupon stacks required for GFY 1968 testing were fabricated, assembled, and delivered to test.

C. ACCOMPLISHMENTS DURING THE QUARTER

1. Material Delivery (Tantalum and Platinum Alloys)

All tantalum and platinum alloys have been received. Material certifications, chemical analysis reports (except for AI analysis of the platinum alloys), fabrication and NDT reports (both supplier and AI) have been completed, recorded, and are on file in the LRHSC In-Process Control Center Configuration Files. This semi-stock fabrication data has been separated into Creep A, B, and C, Earth Burial C, and Impact A and B capsule configurations and will become part of the "as built" data presented with each capsule when it is delivered to test.

A typical material certification report on the material received for the LRHSC program is presented in Figures VI-1, -2, -3, and -4. Figure VI-1 is the material certification from National Research Corporation on heat analysis, finish chemistry, and mechanical properties on the T-111 alloy being used. Figure VI-2 and Figure VI-3 are the ultrasonic report and C-scan on this same T-111 alloy while Figure VI-4 is the mechanical testing certification on tests conducted for AI on this T-111 alloy after receipt of the material.

2. Capsule Piece Part Fabrication

As reported last quarter, the tooling for deep drawing of the T-111 structural shell had been completed and two copper parts had been drawn

METALS DIVISION

NATIONAL RESEARCH CORPORATION

A SUBSIDIARY OF NORTON COMPANY

45 Industrial Place, Newton, Massachusetts • 02164

T-111

MATERIAL CERTIFICATION

CUSTOMER: Atomics International
PRODUCT: NRC Tantalum Alloy T-111 Sheet
(7) .125" THK. x 2" x 2"

CUSTOMER NO: N 7261613
NRC NO: 53434 Rev. #4

WEIGHT: 5.0 Lbs. 16 Pcs.

HEAT NO: 8102

CERTIFIED HEAT ANALYSIS IN PPM

O	H	N	C	Co	Cr	Fe	Mo	Nb	W	Hf	Zr
26	2	35	32	10	<1	5	400	200	7.98%	2.02%	400

FINISH CHEMISTRY

O	H	N	C	Fe	Mo	Nb	Ni	W	Hf	Zr	
21	1	43	43	5	25	250	<1	7.69%	1.78%	400-----	SAMPLE A
14	1	30	30	5	25	250	<1	7.89%	1.85%	400-----	SAMPLE B

MECHANICAL PROPERTIES

	UTS	2% YIELD	ELONG.	VHN-TENSILE
SAMPLE A	97,100 Lbs.	79,000 Lbs.	33%	255-257.5
SAMPLE B	96,200 Lbs.	78,500 Lbs.	29%	250.5-262.5
SAMPLE C	99,400 Lbs.	81,600 Lbs.	21%	261-261.5

VHN-TRAVERSE- Hi-272, Lo-260, Ave-266, Var-12

SAMPLE A--GRAIN SIZE: ASTM#6, SAMPLE B--GRAIN SIZE: ASTM#6-- Both 100% Recrystall.

100% Ultrasonic Tested By Automation Industries-- No Detectable Flaws

100% Fluorescent Penetrant Inspected-- No Detectable Flaws

DATE: FEB. 2, 1968

SIGNED:
L.F. MOOLIC
QUALITY CONTROL SUPERVISOR

Cable: NARMET Telephone 617 969-7690

Figure VI-1

AI-AEC-12676

FEB 12 1968

Corrected T.R. Rec'd 3/5/68
K.W.

Date 1-22-68

☐ 4000 1st St., Richmond, Calif. 94801 Ph. 236-9668
☒ 4000 Lockbourne Pl., Columbus, Ohio 43207 Ph. 491-3000
☐ Shelter Rock Rd., Danbury, Conn. 06813 Ph. 748-3581
☐ 1106 Providence Rd., Media, Pa. 19063 Ph. 565-2280

☐ EAST OIL, A Co., Room 100, 6106 14th, Dallas, Tex. 6/1/44
☐ 6561 North Ave., Santa Fe, N.M., Wash. 2-100-19, 11/13/44
☐ 6106 Ruskon St., Houston, Texas 11036 14th, 11/26/44
☐

Customer National Research Corp. Cust. P. O. No. M 27430 Item 7
Attn. Mr. Goad Job Order No. _____
Street Address _____
City & State Newton, Massachusetts ☐ PENETRANT ☐ MAGNETIC PARTICLE
Material Plates T-111 Heat & 102 S/N 1 thru 16 ☐ ULTRASONIC ☐ EDDY CURRENT
.125" x 2" x 2" Accpt. Std. ☐
Spec. ASTM-E214, 4/64" P.B.H. & All procedure 67-UT-234
Test Equip. UM 715 Reflectoscope 10N Tech. Data scanned & "C" scan paper recorder
P/R 3/8" diameter lithium sulphate spherically focused 1SMHz transducer, Axfex
Set up Data calibrate to record loss of black reflection as shown from 4/64" dia. flat bottom
holes in test standard

TEST RESULTS

NO REPORTABLE INDICATIONS DETECTED

ACCEPTED 16 PIECES S/N 1 thru 16

[illegible]

Figure VI-2

AI-AEC-12676

Item 1, P.O. 7261613
Heat # 8048

C-7312 17 November 1967
VM 715/10N P-R
3/8" Dia. x 15 MHZ
Focused Lithium
Sulphate Transducer
4.4 x 1 sensitivity,
water path - 7/8"

STANDARD

AI-AEC-12676
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Figure VI-3

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METALLURGICAL SERVICE LABORATORIES

division of
TESTING ENGINEERS, INC.

11008 S. NORWALK BLVD. SANTA FE SPRINGS, CALIF. 90670
(213) 723-9541 & 941-3291

LOS ANGELES
SAN DIEGO
OAKLAND
SAN JOSE

TO: ATOMICS INTERNATIONAL
Attn: Mr. Markowitz

DATE: 2-21-68

P.O. No. N711 0037

P/S ~~XXXXXXXX~~ CE68-23206

Lab. No. 17346

Specification: ST0 170NB0051
Rev. 'B'

TENSILE PROPERTIES								
IDENTIFICATION	DIMENSION IN.	YIELD LOAD-LBS	YIELD STRENGTH PSI	ULTIMATE LOAD-LBS	ULTIMATE STRENGTH-PSI	% ELONG IN	REDUCED DIMENSION	R A PERCENT
ROOM TEMPERATURE TENSILE TEST 2-1/8 x 2-1/8 x .125 TIII Alloy Heat #102 Request #1995	.259 .127	2,620	79,700	3,220	97,900	31		
MIN. REQUIREMENTS			65,000		80,000	20		
MAX. REQUIREMENTS			85,000		110,000			
REDUCED TEMPERATURE BEND TEST AT -320° F 105° (D-3T) Satisfactory								
MINIMUM REQUIREMENTS								
MAXIMUM REQUIREMENTS								

YIELD STRENGTH BY EXTENSOMETER AT .2% IN 1"

STRAIN RATE: .002 IN/IN/MIN TO YIELD AND

THEN: .02 in/min

TO FAILURE.

TEST MACHINE CALIBRATED: 12-13-67

WITH NBS PROVING RINGS: 1170, L1375

MATERIAL: Ta-W alloy

HEAT TREAT:

REMARKS:

MEETS SPECIFICATION REQUIREMENTS

METALLURGICAL SERVICE LABORATORIES

T. J. Borchardt
T. J. Borchardt, Mgr. Met. Sect.

Figure VI-4

AI-AEC-12676

successfully to check out the tooling and to determine the blank size required to produce a shell of sufficient length. Drawing of two blanks was attempted.

During the reporting quarter, continuation of the development of this critical fabrication process resulted in obtaining the first successful deep drawn T-111 structural shell on blank S/N 014. However, tool designs and fabrication techniques were modified extensively to produce this first successful shell. Table VI-1 tabulates the fabrication history of this development process. The detail development of this process is presented as follows:

S/N 001 (Reported Last Quarter)

This 10" diameter blank was drawn through the first stage and stalled the press during the second draw. This part was then heat treated at 2700°F for 1 hour at a pressure of 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 stages without further annealing. The part cracked on the fourth draw.

S/N 002 (Reported Last Quarter)

This 8" diameter blank was drawn through the first three stages without annealing. It was then annealed for 1 hour at 2700°F in vacuum and was subsequently drawn through the fourth and fifth (final) stages. Examination of the part revealed superficial galling.

S/N 003 and 004

Two 5-3/4" diameter blanks were drawn through the first stage die successfully. Both parts failed during the second draw. S/N 003 cocked in the drawing process while S/N 004 failed at the end when the part locked in the draw ring. Figure VI-5 shows S/N 003 after drawing through the second die. Figure VI-6 shows S/N 004 which pulled apart at the open end during the second draw.

S/N	Initial Blank Size	Lubricant Used	Die Material	Concentricity Fixture Used	Dies Used										Annealed After X Draw	Anneal Temp.	Anneal Time	Results Obtained (Insert Stage Number)										Surface Checking	Bottom Punched Out	Other (Explain)	Press Loading (Tons)								Part Utilization	Remarks
					0	1	1a	2	2a	3	4	5	Press Stalled	Axial Cracks at Top				Axial Cracks at Bottom	Circum. Cracks at Top	Circum. Cracks at Bottom	0	1	1a	2	2a	3	4				5									
001	10"	Paste	4130 Steel	No	X		X		X				None	None	None													Part pulled apart near top during 4										Develop.		
002	8-1/2"	Paste	4130	No	X		X		X	X	X	None	None	None	1, 2, 3, 4, 5	1, 2, 3, 4, 5	3, 4, 5																				Develop.			
003	5-3/4"	Paste	4130	No	X		X					None	None	None			2						2			2		Part cocked during 2 draw	115		120							Develop.		
004	5-3/4"	Paste	4130	No	X		X					None	None	None									2				Pulled apart during 2 draw	115		120							Develop.			
005	5-3/4"	STP	4130	No	X		X		X			2	2700°F	1 Hr.	3										3				115		120		110				Develop.			
006	5-3/4"	Graphite & STP	4130	No	X		X		X			2	2700°F	1 Hr.											3				115		120		110				Develop.			
007	5-3/4"	Paste	4 & 5 Al Bronze	No	X		X	X	X	X	X	3	2700°F	1 Hr.	3		5												115		120	115	110	75	75		Develop.			
008	5-3/4"	Paste	4 & 5 Al	No	X		X	X	X	X	X	3	2700°F	1 Hr.	3		5												115		120	115	110	75	75		Develop.			
009	5-3/4"	Paste	Al Bronze	No	X	X	X	X	X	X	X	3	2550°F	1 Hr.					4	4				A			Uncleaned prior to anneal, also too short	115	110	105	75	75	70	60		Develop.				
010	5-3/4"	Paste	Al Bronze	No	X	X	X	X	X	X	X	3	2550°F	1 Hr.					4	4				A			"	115	110	105	75	75	70	60		Develop.				
011	5-3/4"	Paste	Al Bronze	No	X	X	X	X	X	X	X	3	2550°F	1 Hr.					4	4				A			"	115	110	105	75	75	70	60		Develop.				
012	5-3/4"	Paste	Al Bronze	No	X	X	X	X	X	X	X	3	2550°F	1 Hr.					4	4				A			"	115	110	105	75	75	70	60		Develop.				
013	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X	X	X	X	3	2700°F	1 Hr.		5			3				4				Used Concentricity Fixture	60	115	110	105	75	75	70	60		Develop.			
014	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X	X	X	X	3	2700°F	1 Hr.													Used Concentricity Fixture	60	115	110	105	75	75	50	60		Develop.	Assigned to B-1a		
015	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X	X	X	X	3	2700°F	1 Hr.													Used Concentricity Fixture	60	115	110	105	75	75	50	60	Qual.		Assigned to B-2a		
016	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X	X	X	X	3	2700°F	1 Hr.													Used Concentricity Fixture	60	115	110	105	75	75	50	60	Qual.		Assigned to Weld Qual.		
017	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X	X	X		None	None														Used Concentricity Fixture	60	115	110	105	75	75	50	60	Exp.		To certify annealing cycle		
018	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X	X	X	X	3	2700°F	1 Hr.													Used Concentricity Fixture	60	115	110	105	75	75	50	60	Test		Assigned to B-3a		
019	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X																			Used Concentricity Fixture	60	115							Test	To be Assigned			
020	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X																			Used Concentricity Fixture	60	115							Test				
021	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X																			Used Concentricity Fixture	60	115							Test				
022	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X																			Used Concentricity Fixture	60	115							Test				
023																																								
024																																								
025																																								

Table VI-1. . T-111 Structural Shell Fabrication History.
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Figure VI-5. T-111 Shell S/N 003
Cocked During Second Draw Through
Steel Die

Figure VI-6. T-111 Shell
S/N 004 Pulled Apart at
the Top After the Draw

S/N 005 through 008

Four T-111 plates were deep drawn through the first and second stages and the parts annealed at 2700°F/1 hour in a vacuum of 1×10^{-6} torr. After annealing the parts were drawn through the third and fourth stages. During drawing of S/N 005 and S/N 006 through the third stage die, the parts locked in the die resulting in the punch being pushed through the nose end of the part. S/N 007 and 008 were drawn through the third, fourth, and fifth stage dies successfully. However, severe galling of the ID and OD of the parts occurred. Figure VI-7 shows S/N 005 with the bottom punched out during the third stage draw.

Figure VI-7. T-111 Shell S/N 005 with Bottom Punched out During Third Stage Draw After Annealing at 2700°F/1 Hr.

Investigation of S/N 005 and 006 failures indicated that the material reduction was too great resulting in a forming force that failed the parts in tension. Two intermediate dies (1a and 2a) were added to reduce the amount of material being moved per draw in order to reduce the forming force on the part.

To eliminate the galling experienced in deep drawing of the T-111 structural shell, new draw rings were fabricated from aluminum bronze. With the draw rings softer than the T-111, any galling or wear should damage the draw ring and not the part.

S/N 009 through S/N 012

Four T-111 blanks 5-3/4" diameter were drawn through the aluminum-bronze dies to form four T-111 structural shells. Annealing of parts between draws was determined by hardness readings measured after each draw. Table VI-2 shows the fabrication sequence used for these four parts.

TABLE VI-2
T-111 SHELL FABRICATION SEQUENCE

<u>Die Draw</u>	<u>Ra</u>	<u>Annealed</u>	<u>Temperature</u>
1	63	No	---
1a	65	No	---
2	66	No	---
2a	67	No	---
3	68	Yes	2700°F/1 hr.
4	66	No	---
5	--	No	---

Immediately after annealing and drawing through die No. 4, all four shells showed longitudinal surface tears and checking on the outside diameter of the parts. After drawing through the final sizing die, inspection of the shells indicated there were four problems to be solved:

Surface Tears

Preliminary analysis indicated aluminum bronze from the dies had deposited on the T-111 and was not removed before annealing. To correct this, NaOH and nitric acid cleaning after each draw was used on all subsequent parts up to S/N 017.

Shell Length

The four shells were under size in length due to trimming of the parts at the open end. The blank diameter was increased from 5-3/4" to 6-1/2" for a trim allowance.

Internal Galling

Examination of the galling on the ID of the shells indicated that the draw punch needed polishing and must be removed in such a manner as to eliminate any cocking of the punch as it is being withdrawn from the shell. A removal fixture was designed to assure alignment of the punch and shell during this operation.

Concentricity

Due to uneven load distribution on the shell during final sizing, the ID and OD were eccentric approximately 0.050". In order to correct this condition a guide and locating fixture was designed to support the shell during drawing and final sizing.

The T-111 shell drawn through the aluminum bronze dies is shown in Figure VI-8. The shell is short in length and has many small surface tears. Figure VI-9 shows the design constraints of the concentricity fixture utilized during the remaining draws to maintain the ID to OD concentricity of the shell to 0.005" total indicator reading (TIR). The punch diameter "A" is concentric to the draw ring diameter "B" by mechanics of tool alignment. Diameter "C" on the T-111 part is round by the mechanics of drawing around the punch on the previous draw. Therefore, by machining the ring, diameter "X" is

6-3/8"

Figure VI-8. T-111 Shell
Drawn from 5-3/4" Dia.
Blank. Part was contam-
inated During Annealing
Resulting in Surface Tears
Appearing in the Drawing
Sequence Immediately
Following Annealing at
2700°F/1 Hr.

Figure VI-9. Concentricity
Fixture Design Capable of
Maintaining .005 TIR Con-
centricity of Shell ID to OD.

concentric to diameter "Y" which, when utilized in the drawing process as shown in Figure VI-10, aligns diameter "C" so it is concentric with diameter "A" within 0.005" TIR. All shells fabricated utilizing this concentricity fixture were drawn with the ID's concentric to the OD's within this 0.005" allowable.

S/N 013

One 6-1/2 inch diameter blank was drawn through the fabrication sequence. After annealing at 2700°F/1 hour following draw No. 3, two longitudinal surface cracks were exposed approximately 3/4" from the open end of the part. The end 3/4" was removed from the open end and the part drawn through the fourth and fifth dies. During the fourth draw two small surface tears developed approximately 1/8" from the tangent point of the closed end. The part was then drawn through the final die to see if any new tears developed. No new tears were visible. However, the two existing tears opened up.

S/N 014

This 6-1/2" diameter blank was drawn through all dies up through the No. 3 die and held from further fabrication pending metallographic examination to determine the proper annealing cycle. (See Section V - Process Development).

After careful examination of both the T-111 structure drawn through the first six of eight dies and the annealing time/temperature cycle being employed after the sixth drawn, the T-111 shell was annealed at 2700°F for 1 hour and successfully drawn through the final two stages of the fabrication process. Figure VI-11 shows this T-111 structure after passing through the eight stage deep drawing process. The shell is 1.674 inches in diameter and 8.000 inches long. Six and one-half inches is required for the structural shell while the last 1-1/2 inches can be used in the before and after welds required when the T-111 shell is electron beam welded to the T-111 end cap.

This was the first shell successfully fabricated and was designated for use on Creep Capsule B-1a, the first creep capsule to be delivered to test. The

Concentricity Fixture

T-111 Shell

Al-Bronze Die

Figure VI-10. T-111 Shell Ready to be Deep Drawn
Through Die Number 7.

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STRUCTURE SHELL

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Figure VI-11. T-111 Structure Shell

fabrication sequence for the T-111 plate as it passed through the forming sequence is shown in Table VI-3.

S/N 015 and 016

Fabrication of these two structural shells was initiated to certify the tooling. After drawing through six dies and annealing at 2700°F for 1 hour, the two parts were drawn through the No. 7 die. Examination by chemical etching disclosed that both parts had small superficial imperfections. The two parts were returned to AI for thorough examination and evaluation. The minute cracks in S/N 016 were scarfed out and the part drawn through the final stage. No surface imperfections or additional cracks were found. In order to duplicate the metallurgical interface involved when welding the T-111 end cap to the T-111 shell, this capsule was assigned to weld development. This structural shell was cut into 3/4" sections, annealed at 3000°F/1 hr (the shell and end cap annealing temperature/time cycle) and prepared for welding with formed T-111 end caps. S/N 015 was examined and the decision made to scarf out the minute cracks and draw the shell through the eighth and final die. This was accomplished successfully and the shell assigned for use on Creep Capsule B-2a, the second creep capsule to be delivered to test.

S/N 017

In an attempt to produce a T-111 structural shell without annealing after the sixth draw, S/N 017 was drawn through the seventh die. Figure VI-12 confirms the decision to anneal after the sixth draw.

S/N 018

In an attempt to produce a T-111 structural shell without intermediate cleaning, after each draw, S/N 018 was drawn through six draws, cleaned and annealed at 2700°F/1 hr and successfully drawn through the final two dies.

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PART NO N73420005-01
 SERIAL NO 014

T-III DEEP DRAW FORMING
SEQUENCE

DATE 2/14/68

T-III BLANK

DRAW#0
LOAD 600

REMOVED -

DRAW#1
LOAD 1150

REMOVED .13"

DRAW#1A
LOAD 1100

REMOVED .075"

DRAW#2
LOAD 1050

REMOVED .095"

HOLD DOWN LOADS ON DRAWS
 # 1A, #2, #2A, #3, #4 & # 5 = 90 lbs.

DRAW#2A
LOAD 75.0

REMOVED .0.75"

DRAW#3
LOAD 75.0

REMOVED .070"

DRAW#4
LOAD 50.0

REMOVED -

DRAW#5
(FINAL)
LOAD 70.0

REMOVED .125"

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Table VI-3. T-111 Fabrication Configuration During Eight Draw Sequences which Produced the First T-111 Structural Shell

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Figure VI-12. T-111 Shell S/N 017 Drawn Through 7 or 8 Draws
without Annealing

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S/N 019 through 022

These shells are being fabricated and have been drawn through the first four dies. Figure VI-13 shows these four parts after the second draw.

Figure VI-13. Four T-111 Blanks After
Second Draw

3. T-111 Structural End Cap Fabrication

T-111 Structural End Caps

Four T-111 blanks for structural end caps were drawn through the first stage die, annealed at AI, and sized to the final configuration of the end cap.

During the second stage die operation the boss on the end cap failed to form. This was due to bridging of the punch load to the female die so that force was unavailable to form the boss. Four new blanks were prepared with

the material for the boss protruding from the blank (see Figure VI-14). These four blanks were drawn through the final die and inspected. Two problems developed.

- 1) Due to the small radius at the boss to plate (0.010R), the material separated at this point resulting in a 0.010"-0.015" deep crack.

- 2) Due to the blank configuration coupled with the high loading of the press (approximately 70 ton) the T-111 material thinned at the nose.

Evaluation of the blank design and the forming process resulted in the following modifications:

- 1) A full radius was provided at the boss-to-plate transition to eliminate the cracking of the material at the boss during forming.

- 2) To eliminate the thinning at the end of the cap, tapering of the blank plus forming at a reduced load was employed. The constant load was set at 30 ton. The blank size is shown in Figure VI-15.

Three T-111 blanks were prepared for drawing and were formed to the "structural end cap with the feed-through" configuration. A boss formed without cracking on these parts but it was shorter than desired for welding (see Section V).

A metallographic analysis of sectioned T-111 end caps was conducted in the as-received and heat treated conditions. This effort was directed at determining what degree of work had been done in the vicinity of the boss and how this structure responds to heat treatment. Inspection of the wall thickness indicated that the 0.140 dimension of Figure VI-15 should be increased to 0.146 and the 0.120 dimension decreased to 0.118 to provide the 0.127 constant wall thickness. Figure VI-16 shows the wall measurements taken on the T-111 end caps formed from the blank design shown in Figure VI-15.

Figure VI-14. T-111 Structural End Cap Blank

Figure VI-15. Revised T-111 Structural Cap Blank Design

Figure VI-16. End Cap T-111 Structural Cap Wall
Variations After Forming

Ten new end cap blanks were prepared to the new blank design, satisfactorily formed at the 30 ton constant forming force, cleaned and annealed at 3000°F/1 hr, and delivered to weld development. Five of the end caps had the feed-through attachment boss lengthened by T-111 "TIG" weld build up to provide a satisfactory weld joint for the EB welding of the Ta-10W pressure feed-through tube to the T-111 boss. The remaining five caps were machined and an attempt made to EB weld the Ta-10W tube to the short boss on these caps. The weld development progress is reported in Section V.

4. Ta-10W Inner Liner Flanged Cap

Tooling for this part has been completed as shown in Figure VI-17, checked out using copper blanks, and is presently being employed to fabricate three Ta-10W end caps to certify the tooling.

An inner liner flanged cap fabricated from copper to check out the tooling is shown in Figure VI-18.

5. Capsule Assembly Tooling

All assembly tooling required for the LRHSC Development Program has been received. Six major assembly tools are employed in this program. Photos of this tooling are shown in Figures VI-19 through VI-24 along with a description of their functions.

The two weld heads 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 are shown in Figures VI-25 and VI-26.

6. Creep Capsule B-1a Fabrication and Assembly

The T-111 structural shell S/N 014 assigned to the first creep capsule B test was machined to final dimensions to provide a constant wall thickness of $\pm .0005$. The final 1-1/2 inches of this shell was removed and machined to provide the T-111 shell "Before" and "After" weld material with the T-111 end caps.

Figure VI-17a. Ta-10W Inner Liner Flanged
Cap Initial Coining Die

Figure VI-17b. Ta-10W Inner Liner Flanged
Cap Final Coining Die

Figure VI-18. Copper End Cap Used to Checkout
Inner Liner Flanged Cap Tooling

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Figure VI-19. T-6001638 — E. B. Weld Fixture,
Radioisotope Liners

This assembly fixture is used for all E. B. welds of the capsule up to the point of simulated fuel loading and prototype thermal environment of the capsule. This fixture is capable of performing three liner welds during a single setup.

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Figure VI-20.

T-6001643 - Press-on Tool, Capsule
Assembly, Radioisotope

The function of this tool is to fit all components
together prior to electron beam welding on
E.B. Weld Tool T-6001638.

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Figure VI-21. T-6001654 - E.B. Weld and Assembly
Fixture, Large Radioisotope Element
The function of this tool is to permit assembly of
the capsule structure cap and shell while in the
vacuum chamber.

Figure VI-22. T-6001656 - E.B. Weld Fixture Structure
Shell Feed-Through Cap to Outer Liner Cap

This tool is a locating fixture for the electron beam
welding of the outer liner end cap to the T-111
structural shell

Figure VI-23.

T-6001645 - Chill Block and Automatic
Weld Head Locator, Radioisotope

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.

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Figure VI-24. T-6001650 - Plug Locator
Inner to Outer Liner Radioisotope

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

Figure VI-25. TIG Weld Heads

Figure VI-26. TIG Weld Heads

The Ta-10W pressure-feed-through tube was welded to the T-111 end cap and the cap final machined to provide proper fitup for welding with the T-111 shell. After welding of the pressure-feed-through to the end cap, helium leak check and dye check inspections were completed satisfactorily on the assembly.

The T-111 shell (S/N 014) was helium leak checked, ultrasonically and dimensionally inspected, and the as-built OD, ID, wall thickness and length were recorded. Weld development of the T-111 shell to cap was completed and the T-111 shell EB welded to the T-111 cap. The capsule was then helium leak checked, ultrasonic inspection of the weld completed, the feed-through flange brazed to the Ta-10W tube, and the capsule proof tested successfully at 10,000 psi. This capsule was designated Creep Capsule B-1a and delivered to test on March 29, 1968.

The "Before" and "After" weld samples prior to EB welding of the first creep B T-111 shell to T-111 end cap weld are shown in Figure VI-27. The T-111 structural shell and the T-111 end cap with the Ta-10W tube welded in place are shown prior to final EB welding in Figure VI-28. The first "B" type capsule after welding and prior to brazing of the feed-through flange is shown in Figure VI-29.

7. Creep Capsule B-2a Fabrication and Assembly

The T-111 structural shell S/N 015, assigned to the second creep capsule B test was machined to size, inspected and welded to the T-111 end cap with the Ta-10W pressure feed-through tube attached. This capsule was successfully inspected ultrasonically, as built dimensions recorded, the feed-through flange brazed to the Ta-10W tube, and the capsule proof tested at 10,000 psi. This capsule has been delivered to test.

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Figure VI-27. T-111 Cap-To-Shell "Before" and "After" Weld Material

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Figure VI-28. Creep Capsule B-1a T-111 Shell and T-111 End Cap Prior to E.B. Welding

Figure VI-29. Creep Capsule B-1a After
E. B. Welding or Cap to Shell

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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 at 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. The components for three 1200 l/sec vacuum ion pumps and vacuum chambers with power supplies and instrumentation readout have gone through initial checkout and repair. The design, construction, and integration of all components required for the first rig is complete. The assembly has completed tune up and shake down runs and the first creep test is in progress. Because a number of minor difficulties showed up in the tune up and shake down process, the final

design and construction of the remaining two systems was delayed. These three systems will be assembled and disassembled many times next year for the short term creep test. Therefore, it was deemed desirable to work out all the bugs with the first system before building the other two. The design and construction of the following two is scheduled for early next quarter.

C. PROGRESS DURING THE QUARTER

1. Environmental Chamber

Very few changes from the initial design were required for the chamber. Figure VII-1 shows the location of the feed-throughs on the upper chamber flange. The power, window shutter rotator, and instrumentation feed-through have not been changed. However, the burst diaphragm and pressure lead go through the same flange. This moves the burst diaphragm into a location of more constant temperature. It also moves the transition joint between the Ta-10W and stainless steel pressure tubes out of the main chamber and away from the furnace into a cooler region. If necessary, additional cooling coils could be added to that region but it has not been required. The remaining port was then sealed. It may be of interest to note that the one rotational motion feed-through services both sight windows.

Early in the quarter it was noted that the sight windows over-heated when the shutters were removed to permit optical strain measurement. The radiation from the two sight holes in the furnace caused two hot spots on the windows. To eliminate this, a set of four stainless steel shields were placed inside the window ports. Holes were punched in them for viewing. The windows now remain cool even with the capsule at 2500°F. The nominal test temperature is 2000°F.

The remaining two systems will be built to approximately the same design. Only minor modifications will be made to ease assembly.

Pressure Supply

Rupture
Diaphragm
and
Expansion
Baloon

Blank

Thermocouple

Shutter Rotation

Furnace Power

Figure VII-1. View of upper chamber flange showing the location and function of the high vacuum feed-throughs

2. Furnace Design

After the baseline tests of a basic furnace design indicated that the thermal gradient in the furnace at 2000°F was less than 2.5°F, the furnace was modified for the access ports and sight tubes. The alumina sight tubes encroach on one diameter of the capsule for viewing. The obverse diameter was to be viewed through the holes in the tubes as shown in Figure VII-2.

The following tests indicated that the heat leak through the sight port overheated the windows, and decreased the temperature of the side of the capsule simulator facing the ports. The simulator used in these tests was a tantalum tube about 0.020" in thickness. The thin section provided a more sensitive test of the gradient compared to an 1/8" thick structural shell. The sight tubes provided an excellent means of viewing the edges of the capsule at temperature. Removing the heat shields opposite the viewing ports, however, was required to view the capsule edges. This was established in the obverse view where the shields were not punched. In this case, it was virtually impossible to distinguish the capsule edges.

To alleviate the window overheating, a series of steel reflectors were added to the two window ports. For the capsule cooling, a set of four cylindrical (annular) baffles were added inside the furnace around the capsule. To aid the lateral viewing, holes were punched in the appropriate places opposite the viewing port.

While the furnace was at power, the windows remained cool even with the central furnace temperature at 2500°F (the creep test temperature is 2000°F). The capsule simulator was cooler on the ends than the middle indicating the baffles were an effective technique for smoothing out the gradients caused by the sight holes. The circumferential gradient decreased from 150°F to <2°F at 2000°F.

Figure VII-2. Strain access subassembly showing the relationship of sight tubes, black body, and radiation shields with capsule.

The cylindrical baffles, added to the center of the furnace to improve the thermal profile, presented some problems in sighting the capsule from the obverse direction, i. e. perpendicular to viewing down the bore of the sight tubes. Some improvements were required in order to view the capsule through the five window shields, the three outer furnace shields, the four inner shields, the four baffles, the alumina tube and line up on the cold spot through the second alumina tube, four baffles and three of the four inner furnace shields. The sight holes in each of these were made as small as possible to reduce temperature perturbations. The view down the sight tubes was excellent.

During the test the thermocouple at the top of the furnace where the pressure tube would be was indicating 1800°F when the furnace central temperature was 2000°F. This would be too hot for a braze joint. It was

also noted that there was a shift in indicated emf caused by the vacuum feed-throughs. The shift was uncompensatable. The history of the problem and its solution is detailed in the next section.

To alleviate the difficulty with the lateral sight ports an additional set of sight tubes was added to the furnace. Their position in the cylindrical baffles is shown in Figure VII-3. The axial viewing planes are $3/8$ " apart. The positioning of the capsule provided by the tubes is evident in Figure VII-4. The location of the baffles and sight tubes inside the furnace is shown in Figure VII-5.

When the test was reassembled, the capsule simulator was made of 0.127" thick T-111 and a Ta-10W lead tube was added. To determine the temperature of the braze joint a 0.215" ID x 0.064" wall Ta-10W tube was bent in a right angle, fastened to the top of the capsule and extended through the upper heat shields. The design location of the braze joint was then instrumented.

Figure VII-3. View of the four cylindrical thermal baffles and two pairs of alumina sight tubes

Figure VII-4. End view of baffles and sight tubes showing how the tube scallops align the creep capsule

Figure VII-5. Location of sight tubes and baffles in creep furnace

At 2000°F central temperature, the two views of the capsule were excellent, there was a negligible radial thermal gradient caused by the additional sight tubes and the temperature of the Ta-10W tube 2" out of the furnace was reduced to 800°F.

There was then a 30°F axial ΔT from the capsule midplane to the upper end. This was due to the thermal drain from the lead tube.

To reduce the drain four horizontal heat shields were added to the lead tube inside the cylindrical baffles at the top of the furnace to determine the relative contribution of radiation and conduction heat losses. This reduced the 30°F gradient by 4°F at 2000°F indicating that the losses were mainly conductive.

One approach to increasing the temperature of the lead tube (within the furnace), decreasing the lead tube-capsule ΔT , and hence the heat drain, would be to add a guard heater to the present furnace. This would require separate control, additional feed-throughs, and a more complicated furnace. A change was made which should essentially do this without adding an additional set of windings.

The portion of the cylindrical baffles covering the lead tube were cut away exposing the tube to the direct radiation of the heater coils. A set of horizontal shields were then added to the lead tube. While thin, these provide some conductive path from the heater coil to the lead tube and also serve as heat shields in the vertical plane to prevent the capsule from radiating to the cooler furnace end shields.

This method provided an excellent method of reducing the drain. The total variation throughout the capsule at 2000°F was less than + 4°F.

The furnace runs well after at least 30 thermal cycles between 1000 and 2000°F and over 1000 hours in excess of 1500°F; 500 of those being above 2000°F.

3. Uncompensated EMF Shift Problem

In order to better understand the T/C shift problem a little history is included. For illustration of some of the methods examined, Figure VII-6 is included.

Based on a number of Pt alloy thermocouple drift studies, the hot zone wire diameter selected for this program was 0.040". While there are a number of manufacturers who can supply variable diameter vacuum T/C feed-throughs, most of them utilize an epoxy type seal. The epoxy is not desirable for bakeable high vacuum systems. The brazed type assembly (Ni or Fe base tubes brazed to a high fired ceramic or glass) was selected for this program. This limited the number of suppliers and available tube sizes. With the exception of one supplier the maximum tube inside diameter found was 0.035 to 0.040". This one supplier can supply the tubes to as large as 0.050 to 0.055".

The first T/C harness arrangement is shown in Figure VII-6a. A series of Pt alloy wires were brazed to feed-through tubes. The appropriate legs of each T/C were then spot welded to the feed-through subassembly. It was found that when heat was applied to either the spot weld or the braze joint, a shift in the apparent T/C emf was noted. A number of different spot welding techniques and tips (W, Cu-W, Cu) were tried but did not eliminate the problem.

The arrangement was then changed to that shown in Figure VII-6b. Relocation of the braze to a cooler area and elimination of the spot weld seemed to minimize the shift in preliminary tests. However, while the system was running during one test, ice was packed around the braze joint and caused a shift in the emf output of the assembly (from 2000°F to approximately 1930°F). It was felt that the combination of braze and mechanical junction of the sensor to the feed-through tube was causing the problem.

Figure VII-6. Representation of a number of T/C sealing techniques for feed-throughs

A harness was then made up with three T/C's as in Figure VII-6b, c, and d. To make c and d the ends of the wire that were to go through the feed tube were swaged down to 0.030". The portion of the wire in the furnace remained at 0.040". After swaging, the wires were cleaned in nitric acid to remove any die wash and heat treated at 2700°F for 10 minutes. A supplier of insulation compatible with the 0.050" tube ID and 0.030" wire OD was finally found. The assembly was then tested for shift utilizing the copper freezing point (1981.9°F) as a reference. The indicated temperature of the T/C assembled like Figure VII-6b shifted whenever heat (+30°F) or ice (- ~ 40°F) was applied. The greatest shift that could be caused to the T/C's assembled like Figure VII-6c and VII-6d was 3°F.

The T/C harnesses were then assembled utilizing the technique of Figure VII-7d, calibrated and assembled to the creep rig chamber.

Operation of the rig indicated that the emf shift problem has been solved.

4. Strain Measurement

While the view of the capsule diameter at 2000°F was considered excellent, a series of filters were tested. It was found that some filters did help, and they have been incorporated into the telemicroscope.

After operation of the furnace at 2500°F for 100 hours (to thermally soak a coupon stack), it was noted that the weight of the thin cylindrical baffles caused the alumina sight tubes to creep. It was sufficient to obscure the cold viewing spot from the cathetometer. While this is not expected to cause a problem at 2000°F, a support fixture for the baffles will be incorporated into the furnace design as insurance for the longer term tests.

5. Pressure Attainment and Control

The pressure control system can best be explained by a discussion of Figure VII-7, a view of the console and Figure VII-8, a simplified block diagram of the system. From the front of the console, one can see the

pressure supply and bleed bottles, a 0-100 psi recorder sensing the ΔP across the transducer, a 0-5,000 psi gauge, a test timer and reset counter, the DC power supply, a proportioning signal comparator, various switches controlling the motor valves and ramp generator, and manual meter valves.

The basic principle of the system is one of bleed and feed. In operation, the signal from the transducer is compared to a known controlled voltage. If the signal from the transducer is lower than the controlled signal, indicating that the capsule pressure is below that required, the main pressure motor valve opens. This increases the pressure in the capsule. When the signals balance, the valve closes. Since there is a constant bleed from the system the pressure soon falls below the set point; the valve reopens and the cycle continues. Because there is a ΔP across the main pressure valve, there will be a slight over pressure after the valve closes, and a small under pressure when opening because of the hydraulic impedance in the system. This is controlled by meter valves and line sizes. In practice the regulator keeps the ΔP across the motor valve between 100 and 200 psi. This keeps the total over- and under-pressure to less than 5 psi on the bleed and feed cycle. For the first test, B-1a, the initial pressure is 2840 psia. This is the valve shown in Figure VII-8. The total variation on the chart in the figure is less than 1.5 psi. This is very small compared to the magnitude of the pressure.

The standard electrical signal that the transducer compares to is the voltage produced from a constant DC power supply. This is reduced by passing through a linear 10 turn pot which turns at 1 rph. To hold a constant pressure the pot motor is turned off. To increase at a constant rate the motor is simply turned on. Since the pot and motor are linear the ramp rate is changed by merely altering the input voltage. For instance, if the DC power supply is set a 10V the output of the ramp generator is 1 V/hr. This

Figure VII-7. Front View of Pressure Control Console

Figure VII-8. Block Diagram of LRHSC Pressure Control System

corresponds to 10 psi/hr. At 8.85 V the rate is 0.885 V/hr and 0.85 psi/hr. Three ranges of diaphragms cover the 0.1 to 10 psi/hr required for the program. A 1 psi diaphragm is used for the 0.1 psi/hr range; the 10 psi diaphragm for the 1 psi/hr range, and 100 psi diaphragm for the higher ranges. The control bands are ± 0.015 , ± 0.15 psi and ± 0.5 psi respectively for the diaphragms. These are installed in the systems as required for each test.

When the pot in the ramp generator reaches its full 10 turns, the transducer is also at the top of its range. When this occurs, the reset motor valve opens and reduces the ΔP across the transducer to zero. It then closes. At the same time the ramp generator is reset to zero, and the regulator starts up to increase the ΔP across the main pressure motor valve by the appropriate amount. A counter then indexes to indicate the event.

The motor valves and ramp generator have both automatic and manual control modes. There are a number of automatic shut down modes which are built into the system for safety. The most important one is for the capsule rupture event.

Normally if the capsule ruptures this would indicate a loss of pressure, therefore, demand more pressure. However, when rupture occurs, bleed far exceeds the feed supply and puts a back pressure across the transducer. This trips the differential pressure switch which removes all motor valves from automatic mode and closes them. When the back pressure ΔP across the transducer reaches 300 psi, the reference pressure dump valve opens and equalizes the pressure across the transducer and closes. This saves the transducer diaphragm from rupture. The system cannot now be restarted automatically. It remains in the all valves closed condition until reset manually.

6. Creep Testing

A creep capsule designated B-1a was installed in the chamber and is now under test. "B" designates that the test sample is the structural shell alone;

"1" designates the stress mode: a constant stress until 0.1% diametral strain is achieved (in about 60 hours), and an additional stress rate until the capsule creeps to 1% total strain (in about 100 hours), and the test is terminated; the "a" indicates the heat of material used.

After the lead tube is formed to that configuration shown in Figure VII-9, it is assembled to the upper support plate (top flange). The lead tube is field brazed through one of the flanges and a high pressure tube fitting is attached. This method minimizes the number of critical high pressure joints requiring stringent inspection. The joints that do require detailed inspection are the end cap to structural shell, lead tube to end cap (both are EB welds) and the Ta-10W stainless steel transition braze. The feed-through chamber joint is required to withstand only 1 atm ΔP .

The thermocouples are attached to the capsule by means of 0.005" thick, unalloyed tantalum foil bands. Small cups attached to the bands shield the T/C's from direct radiation from other parts of the furnace. Figure VII-10 shows the capsule and thermocouple locations, positioned on the upper support plate. The furnace is then brought up around the capsule and bolted in place as shown in Figure VII-11. The assembly is then lowered into the chamber as in Figure VII-12 and sealed. The internal pump and furnace heater are then turned on and the system is sorption pumped down to 10^{-4} torr and baked out. The sorption pump cart and sealed chamber are shown in Figure VII-13.

After the chamber and pumps bake out at this intermediate level, the system is sealed from the roughing cart and the ion pump is started. After the system pumps down to the 10^{-7} torr range the furnace power is brought up to 1400-1500°F and held while the pump heater is shut off and the ion pumps cool down. The cooling water is then added to the chamber and the capsule temperature increased to the 2000°F test temperature.

Figure VII-9. Capsule and Lead Tube Configuration

Figure VII-10. Capsule installed to upper support flange. The three highly reflective surfaces on the capsule are the unalloyed tantalum foil T/C straps

Figure VII-11. Completed furnace and capsule sub-assembly

Figure VII-12. Furnace and Capsule Subassembly being lowered into the test chamber

Figure VII-13. Start of vacuum roughing cycle. The operator is valving in sorption pumps on the roughing cart to the creep test chamber

During this period the interior of the capsule is also evacuated and baked and then filled with high purity helium. An expandable chamber is added to the outlet of the burst diaphragm and the capsule is pressurized. The test assembly is shown in Figure VII-14.

The function of the expanding chamber and burst diaphragm are to prevent the pressure inside the vacuum chamber from exceeding 2 atm without letting air in the chamber in the event the capsule ruptures and the shut down mechanism in the control system fails to operate. The burst diaphragm ruptures when the pressure in the chamber exceeds 1 atm over the external pressure. This gas is then collected in a weather balloon which unfurls and prevents air from bleeding back into the system to ruin the capsule and furnace.

Figure VII-14. Test Assembly in Operation

The gas analysis sampling is shown in Figure VII-15. During evacuation of the chamber the interior of the capsule is also evacuated through the sampling system. When the test is started the system is back filled with helium, a sample taken and then analyzed. The sampling system is normally isolated from the capsule during test, however, a sample can be taken during operation if required.

The first test has just begun. There are insufficient data to report at this time.

Figure VII-15. Gas sampling system. One of the sample bottles have been removed and access ports plugs

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VIII. COUPON COMPATIBILITY AND COATING DEVELOPMENT

A. 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.

B. SUMMARY OF TASK STATUS

To date all of the coupon stacks which were scheduled for testing this fiscal year have been tested. The new schedule and task status is shown in Table VIII-1. Since the last quarter, only one stack has been metallographically examined. This stack was CS7 (2500°F - 10 hr.).

Observations which were made on CS7 gave the first real Pt-Ta reaction data. It was discovered in CS2 (2000°F - 100 hr.) and CS4 (2250°F - 10 hr.) that the "one side only" CVD alumina coating operation leaves a very hard, thin film of Al_2O_3 on the back face of the T-111 coupons. This thin layer was sufficient to prevent the predicted Pt-Ta reactions. To prevent this interference, the back faces of the CVD coated T-111 disc in CS7 were machined smooth leaving a metal finish to be mated with the Pt coupon.

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	Complete
CS7*	1-03-68	2500	10^{-8}	10	Complete
CS8*	1-08-68	2500	10^{-8}	100	Complete
CS5	1-25-68	2250	10^{-8}	100	Complete
CS6	2-03-68	2250	10^{-8}	1000	Complete
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	

* Tested in Creep Test Rig #1

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

C. ACCOMPLISHMENTS DURING THE QUARTER

All compatibility tests scheduled for GFY 1967 have been completed. Throughout the test series the performance of the compatibility test rig (vacuum chamber and tantalum-wire furnace) has been excellent. Only two modifications were made to the system since the first start up. This was the replacement of the unnecessary side window with a blank flange and the addition of several new shields to cool the bell-jar and window during operation at 2250°F.

All of the 2500°F tests (CS7 - 10 hr and CS8 - 100 hr) were tested in creep testing Rig # 1. These tests were satisfactorily run in conjunction with the check out of the rig for isothermal creep testing. In one of these tests, a coupon stack was actually assembled into the body of a LRHSC capsule simulant.

Only one stack, CS2 (2000°F - 100 hr) has completely undergone post-test analysis through electron beam metallographic analysis. The coupon stack as it was examined was identical to the array shown in Figure VIII-1. The stack interfaces were numbered 1 through 13, beginning with the interface between the Al_2O_3 spacer disc and the double coated T-111 coupon. The microprobe sample shown in Figure VIII-2 shows a portion of the sectioned coupon stack. Evaluation traces were made down the stack center line, the region through the welds in the T-111 coupons, and through the tapered edges (corresponding to the reference diameters A, B and C of Figure VIII-1). Using these three traces, the effects of the weld structure and vapor transport on compatibility could be evaluated.

The results of this investigation enhance the findings reported in Section VIII of the last quarterly report. Again, the interfaces of interest were between iron titanate and both T-111 and Ta-10W. No other significant reactions were observed. Figure VIII-3 shows the distribution of the elemental constituents present at interface 10 (Pt, Rh, Fe and Ti) where an iron titanate coated platinum rhodium disc is opposite a bare platinum disc. Note that the distribution is normal except for a slight diffusion of Fe into Pt. Figure VIII-4

Figure VIII-1. Schematic Cross-Section of Capsule CS-2, Locating the Coupons and Coatings, and Defining the Interface Numbers and Reference Diameters

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Figure VIII-2. Electron beam microprobe
sample cut from compatibility coupon
stack # 2. Note the scribed location
of the major diameters

Fe X-rays

Pt-X-rays from Pt-10Rh Coupon

Ti X-rays

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

← Some Fe diffusion into the Pt-10Rh coupon

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

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

400X Photographs, $22\frac{1}{2} \mu$ per large division

was observed at interface 5 where Pt-Rh coated with iron titanate was in contact with Ta-10W. This was one of the interfaces where significant reaction was noted metallographically. Here it may be observed that the iron, which was a constituent of the coating, has left the coating and diffused into the Pt coupon.

From these observations, it may be postulated that the iron oxide component of the coating ($\sim \text{Fe}_2\text{O}_3$) was reduced to Fe by the tantalum which oxidized and the iron was removed to the Pt by diffusion. The photograph in Figure VIII-4 indicates that this reaction went to near completion with no effect on the titanium distribution.

Other significant observations made by use of the microprobe were that there was no apparent difference in the compatibility behavior of the EB welded T-111 and the remaining wrought structure of the coupon disc. It was also revealed that the CVD alumina coating process deposited Al_2O_3 on both sides of the disc, as seen in the edge profiles of Figure VIII-5. The intention was to coat only one side allowing the other to be used for base metal testing. Also, in the survey of the coupon edges, there was no evidence that vapor transport diffusion reactions had occurred between any coupons.

Metallographic analysis of CS7 (2500°F - 10 hr) gave the first data on a reaction between Pt-10Rh and T-111. This reaction, a portion of which is shown in Figure VIII-6, measured approximately 0.0015 inches wide. Upon examination at higher magnification (1000X), a distinct columnar region of the reaction zone was observed. This is shown in Figure VIII-7. The nature of this reaction is being investigated.

Pt X-rays from Pt-10Rh Coupon

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

Ta X-rays from Ta-10W Coupon

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

Figure VIII-4. These four area scan photographs were taken at Interface 5 on diameter B and illustrate (1) the Ti remains in the coating, (2) the Fe separates from the coating and diffuses into the Pt-10Rh, and (3) there is little or no diffusion of Pt and Ta into the coating

400X photographs, 22-1/2 per large division

Figure VIII-5. These area scan photographs, taken at the tapered ends of the T-111 coupons located between Interface Numbers 6 and 7, and 11 and 12, show $\text{Al}(\text{Al}_2\text{O}_3)$ on both sides of each coupon, whereas (see Figure 1) only Interface Numbers 6 and 11 were expected to be coated with Al_2O_3 . 200X Photographs
45 per large division

Figure VIII-6. Photomicrograph of a reaction occurring at an interface of Pt-10Rh and T-111 in CS7 (2500°F - 10 hours)

Figure VIII-7. High magnification view of the reaction area between Pt-10Rh and T-111 in CS7. Note the columnar structure at the T-111 boundary

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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 (~1150°F) on a granite target at various velocities and impact angles.

B. SUMMARY OF TASK STATUS

The impact testing will be accomplished in FY 1969. No effort is planned for FY 1968.

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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 1969. No effort is planned for FY 1968.

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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.