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LOW COST HIGH PERFORMANCE GENERATOR TECHNOLOGY PROGRAM

Component Development
June 1975

PREPARED FOR NUCLEAR RESEARCH AND APPLICATIONS DIVISION, ENERGY RESEARCH AND DEVELOPMENT
ADMINISTRATION UNDER ERDA CONTRACT E(49-15) -3075 (FORMERLY SNSO-3)

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TELEDYNE ENERGY SYSTEMS

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LOW COST HIGH PERFORMANCE
GENERATOR TECHNOLOGY PROGRAM

COMPONENT DEVELOPMENT

VOLUME 3

TES-SNSO-3-26

June 1975

Prepared for
U. S. Energy Research and
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Under Contract SNSO-3

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TELEDYNE ENERGY SYSTEMS

110 West Timonium Road
Timonium, Maryland 21093

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FOREWORD

This document was prepared for the United States Energy Research and Development Administration (Division of Advanced Nuclear Energy Systems) by Teledyne Energy Systems under Contract SNSO-3.* This is Volume 3, "Low Cost High Performance Generator Component Development," which delineates the development efforts for the system, thermoelectric hardware (Task III) and the heat source (Task IV) for the period July 1974 through June 1975.

The following organizations actively participated in this study:

Teledyne Energy Systems

Minnesota Mining and Manufacturing Company

Los Alamos Scientific Laboratory

Oak Ridge National Laboratory

*Contract Number Revised to E(49-15)-3075

TABLE OF CONTENTS

Title Page	i
Notice	ii
Foreword	iii
Table of Contents	iv
List of Figures	v
I. INTRODUCTION	I-1
II. SYSTEM AND THERMOELECTRIC COMPONENTS	II-1
A. Liaison with 3M	II-1
B. Cold End Hardware	II-2
C. Hot Side Dielectric	II-26
D. Conoseal Heat Source Cavity Closure Test	II-28
E. Fueling Cavity Friction Tests	II-29
F. References	II-32
III. HEAT SOURCE COMPONENTS	III-1
A. Heat Source Module Test Program	III-1
1. Module Definition and Test Objectives	III-1
2. Capsule Material Development	III-14
3. Zirconia/Pt-3008 Alloy Compatibility	III-22
4. Fuel Form	III-24
5. Thermal Mockup Experiments	III-30
B. Vent Development	III-37
1. Design	III-37
2. Vent Flow Tests	III-44
C. Graphite Oxidation Experiments	III-46
D. Heat Source Thermal Mockup	III-53
E. Acknowledgement	III-55
F. Bibliography - Drawings and Memos	III-56

LIST OF FIGURES

- II-1 Spring/Piston Type Cold End Hardware
- II-2 Square Follower Cold End Hardware
- II-3 Flexible Sleeve Cold End Hardware
- II-4 Flexible Sleeve Cold End Hardware
- II-5 Thermoelectric Converter Section
- II-6 Single Couple Demonstration Model
- II-7 Sleeve Wrapping-Masking Tool
- II-8 Sleeve Wrapping Tool
- II-9 Slot Punching Tool
- II-10 Slot Punching Tool-Section
- II-11 Sleeve Forming Tool-Section
- II-12 Interface ΔT Test Setup
- II-13 Temperature Drop Across Contact Gap vs Contact Pressure
- II-14 Temperature Drop Across Contact Gap vs Cover Gas Pressure
- II-15 Flexible Sleeve Cold End Hardware Evaluation Test Fixture
- II-16 Coefficient of Friction Determination

- III-1 Task IV Heat Source Technology Flow
- III-2 25 W(t) Heat Source Test Module
- III-3 50 W(t) Heat Source Test Module
- III-4 75 W(t) Heat Source Test Module
- III-5 Test Heat Source Graphite Details
- III-6 ZrO_2 End Fillers (75W Capsule)

LIST OF FIGURES (CONT.)

- III-7 Heat Source Module Configurations
- III-8 Heat Source Module Test Schedule and Sequence
- III-9 Mellen Furnace - Vacuum Line Arrangement
- III-10 Pt-3008 0.39 in. Diameter Rod - ORNL Process Flow Chart
- III-11 Pt-3008 0.020 in. Gage Sheet - Typical ORNL Process Flow Chart
- III-12 25 W(t) Fuel Capsule Piece Parts
- III-13 75 W(t) Fuel Capsule Piece Parts
- III-14 25 W(t) Prototype Fuel Pellet
- III-15 LCHPG Fuel Pellet - 25 W(t) L/D 0.65 Typical LASL Process Flow Chart
- III-16 Test Fixture
- III-17 Electrically Heated Capsule : 75 W(t) Configuration
- III-18 Electrical Schematic : Thermal Mockup
- III-19 Thermal Mockup - Heat Source Test
- III-20 LCHPG Filter Vent (75 W(t) Capsule)
- III-21 Helium Vent Micrographs
- III-22 Filter Plating Tool
- III-23 Filter Vent Alignment and Insertion Tool
- III-24 LASL Vent Design (25 W(t) Fuel Capsules)
- III-25 Cold Flow Test Diagram
- III-26 LASL Vent Flow Test Apparatus
- III-27 Schematic Diagram of the LASL Vent Flow Test Apparatus
- III-28 3-D Carbon/Carbon Oxidation Test Specimens
- III-29 Tube Furnace Test Setup
- III-30 Thermal Mockup

I. INTRODUCTION

Component development efforts in support of the Low Cost High Performance Generator (LCHPG) seriously commenced during government fiscal year 1975 although specific tasks such as the selenide thermoelectric material development at the 3M Company and certain vacuum system cold end hardware designs were well progressed at the beginning of the period. It was, in fact, the promise of the selenides to double overall efficiencies of traditional thermoelectric converters, plus promising work done in design of the curium-fueled ERTG (Economic Radioisotopic Thermoelectric Generator) which boosted incentives for development of components for the design of the LCHPG. Within this period the technology for dynamic systems also matured, systems which, if built, would require large radioisotope heat sources which could be quite similar to the LCHPG heat sources currently being developed. New heat source safety criteria were created and released by ERDA containing specifications which invited the complete re-evaluation of heat source technology as used on currently active or scheduled space applications.

This Volume 3 of the LCHPG final report documents status of the component development effort both in the thermoelectric module area and in the heat source task. In addition, related efforts by outside facilities are identified and a bibliography of heat source development documents is presented.

II. SYSTEM AND THERMOELECTRIC COMPONENTS

A. LIAISON WITH 3M

The selenide thermoelectrics are being developed by the 3M Company under a program sponsored by ERDA/DANES. In order to adapt the technology to the LCHPG system, technical liaison was maintained between 3M and Teledyne Energy Systems (TES) to define the hardware configuration requirements as well as the thermoelectric performance characteristics appropriate to RTG design. In addition, feedback of system related information to the 20 watt module fabrication activity at 3M was accomplished to allow considerations of generator design in this initial demonstration device.

The thermoelectric design parameters (i. e. efficiency, size, number of elements) for the LCHPG system design efforts were obtained in meetings with ERDA, 3M and TES personnel. The information covered operating regions of 800°C to 1000°C hot junction temperature and 125°C to 225°C cold junction. Sizes and weights were obtained for the thermoelectric module components such as the square followers cold end hardware, thermoelectric materials, thermal and electrical insulation and joining electrodes. In addition, the required spring pressures of 300 psi for the "N" leg and 150 psi for the "P" leg were established as reasonable goals for the initial LCHPG designs. The spring force is an important parameter since it exerts large influence on the design of the isolation hot frame.

Of particular significance to the system performance is the thermal insulation in the thermoelectric converter section of the generator. Through contact with 3M and the Carborundum Corporation, the Fiberfrax materials were established as leading contenders for this application. The 3M Company has identified some newer formulations such as the 660-H paper which promises to yield thermal conductivity close to that of Min-K.

Measurements of the thermal properties of the material are planned to occur at the 3M Laboratories during FY 1976.

The 20 watt module is the largest array of selenide thermoelectric elements fabricated to date. An important goal of the device is the development of system oriented production techniques and the demonstration of technology progress. To this end, the technical interfacing concerning the production of selenide thermoelectric modules will continue between 3M and TES.

B. COLD END HARDWARE DEVELOPMENT

1. System Description and Performance Objectives

a. Concept Development

During the early phase of the LCHPG study program it became apparent that an improved method of conducting heat from the thermoelectric element cold junction to the heat sink/radiator area could significantly improve generator performance and simplify its assembly. As a result, the flexible sleeve cold end hardware idea was conceived.

The then known devices in use in space power RTG's, utilizing conventional dual element couples, involved the use of springs and pistons of some type on the thermoelectric module cold end of the generators (see Figure II-1).

One recent modification of the piston configuration is the "square followers" concept of the 3M Company (Figure II-2). The piston type devices, even in their simplest form (HPG S/N 02) require a relatively massive structure to engage and support the piston such as a cold frame or a module bar. The many non-bonded interfaces, both clamped and sliding (module bar to housing, piston to conductor and piston to cylinder wall), contribute significantly to the relatively high temperature drop between the cold junction and the heat rejection member (housing and fins), associated with these devices.

FIGURE II-1

SPRING-PISTON COLD END HARDWARE

FIGURE II-2

SQUARE FOLLOWER COLD END HARDWARE

The square follower configuration does away with the module bar or cold frame structure almost completely, replacing it with a small rail or flange against which the followers are pulled by the screw-spring connecting them. The thermal path in this case has two non-bonded interfaces, the alignment ball to follower and follower to rail. The alignment balls are bonded to the conductor strap.

In the flexible sleeve type cold end device (Teledyne Energy Systems Concept, Figure II-3) a minimum amount of material is used to achieve a highly conductive thermal path with only one non-bonded interface, located between the BeO dielectric wafer and the copper conductor strap.

The flexible sleeve cold end hardware assembly is composed of the following details:

Foil sleeve

Spring

End cap

Insulation wafer

Plated recess (in housing)

The foil sleeve is fabricated by wrapping twenty layers of a ribbon of 1145-0 aluminum foil .001 in. thick on a mandrel. The foil ribbon is procured to the desired width and then the edges on both sides are plated, using several steps, which result in a pure tin outer surface and a total surface thickness build-up of approximately .0003 in. While still on the mandrel, the final wrap is tack spot welded to the adjacent layer to preserve the form. The tube is then heated to fuse the tin and thus join the layers conductively at both edges.

The bonded tube is placed in a special tool that punches eight equally-spaced longitudinally oriented slots in the tube wall. The slots extend less than the full tube length and are offset toward one end so as to produce a narrow ring at one end and a

FIGURE II-3

FLEXIBLE SLEEVE COLD END HARDWARE

wide ring at the other. The tube is then inserted into a forming tool that sizes the end rings and flattens and bows outward, in the length wise direction, the layered columns that join the end rings, formed by the slots.

The spring is made of round 17-7 Ph stainless steel wire in the conventional manner with the end coils closed and ground flat and square.

The end cap is machined from 6061-T6 aluminum alloy rod in the form of a shallow cup. This cup is plated using the same approach as used to plate the foil ribbon edges. The inside of the cap flange, a ring around the inside end face and the complete outside end face are plated. A 0.020 in. thick beryllia (BeO) wafer is metalized for attachment to the cap with tin-silver solder.

The housing wall recesses, machined to receive the sleeve and spring, are plated in the same manner and in the same areas as the inside of the cap.

A flexible sleeve unit is assembled by soldering a cap to a sleeve, placing a spring inside, and soldering this assemblage in the housing recess. Figure II-4 illustrates the installed arrangement as well as the before and after spring deflection condition of the sleeve. Figure II-5 is an exploded pictorial representation of a pair of sleeves as they would be positioned in a generator. A photograph of a single couple cold end hardware demonstration model is shown in Figure II-6.

The flexible sleeve cold end hardware configuration promises to minimize thermal resistance between the thermoelectric cold end and the radiator, reduce weight, and maintain design end loads for elements without introducing bending and shear stresses to the elements. The hardware was designed to operate in vacuum at ΔT 's of 54 °F (30 °C) or less. A reduction of required spacing between N and P thermoelements results in an overall size reduction of the module. The flexible sleeve cold end hardware is uniquely suited for the square or rectangular generator configuration.

Legend

- ① Flexible sleeve, made from a wrapped tube of .001 in. thick 1145-0 aluminum alloy or copper foil.
- ② Spring, made from 17-7 Ph cond. C stainless steel wire.
- ③ End cap, made from machined round bar of 6061-T6 aluminum alloy.
- ④ Electrical insulation, .020 in. thick. Beryllia with one face metallized and fused to end cap.
- ⑤ Bonded connection, heat fused Sn solder.

FIGURE II-4

FLEXIBLE SLEEVE COLD END HARDWARE

FIGURE II-5

T/E CONVERTER SECTION
(insulation omitted)

FIGURE II-6

SINGLE COUPLE DEMONSTRATION MODEL

2. Manufacturing Development Effort

In order to fabricate the flexible sleeve, described in the preceding section, certain tools and facilities were designed, fabricated and procured. These items are listed below.

Pre-plating ribbon wrapping-masking tool

Sleeve wrapping tool

Sleeve punching tool

Sleeve forming tool

Cleaning bath

Electroless plating bath, zincate

Electroplating facility, Cu

Electroplating facility, Sn

The flexible sleeve design requires that only the foil layer edges be bonded (fused) together in the completed detail. A means was devised for masking the foil ribbon, and producing a spaced layer spool of the ribbon suitable for immersion in the cleaning and plating baths and subsequent sleeve wrapping. This device, the pre-plating ribbon wrapping-masking tool is shown in Figure II-7. A supply spool of foil ribbon is placed on one end spindle and a supply spool of rubberized fabric ribbon on the other end spindle. Both supplies lead to the central spindle teflon hub, foil to the bottom, rubber strip to the top. The ends of both ribbons are taped to the teflon hub and the rubber strip, which is narrower than the foil, is led over a guide follower which rides the layered spool. The guide follower precisely locates the masking ribbon on the aluminum foil so as to expose the desired edge width on the foil for plating. The drag of both feed spindles is adjustable in order to provide for adequate wrapping tension in the ribbons for sealing between layers.

The completed interleaved coil, including the teflon hub, is transferred to a plating

FIGURE II-7

SLEEVE WRAPPING-MASKING TOOL

handling tool (the exposed foil edges are delicate) which is used to suspend the coil in the various solutions for cleaning and plating, and to move it from step to step. When the plating is complete the coil is transferred from the handling tool to the sleeve wrapping tool.

The sleeve wrapping tool consists of a feed spindle, a heated wrapping mandrel and a mini-spot welder head mounted on a base stand. The unit is shown in Figure II-8. Twenty turns are wound, then the mini-spot welder head electrode is applied to the coil in two or three places to join the final lap to the adjacent foil layer thus preserving the winding. The ribbon is cut close to the spot welds to separate the foil tube from the foil supply reel. The rubberized fabric masking ribbon is allowed to unwind as it will and collect under the stand as each sleeve is wound.

The mandrel heater is activated to melt the tin plating and thus fuse the foil, at the tube edges, together. As it may be necessary to fuse the plated surfaces in a reducing environment in order to achieve a fluxless bond, the wrapping and heating may have to be accomplished in an inert gas atmosphere containing some hydrogen. In this case the entire tool would be in the glove box.

The fused sleeves are placed in the slot punching tool which is pictured in Fig. II-9 and shown in section in Figure II-10. This tool contains eight (8) punch blades which are driven through the foil tube wall by striking the top inner portion of the tool to force it down into the tool body. Raising the top allows the spring loaded punch blades to retract the sleeve to be removed.

The punched sleeves are transferred next to the sleeve forming tool shown in Figure II-11. This tool is in two halves which, by removing the lock pin, may be hinged open. With the tool open, the rubber hydroform plug is inserted in a sleeve and placed in the tool side with the indexing pins, using these pins in a sleeve slot to orient the sleeve.

FIGURE II-8
SLEEVE WRAPPING TOOL

FIGURE II-9
SLOT PUNCHING TOOL

FIGURE II-10

SLOT PUNCHING TOOL-SECTION

FIGURE II-11

SLEEVE FORMING TOOL-SECTION

The tool halves are closed, the lock pin inserted, and the ram inserted in the central cavity. With the tool resting on a firm surface, ram up, the ram is struck causing the rubber plug to expand the sleeve into the forming cavity. The ram and lock pin are removed, the tool hinged open, the sleeve with rubber plug removed and the sleeve removed from the plug. This completes the sleeve fabrication.

3. Test Effort

The single pressure contact interface of the flexible sleeve cold end hardware configuration was designed for minimum thermal resistance in vacuum based on results of a 4-months development test program which was concluded in January of 1975. The next task was to demonstrate the cold end hardware on a system basis and toward these objectives a procedure was drafted and a test module designed to conduct cold end hardware thermal, material and process evaluation tests. Results of the thermal interface test and status of the cold end module test are presented below.

a. Thermal Interface ΔT Test

The objectives of the test program were to thermally optimize the contact interface between thermoelectric current-carrying copper straps and the electrically isolated caps of the flexible sleeve cold end hardware. Initial screening tests in vacuum at design representative heat fluxes and pressures had shown temperature drops across the interface between 13°F and 230°F for various configurations. The low ΔT design, incidentally, had to be discarded because of poor dielectric characteristics. When it is considered that a reduction in generator cold end temperature drop of 10°F is equivalent to approximately 2.1 pounds in 500 watt RTG weight or 0.1% improvement in RTG efficiency, the importance of the ΔT minimization effort becomes very clear. For reference, the vacuum ΔT of the current SNAP 19 button-piston-spring-bar design is approximately 240°F.

Tests were conducted in a glass bell jar using the test column illustrated between

heater block and cooling manifold (Figure II-12). Heat flow was adjusted based on temperature drop across the titanium alloy shunt which had been calibrated. The operating point was recorded by thermocouples attached to the copper strap. A total of 136 test points were obtained within the limits of parameter variations shown in Table II-1.

In reference to Table II-1, the fixed parameters were selected to be typical for a LCHP generator design. For different design values of axial heat flow, the interface ΔT may be assumed to be directly proportional to axial heat flow, and the effects of shifts in nominal operating temperature - unless they produce changes in compliance between the contacting materials - are probably negligible for the selected configuration.

The majority of data was obtained at an interface contact pressure of 300 psi, although a few higher pressures and a series of lower pressures were tested to evaluate effect of spring loadings on promising designs. The final configuration was characterized in helium, argon, and in vacuum. Tests with specimens of two different contact area were conducted to assess effects related to size such as contact surface flatness and edge effects.

Candidate materials for the contact surface were selected to provide a strong dielectric barrier. One side of the gap will probably be copper, although current carrying straps could be coated with a more compliant material such as lead or tin. Material compatibility with the selenide thermoelectrics need be considered, as well as relative ease of manufacturing and long term stability in operation.

At the on-set of the test program, the spray-on coatings (Al_2O_3 , BeO) appeared to be promising candidates for the dielectric barrier. Flame-sprayed Al_2O_3 was received with a surface finish of 250 $\mu\text{in.}/\text{in.}$ RMS, plasma-sprayed Al_2O_3 with 120 RMS and the BeO parts were approximately 85 RMS. Thermal resistance between the contact surfaces

FIGURE II-12

INTERFACE ΔT TEST SETUP

TABLE II-1

INTERFACE ΔT TEST PARAMETER IDENTIFICATIONA. Fixed Parameters

Axial heat flow	53 watts/in. ²
Test temperature (copper strap)	390°F

B. Variables

Interface contact pressure	10-800 psi
Bell jar gas pressures	1×10^{-4} torr to 13 psia
Bell jar gases	helium and argon
Interface contact areas	2.6 cm ² and 16.4 cm ²

C. Interface Materials

Al - Cu	Al was 6061-T6, Cu oxygen free
Al ₂ O ₃ - Cu	Al ₂ O ₃ was applied to Al
Al ₂ O ₃ - Sn - Cu	by plasma spray and flame
Al ₂ O ₃ - Sn	spray processes.
Al ₂ O ₃ - TFE - Cu	Teflon was used as thin sheet,
Al (hardcoated) - Cu	and as a coating on hardcoated
TFE - Cu	aluminum.
Al ₂ O ₃ - Pb - Cu	
Al - BeO - Cu	BeO was plasma sprayed, or
BeO - Cu	used in form of 20 mil metal
	plated discs.

in vacuum decreased dramatically with surface finish, ranging from 230 °F to approximately 50 °F. Also, the coarser specimens were quite sensitive to applied interface load levels, and ΔT generally improved with time. The hard, rough oxide managed to penetrate the copper to some extent thereby increasing actual contact area and heat flow. Microscopic study of the mating surfaces suggested heat transfer effective contact area below 10% in the Al_2O_3 -coated specimens and perhaps 20% in the BeO_2 -sprayed parts. These observations led to attempts to improve surface contact area as a means to lower thermal resistance. Progress, however, was stalled by such obstacles as coating porosity and specimen edge recession. Also, the copper strap appeared to deviate from perfect flatness which coated specimens with finishes of 40 RMS or better seemed to require. The best Al_2O_3 ΔT was approximately 60 °F using finishes between 15 RMS and 40 RMS. The much thinner BeO coating had tendencies to flake but ΔT 's were down to 40 °F for a 70 RMS finish, possibly aided by flaking. It was at that point that conformity layers were introduced with success. Reductions of ΔT to approximately 25 °F were achieved using tin which was melted into the gap. Teflon film and lead foil looked promising also. But a problem arose from the use of metallic conformity material, the deterioration of the dielectric barrier. Sectioning of an Al_2O_3 /tin test button revealed impregnation of tin into the porous coating and some tin ridging at outside edges of the specimen. Either event could bridge the dielectric, making this configuration unsuitable. The concept of a conforming barrier, however, was pursued further with a teflon impregnated, "hard coat" aluminum specimen, made by Dupont process which also produces the "Non-Stick-Cookware." These specimens limited gap ΔT in vacuum to less than 30 °F at contact pressures down to 100 psi. A parallel development, the bonding of a 20 mil thick BeO wafer to the test cap also produced favorable vacuum gap ΔT 's at contact pressures in excess of 150 psi.

Test results of two satisfactory cold end hardware dielectric barrier gap configurations, the teflon-coated aluminum hardcoat specimen, and the beryllia oxide wafer specimen are shown in Figures II-13 and II-14. With reference to Figure II-13, the effect of contact pressure on gap ΔT is minimal above 150 psi. At 800 psi contact pressure, the gap ΔT was approximately 10 °F for both configurations.

Additional tests were conducted on the beryllia wafer configuration to characterize gap ΔT as a function of gas pressure (see Figure II-14). Using contact pressures of approximately 200 psi and gas environment pressures between 0.01 mm and 700 mm Hg helium and argon, gap temperatures were seen to be most sensitive to pressures in the range between 0.5 psia and 5 psia and rather insensitive at pressures below and above that range. The ΔT of the gas test specimen at 200 psi contact pressure in vacuum (10^{-4} mm Hg) was 21 °F.

Both of the final dielectric barrier gap configurations, the TURFRAM* TFE process coating and the bonded beryllia wafer approach appear optimized for low thermal resistance. The beryllia wafer configuration was selected for the flexible sleeve cold end hardware for these reasons:

- (1) Low moisture and contaminant absorptivity.
- (2) High dielectric strength
- (3) Possible application as hot end dielectric

A procedure to conduct the cold end ΔT tests is identified in Reference 1. A topical report which further details the test effort will be released as shown in Reference 2. Preliminary test results were released per Reference 3.

*DuPont process licensed to General Magnaplate Corp., Linden, N.J.

FIGURE II-13
TEMPERATURE DROP ACROSS CONTACT GAP
VERSUS CONTACT PRESSURE
FLEXIBLE SLEEVE COLD END HARDWARE DEVELOPMENT

FIGURE II-14
TEMPERATURE DROP ACROSS CONTACT GAP
VERSUS COVER GAS PRESSURE

b. Cold End Hardware Module Test

Components of the thermal interface ΔT test fixture (LCHPB0160000-009) will be utilized to obtain pertinent data regarding the mechanical design, material compatibility, temperature gradient and electrical isolation capability of the cold end hardware flexible sleeve concept on a module basis.

The basic test fixture, illustrated in Figure II-15, consists of two (2) major sub-assemblies, a compression plate assembly and a heater block/shunt assembly. The compression plate assembly which simulates the exterior housing wall of a typical LCHPG consists of an aluminum plate with sixteen (16) instrumented flexible sleeves with springs, and a controllable cooling line (H_2O). The heater block/shunt assembly which simulates the internal components (thermoelectrics, heat source and insulation) of a typical LCHPG consists of a heater block with two (2) 200 watt heaters, sixteen (16) instrumented simulated thermoelectric elements (8 "N" legs and 8 "P" legs) and appropriate Min-K type 1301 insulation. In all, there are 57 thermocouple (CHR/AL) measuring points which will be utilized to monitor temperatures throughout the various phases of the test.

All aspects of the test with the exception of the mechanical installation (compression and measurement of the flexible sleeves i.e. assembly of the compression plate with the heater block assembly) will be conducted in a vacuum with an appropriate cover gas. A procedure to conduct these tests is identified in Reference 4.

C. HOT SIDE DIELECTRIC

The projected LCHPG "hot junction" temperatures, 800-1000°C, exceeds the temperature capability of "mica type" materials and necessitates the selection of a stable oxide ceramic material for the hot side dielectric material. Candidate ceramic oxides include BeO , Al_2O_3 and ZrO_2 .

Development of an integral ceramic oxide dielectric/hot shoe (molybdenum) com-

FIGURE II-15
FLEXIBLE SLEEVE COLD END HARDWARE EVALUATION TEST FIXTURE

ponent, in contrast to a separate dielectric member, offers two distinct advantages: (1) reduced ΔT due to the elimination of a mechanical interface, and (2) simplification of the assembly process by reduction of the number of piece parts. This approach is a logical extension of the LCHPG "cold end hardware" technology (metallized BeO wafer soldered to aluminum alloy substrate) as discussed in Section II-B.

Based on a preliminary review of ceramic/metal joining (Ref. 5, and discussions with J. E. Coureforo, Consolidated Ceramics and Metalizing Corp., Hampton, N.J.) and of oxide ceramic coating technologies, a feasibility study of manufacturing processes for an integral ceramic oxide dielectric/hot shoe appears warranted. The primary developmental problems include: (1) joint thermal and mechanical stability, and (2) compatibility of materials within the converter section. Three technical approaches are recommended for development and evaluation: (1) metallized solid oxide brazed to the hot shoe, (2) plasma sprayed oxide on hot shoe substrate, and (3) chemical vapor deposition of the oxide on the hot shoe substrate. Oxides to be evaluated should include BeO and Al_2O_3 .

D. CONOSEAL HEAT SOURCE CAVITY CLOSURE TEST

LCHPG on-pad or in-field fueling capabilities may be desirable or even necessary for certain mission applications. To provide this capability would require a simple closure procedure for the heat source cavity among other things. Preliminary design and test efforts were conducted to accommodate the conoseal closure mechanism, an Aeroquip system consisting of a metal ring gasket seal and precision grooved diagonal mating flanges which are tightened by a tension bolt on a spring clamp. A procedure was written (Ref. 6) and a fixture built to conduct sealing evaluation and seal torque tests using Conoseal #50888-400, a sealing assembly for a 4-inch circular fuel port. The

intent was to thermally cycle the seal assembly between 525 °F and 200 °F and to monitor helium leakage during cycling, and for a period of 30 days at approximately 500 °F following cycling.

Due to inability to effect a seal with the vendor supplied stainless steel seal rings at rated torques of 140 in.-lbs, the Conoseal closure approach was suspended. It was concluded that a development effort would be required (seal ring materials, flange lubricants) before the conoseal can be used with confidence in this application. Typical helium leakage rates measured before and after vendor re-work of the sealing flanges were 10^{-3} scc/sec and greater.

E. FUELING CAVITY FRICTION TESTS

A test procedure was developed (Ref. 7) and a fixture designed for the evaluation of friction coefficients at elevated temperature. The material combinations of interest would be those suitable for use in the fueling cavity and in sliding contact during the fueling/defueling operations of LCHPG, a noble metal and graphite. The metal will be that of the isolation can whereas the graphite is material used for reentry protection of individual heat sources. To date, friction tests were completed for a piece of 3-D graphite (in a plane perpendicular to "Z") on a sheet of platinum-30 rhodium.

The method for evaluation of static friction coefficients was to slowly incline a plane until the graphite block broke from the metal base surface. The degree of inclination was indexed with the tangents of the angles, and the plane could be locked at any position. Kinetic coefficients were recorded when the plane was balanced such that an induced slide of the block would continue without acceleration. The slide plane and block could be heated to temperatures of 1400 °F and the entire test was performed in a glove box in argon. Test parts were thoroughly outgassed to maximum operating tempera-

tures before the tests. Oxygen content and water vapor levels in the test environment were held below 10 ppm throughout testing.

Initial results of the heat source cavity materials friction test are shown in Figure II-16. Material surface finishes were as received, between 10 and 20 μ inch/inch RMS. Some microscopic high spots were discovered on the test metal sheet which during continued testing with a Poco graphite specimen, caused surface scratching in both metal and graphite, and coefficients of friction up to 0.5. Based on these results, an effort is in progress to identify the cause and nature of the platinum surface imperfections. Also, the method for removal of high spots will have to be specified and inspection procedures for the heat source isolation can material developed.

FIGURE II-16
COEFFICIENT OF FRICTION DETERMINATION
AYCO 3 D GRAPHITE ON PLATINUM-30 RHODIUM

REFERENCES

1. "Flexible Sleeve Cold End Hardware Evaluation Test," Procedure Dwg. LCHPB0110100.
2. "Flexible Sleeve Thermal Interface Development Tests," TES-SNSO-3-29 (to be released).
3. "Preliminary Report of the LCHPG Flexible Sleeve Cold End Thermal Interface ΔT Test," Memo LCHP-060, D. Wang, 10/18/74.
4. "Flexible Sleeve Cold End Hardware Evaluation Test Procedure," Dwg. LCHPB0160000.
5. "Joining Ceramics and Graphite to Other Material," H. E. Pattee, R. M. Evans and R. E. Monroe, NASA SP-5052, 1968.
6. "Conoseal Evaluation Test Procedure," Dwg. NSK 1373.
7. "Friction Coefficient Test for Graphite on Metal at Elevated Temperature," Procedure Dwg. LCHPB0150100.

III. HEAT SOURCE COMPONENTS

A. HEAT SOURCE MODULE TEST PROGRAM

1. Module Definition and Test Objectives

Early in FY 75 planning was initiated to place fueled heat source modules on test at the Los Alamos Scientific Laboratory for a duration of six months. The objectives of the test program are threefold: to demonstrate heat source component fabrication and assembly; evaluate vent performance and obtain fuel-containment compatibility data. A detailed breakdown of the objectives is presented in the table below.

A. Demonstration of heat source fabrication and assembly

1. Material process development and production
2. Capsule fabrication
3. Weld development
4. Fuel production
5. Heat source assembly
6. Specifications

B. Evaluation of vent performance

1. Vent design and fabrication
2. Establish preassembly vent characterization
3. Diagnostic disassembly of vents including metallography
4. Identify constituents and rate of formation in the vent plugging process
5. Determine helium release rates as a function of time

C. Fuel/containment compatibility and behavior (including stoichiometric effects)

The test program, a cooperative engineering effort with LASL, the Oak Ridge National Laboratory and Teledyne will result in data to support the LCHPG heat source development effort. Figure III-1 provides a heat source technology flow diagram which integrates the test program.

Three basic heat source configurations are to be tested with fuel inventories of 25, 50 and 75 thermal watts as shown in Figures III-2, III-3 and III-4. All test modules consist of a fuel capsule or capsules with supporting components within a heat shield (Figure III-5) in the form of a rectangular parallelepiped. The heat shields and threaded end plugs are fabricated from POCO AXF-Q1 graphite which is a fine grained isotropic graphite. This material was selected for the test modules for economics and availability, the primary heat shield material being a 3-D carbon/carbon composite. *

The fuel capsules are aligned and held within the heat shield with supports at the ends fabricated from POCO graphite. A threaded heat shield end plug is used as a closure. The 75 W(t) fuel capsule with three fuel pellets has hemispherical ends. End fillers of yttria stabilized ZrO_2 are used to fill the voids (Figure III-6). The $^{238}\text{PuO}_2$ fuel is in the form of pressed and sintered cylindrical pellets of approximately 25 thermal watts. Platinum - 30% Rhodium - 8% Tungsten, an ORNL developed alloy, is the encapsulation material where the thickness is nominally 20 mils. A thin bulkhead of Pt-3008 sheet stock is included at each end of the 75 W(t) capsule to minimize direct contact between the fuel and the ZrO_2 end fillers.

Three helium vent configurations were initially selected for development in support of the test capsules. The LASL developed vent is essentially a sandwich consisting of layers of Pt-30 Rh woven wire mesh separated by Pt-Rh powder. The composite is then pressed and sintered. The Teledyne developed vent is a ceramic vent where a

*Outgassing product comparison tests between the POCO and 3-D carbon/carbon graphites are described in Section C.

TASK IV HEAT SOURCE TECHNOLOGY FLOW

25 W(t) HEAT SOURCE TEST MODULE

Figure III-2

Figure III-3

75 W(f) HEAT SOURCE TEST MODULE

TES-SNSO-3-26

III-6

Figure III-4

FIGURE III-5

TEST HEAT SOURCE GRAPHITE DETAILS:
HEAT SHIELD, THREADED END PLUG AND CAPSULE SUPPORTS

FIGURE III-6

ZrO₂ End Fillers (75 W Capsule)

platinum coated ZrO_2 rod is pressed and bonded into a Pt-3008 housing.

Six heat source test modules, five fueled and one unfueled unit applied as a "control" will be placed on test for a six month or more duration. Three units will be tested in a high purity helium environment which is the anticipated environment of the LCHPG heat source compartment. The remaining three units will be tested in a vacuum which may accelerate reactions and also represents an environment where the helium is lost to space due to a compartment puncture, etc. The "control" heat source may be most effective in evaluating effect of impurities within the encapsulation system, independent of fuel, that may be detrimental to vent performance at temperature. Figure III-7 provides a schematic of the six test modules of interest and illustrates the various concepts with respect to thermal loading, capsule temperature, vent and gas environment. The capsules and their vents for the 50 W(t) modules are oriented as illustrated to obtain one vent operating at a somewhat higher temperature than the other. The temperature of each capsule and heat shield will be monitored with platinum sheathed Pt-Rh thermocouples. Capsule temperature will be controlled at 1000°C for four units and at 1100°C for the other two units. Figure III-8 presents a schedule and associated on-test sequence for the heat source modules.

FIGURE III-7
HEAT SOURCE MODULE CONFIGURATIONS

HEAT SOURCE MODULE TEST SCHEDULE AND SEQUENCE

Each heat source will be housed within a test fixture (Figure III-16) which consists of a double containment of two concentric chambers. Each unit is then inserted into a Mellen type furnace as shown in Figure III-9. The double containment was selected to have a more reliable leak tight chamber in addition to the requirements of nuclear safety. The inner can or chamber immediately enclosing the heat source module is constructed from Inconel 600. The other containment is fabricated from austenitic stainless steel, type 310. The actual test duration for each module will be determined at a later date and will be dependent upon other supporting technology as well as the knowledge gained as each module in turn is diagnostically disassembled and evaluated. Post test evaluations of heat source components will include visual observations, swipe tests, chemical analysis, microstructure examination, microprobe gradients and scanning electron microscope analysis.

FIGURE III-9

MELLEN FURNACE - VACUUM LINE ARRANGEMENT

2. Capsule Material Development

a. Characteristics

Pt-3008 is an ORNL developmental alloy of the nominal composition Pt-30 wt. % Rhodium-8 wt. % Tungsten specifically designed for isotopic fuel capsule applications. At the onset of the Task IV Heat Source Module Demonstration effort, the following alloy characteristics led to its selection as the prime fuel encapsulant/clad material:

- Excellent air oxidation resistance, particularly at temperatures circa 1000°C and below.
- High melting point

Base alloy 2000°C

Pt-3008/carbon eutectic 1840°C^(a)

- Preliminary results of compatibility tests (PuO₂/Pt-3008 and Curia^(b)/Pt-3008) in the 900-1100°C temperature range.
- The portent of reasonable toughness (tensile ductility and strength), particularly in the intermediate temperature range, typically 200-900°C^(c).
- The indication of acceptable alloy producibility and fabricability.

In general, the results of continued studies of the Pt-3008 alloy conducted at ORNL, LASL and DWDL concurrently with this Task IV program have further substantiated the promising characteristics of this alloy. Several of the more significant observations include the following:

(a) R. G. Donnelly, Isotopic Power Materials Development Progress Report for August 1973, October 1973, ORNL-TM-4383, p. 15.

(b) Curia, CmO₂, contains 5 + % PuO₂ as a normal decay product.

(c) The LCHPG modular heat source design philosophy is based on a low ballistic coefficient reentry body, with resultant low terminal or impact velocity and moderate terminal or impact temperature.

- Good compatibility between PuO_2 /Pt-3008/graphite has been observed at temperatures in the range 900-1100°C for times to 8000 hours.
- Thermally aged Pt-3008 (either alone or in contact with PuO_2 fuel or graphite) exhibits a "ductile" fracture morphology and acceptable levels of tensile ductility and strength within the temperature range of ~400°C to 900°C and perhaps beyond.
- The response of Pt-3008 to high strain rate, circa 150 ft/sec., uniaxial deformation is quite unique and beneficial for this application. In the temperature range 400°C-1100°C no degradation in fracture ductility was observed at test velocities of from ~0.05 in/min. to ~150 ft/sec. In fact, at 900°C and 1100°C both strength and ductility were enhanced by a factor of 2X at the higher strain rate.
- The attractive air oxidation resistance of Pt-3008 (based on weight change measurements) was confirmed at 600°C, 800°C and 1000°C for test times of up to 10,000 hours.

The mechanical response, particularly fracture ductility and morphology, of Pt-3008 is very sensitive to minor trace impurities and their interaction (segregation) with grain boundaries. Boron, at levels $> \sim 10$ PPM, has been related to a high temperature ductility minimum, circa 1100°C, and a pronounced impairment of hot workability. Silicon grain boundary segregation has been associated with reduced hot ductility circa 1300°C. In addition to boron and silicon, other trace elements of potential significance in the metallurgical behavior of Pt-3008 include P, Fe, Cr, and Cu. Trace element effects on Pt-3008 is an area of continuing study.

b. Pt-3008 Alloy Producibility

Pt-3008 alloy sheet and bar stock required for capsule piece part and "TES Vent" fabrication were produced by ORNL without difficulty. Typical process flow charts for the fabrication of 0.39 in. diameter bar and 0.020 in. gage sheet are outlined in Figures III-10 and III-11, respectively. Because of limited material availability and prior processing experience melts were limited to a nominal 1 - 1-1/2 pound size. Low boron levels were readily attained by the ORNL "de-boronizing Ar-H₂ arc melt process." The efficacy of the boron removal technique was evidenced by analyzed product boron levels, typically << 5 PPM, and excellent hot workability. Minor problems during arc melting of several heats led to moderate melt contamination with Cu, Fe, Cr and Ni. Product yields and quality were good and amply demonstrated the good producibility of Pt-3008 sheet and bar.

As noted in Figure III-11, two sheet processes were developed to yield two types of sheet product, process route 'A' yielding a "recrystallized" product and process route 'B' yielding a "stress relieved" sheet product.

c. Pt-3008 Alloy Fabricability

Piece parts for both 25 W(t) and 75 W(t) capsule parts were successfully fabricated and/or machined by ORNL. Typical 25 W(t) and 75 W(t) product capsule piece parts are illustrated in Figures III-12 and III-13, respectively.

Fabrication of the tubular capsule bodies was readily accomplished by

- (1) roll forming the tubular body using recrystallized sheet,
- (2) electron beam welding the longitudinal butt weld,
- (3) cold sizing, where required, and grinding to required I.D. and O.D., and
- (4) machining to required length and end shape.

Figure III-10

Pt-3008 0.39 in. Diameter Rod
ORNL Process Flow Chart*
Heat No. Pt-4R

*Data abstracted from ORNL-TM-4756, November 1974.

Figure III-11

Pt-3008 0.020 in. Gage Sheet
Typical ORNL Process Flow Chart

FIGURE III-12

25 W(t) FUEL CAPSULE PIECE PARTS

FIGURE III-13

75 W(t) FUEL CAPSULE PIECE PARTS

A forming problem was encountered in attempting to "deep-draw" the hemispherical dome ends for the 75 W(t) capsule. With the exception of one heat, all attempts to "deep-draw" the dome configuration using "recrystallized" sheet and multiple draw/stress relief cycles were unsuccessful due to surface cracking. The "deep-draw" deficiencies of recrystallized sheet necessitated the development of the "stress relieved" sheet. Dome ends were readily formed using stress relieved sheet and a two step draw with an intermediate stress relief anneal.

All formed and/or finish machined capsule piece parts were acid cleaned and vacuum annealed at 1000°C for one hour prior to final inspection and shipment to LASL.

3. Zirconia/Pt-3008 Alloy Compatibility

An "end-filler" or "crush-up" compliance material, as shown within the heat source of Figure III-4, is employed within the hemispherical dome ends of the 75 W(t) capsule. A yttria stabilized fibrous zirconia material, ZIRCAR board type ZYFB6, ^(a) (60 lbs/ft³ or approximately 16% theoretical density) was selected for this application. End fillers, part Nos. LCHPB0130007-001 and -002, were machined from 1" x 1" x 8" board. Typical components are shown in Figure III-6.

The selection of an oxide ceramic filler material, low density stabilized ZrO₂, ^(b) raised the questions of ZrO₂/PuO₂ fuel and ZrO₂/Pt-3008 compatibilities. The probability of a direct fuel/zirconia end-filler material interaction was alleviated by the introduction of a 0.020" thick Pt-3008 alloy disc between the fuel pellet and the fibrous stabilized zirconia end-fillers. The potential interaction between zirconia and the noble metal Pt-3008 alloy is a real concern. Work by A.S. Darling ^(c), et al (Johnson Matthey & Co., England) has shown that under unique conditions (i.e. a nil oxidizing potential environment, such as "gettered" argon or a dynamic high vacuum) platinum or platinum alloys may react with refractory oxides such as Al₂O₃, ZrO₂ and ThO₂. A MgO/Pt reaction was not observed. The extent or rate of the noble metal/oxide reaction is strongly influenced by such factors as temperature (typically >1100 °C), time, surface and contact area of the reactants, in addition to the oxygen content of the atmosphere.

^(a) ZIRCAR board type ZYFB6 is a product of ZIRCAR Products, Inc., 110 N. Main St., Florida, N.Y. 10921.

^(b) NOTE: Alternate end-filler materials were explored, the prime candidates being "low density MgO" and low density powder or fiber metallurgy platinum alloy compacts. MgO was rejected because of excessive vapor pressure. The low density platinum compact offers attractive potential for impact energy absorption. However, in view of the severe impact this approach has on the heat source system (i.e., high weight and cost penalties and possible deleterious effects from the high surface area of the noble metal contact) continued evaluation of the ZrO₂ end-filler system was recommended.

^(c) A series of articles by A.S. Darling, et al, appearing in Platinum Metals Review: Vol. 14, No. 2, April 1970; Vol. 14, No. 3, July 1970; Vol. 14, No. 4, Oct. 1970; Vol. 15, No. 1, Jan. 1971.

In order to elucidate the question of low density ZrO_2 compatibility with Pt-3008 under potential LCHPG heat source conditions, a test matrix was initiated at ORNL (H. Inouye). Eighteen rectangular samples, nominal $1/16'' \times 3/8'' \times 1\ 1/2''$, of ZIRCAR ZYFB6 board (fibrous yttria stabilized ZrO_2) were provided by Teledyne Energy Systems. Tensile specimens of 0.020 in. Pt-3008 sheet (Heat No. LCPT-4) were employed in the following compatibility test matrix:

<u>Material Combination(s)</u>	<u>Atmosphere</u>
Pt-3008 (control)	Vacuum 10^{-8} torr
Pt-3008/ ZrO_2	Vacuum 10^{-8} torr
Pt-3008/ ZrO_2	Helium 600 torr (static)

In all cases exposure temperature is 1050°C (1922°F). Preliminary data (visual inspection and 1093°C tensile properties at slow strain rate) after 1000 hours exposure are suggestive of a possible slight interaction between the Pt-3008 and ZrO_2 . However, the interaction does not appear to be detrimental. After aging in helium a reduction in strength was noted as opposed to a moderate increase in strength of specimens aged in a vacuum. The observed difference in ductility was negligible. Further tests of the 1000 hour exposed samples and extension of the exposure time (preferably 2000 - 5000 hours) will be required to reach a definite assessment of ZrO_2 /Pt-3008 compatibility.

4. Fuel Form

Each fuel pellet of PuO_2 is nominally 25 watts(t) with an L/D of approximately 0.65. The diameter is $0.898 \pm .005$ inch and the length is $0.586 \pm .005$ inch. Each end of the pellet is chamfered at 45° and 0.06 inch. The chamfer appears adequate to allow space behind the cylinder-end closure weld (specifically for the 25 watt capsules) such that the local heat affected zone of the capsule weld is remote from the fuel. Secondly, the fuel should not readily cut into the encapsulation material when considering a terrestrial impact environment. The gaps designed between fuel and capsule shell are 15 mils on the diameter and 10 mils minimum in the axial direction.

The basic fuel pellet parameters selected for LCHPG heat source module test series is as follows in addition to the dimensions presented above.

Fuel type	- Stoichiometric $^{238}\text{PuO}_2$ (80% enriched)
Fuel form	- Hot pressed cylinder, chamfered edges, L/D of 0.65
Density	- 88 to 90% theoretical density
Nominal grain size	- 10 microns

The development of tooling and process parameters for the production of $^{238}\text{PuO}_2$ LCHPG fuel pellets, per the above cited target parameters, was conducted at LASL under the direction of R. Kent in conjunction with the Plutonia Fuel Technology Program.

Prototype and test LCHPG fuel pellets were processed at LASL in 10 lots, primarily in the September 1974 through January 1975 time frame (prototype lots HPZ-90 and -94, nominal four pellets/lot; and "test" lots, nominal two pellets/lot, HPZ-102, -105, -106, -107, -113 and subsequently HPZ-124 in May 1975). As shown in Table III-1, size control has been excellent. Seven of eight fuel pellet lots are well within the target

TABLE III-1
DIMENSIONAL AND DENSITY CHARACTERISTICS FOR PROTOTYPE
AND TEST LCHPG FUEL PELLETS*

	<u>Sintered Pellet Size and Density</u>			
	<u>Diameter-in.</u>	<u>Length-in.</u>	<u>% Theoretical Density</u>	
Target	0.898 ± .005	0.586 ± .009	88 to 90	
Lot HPZ-90*	0.902 ± .001	0.585 ± .001	88.5	Flow sheet process per Figure III-15
Lot HPZ-94*	0.899 ± .001	0.582 ± .001	89.6	Flow sheet process per Figure III-15
Lot HPZ-98	0.900 ± .001	0.575 ± .001	90.4	Seasoned at 1225°C.
Lot HPZ-102	0.902 ± .001	0.590 ± .001	87.6	Season 32.4 ks at 1200°C plus 32.4 ks at 1250°C
Lot HPZ-105	0.897	0.593	88.3	Season 43.2 ks at 1250°C, ball milled 43.2 ks
Lot HPZ-106	0.901	0.594	87.2	Season 43.2 ks at 1250°C, ball milled 43.2 ks
Lot HPZ-107	0.900	0.580	89.5	Season 43.2 ks at 1250°C, ball milled 43.2 ks
Lot HPZ 113 ^(a)	0.892	0.583	90.8	Season 10.8 ks at 1150°C,
Lot HPZ 113 ^(a)	0.892	0.580	91.4	Season 10.8 ks at 1150°C, sinter 21.6 ks at 1527°C
Lot HPZ-124	Currently (May '75) in process.			

(a) Processed from feed powder lot 107791 processed at SRP in 1971.

* All data was extracted from LASL monthly reports CMB-2126, CMB-2139, CMB-2150, CMB-2164 and CMB-2176.

dimensional limits. In contrast, the percent theoretical density for these eight lots covered a span of 87.2% to 91.4% versus a target of 88-90%. Four of the eight lots met the target density range. Pellet quality as evidenced by visual examination was good for all fuel pellet lots. The occurrence of surface craze cracks was negligible. A typical LCHPG fuel pellet is shown in Figure III-14.

After fabrication fuel pellets were stored in closed containers at ambient temperature. Estimated pellet temperature during storage is 500-600°C. As of May 1975 no visual degradation of the stored fuel pellets has been noted. Selection of specific fuel pellets for encapsulation in the June-July 1975 period shall be made by LASL.

The typical process sequence and nominal process parameters employed by LASL in the production of LCHPG fuel pellets are illustrated in Figure III-15. The occurrence of a change in the characteristics of the SRP feed material, first noted in feed lots processed at SRP in July 1973, significantly altered powder particle size control, powder reactivity and hence hot pressing characteristics and density control. As noted in Table III-1, changes in pre-hot-press process parameters, primarily "seasoning temperature," were introduced in order to improve density control and alleviate this interim problem.

Spectrochemical analytical data for several LCHPG "test" fuel pellet lots and respective SRP feed material lots are summarized in Table III-2. Inspection of these data yield the following observations:

- Major impurities common to both the SRP feed material and the LCHPG fuel pellets include Al, Ca, Fe, Ni and Si together with somewhat lower levels of Cr, Na, Pb and Ta.
- No significant systematic change in impurity levels during fuel pellet fabrication are apparent.

LASL

HPZ . . 90 . . 2

AFTER 1350 C HEAT TREAT

1 CHP PROTOTYPE

FIGURE III-14

25 W(t) PROTOTYPE FUEL PELLETS

FIGURE III-15

LCHPG FUEL PELLET - 24 W(t) L/D 0.65
TYPICAL LASL PROCESS FLOW CHART

TABLE III-2

SPECTROCHEMICAL DATA FOR LCHPG TEST FUEL PELLETS
AND PERCURSOR SRP FEED LOTS

Species	Spectrochemical Data* - PPM by Weight							
	SRP Lot 401131	Pellet Lot HPZ-94	SRP Lot 404199	Pellet Lot HPZ-98	SRP Lot 402141	Pellet Lot HPZ-102	SRP Lot 405204	Pellet Lot HPZ-105
Al	220	140	75	180	100	55	40	80
B	1	1	< 1	< 1	2	< 1	< 1	< 1
Ba	< 4	35	< 4	4	< 4	10	< 4	4
Be	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
Bi	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
Ca	140	370	190	110	160	190	180	170
Cd	< 4	< 4	< 4	< 4	< 4	< 4	< 4	< 4
Co	< 2	< 2	< 2	< 2	3	< 2	< 2	< 2
Cr	60	70	30	30	140	35	20	55
Cs	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20
Cu	6	6	10	3	8	4	5	4
Fe	450	260	220	130	570	190	130	< 210
Hf	< 4	< 4	< 4	< 4	< 4	< 4	< 4	< 4
K	7	15	15	4	< 4	7	4	4
La	< 4	< 4	< 4	< 4	< 4	< 4	< 4	< 4
Li	< 4	< 4	< 4	< 4	< 4	< 4	< 4	< 4
Mg	15	20	20	4	3	9	20	15
Mn	6	10	6	3	15	10	3	8
Mo	3	6	6	< 2	< 4	5	< 2	8
Na	220	< 50	50	130	< 50	< 50	< 50	< 50
Nb	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Ni	100	80	40	25	140	60	50	70
Pb	100	90	70	15	55	15	35	< 4
Rb	< 4	< 4	< 4	< 4	< 4	< 4	< 4	< 4
Re	< 4	< 4	< 4	< 4	20	7	< 4	4
Si	120	170	200	170	410	350	90	240
Sn	4	50	4	< 4	10	< 4	< 4	4
Sr	< 4	15	< 4	15	30	70	< 4	20
Ta	<100	90	<100	220	<100	260	<100	270
Ti	7	10	10	6	9	10	6	8
V	< 2	< 2	< 2	< 2	< 2	< 2	< 2	< 2
W	< 10	< 10	10	< 10	< 10	< 10	< 10	< 10
Y	< 4	< 4	< 4	< 4	< 4	< 4	< 4	< 4
Zn	35	8	20	4	30	5	9	5
Zr	4	4	10	< 4	< 4	4	7	4

*Data extracted from LASL Monthly Reports CMB-2139, CMB-2150, CMB-2164 and CMB-2176

5. Thermal Mockup Experiments

Prior to placing PuO_2 fueled heat source modules on test, it was initially planned to fabricate an additional test chamber to receive an electrically heated module. With this prototype apparatus and numerous thermocouples, the temperature of heat source components and test apparatus components could be obtained as a function of heater output and furnace output. Secondly, these tests would be conducted with helium or a vacuum within the test apparatus.

The test apparatus is of a double shell construction as shown in Figure III-16. Reference is also made to Teledyne drawing LCHPB0150000, "Test Fixtures - Details and Assemblies." The electrically heated capsule assembly of Figure III-17 (dwg. LCHPB0150001) was fabricated from Pt-3008 shells of .020 in. thick and contained a 120 V, 200 W Watlo heater. A nickel 200 sleeve was assembled between the heater and the capsule cylindrical shell to provide an efficient conductive heat path.

The instrumentation consisted of eight (8) thermocouples attached to the following locations:

- Two thermocouples attached to the test apparatus inner can.

- Three thermocouples attached to the capsule outer surface.

- Three thermocouples attached within the graphite heat shield.

An electrical schematic of the thermal mockup is shown in Figure III-18.

The thermal mockup was assembled and placed on test at LASL where the initial environment within the apparatus was high purity helium gas. Without applying external heat with the Mellen furnace, the heat source was tested at 25, 50 and 75 watts (t). The temperature was allowed to stabilize at each thermal level. The furnace was then turned on and during the excursion the capsule heater shorted out. When allowed to cool down the heater indicated a positive electrical continuity. However, an electrical short always occurred at elevated temperature suggesting that the short may have resulted from

Inner Can: Inconel 600

Outer Can: 310 SS

FIGURE III-16

FIGURE III-17

ELECTRICALLY HEATED CAPSULE : 75 W(t) CONFIGURATION

FIGURE III-18

ELECTRICAL SCHEMATIC : THERMAL MOCKUP

differential thermal expansion. However, thermocouple data was obtained at the above power increments. Figure III-19 presents a plot of capsule, heat shield and inner can temperatures as a function of thermal power input.

Future application as a thermal mockup for this test apparatus has been discontinued. Therefore, similar data with a vacuum environment within the inner chamber will not be obtained at this time. However, during the next phase of the program a thermal mockup will be designed, fabricated and tested to further thermal transport research. This is particularly important with respect to a vacuum environment. No conductive gas is available. Hence, the heat transfer is primarily by radiation and some conduction depending on the degree of component to component contact.

Assuming a capsule emissivity of 0.16 obtained from previous applications with platinum-rhodium alloys, a thermal analysis was conducted to correlate with the measured temperatures at the various thermocouple locations. The results compared favorably. The measured and analysis predicted temperatures for the capsule and inner can are presented in Table III-3.

TABLE III-3

<u>Power Level (watts)</u>	<u>Measured Capsule Temp. (°C)</u>	<u>Predicted Capsule Temp. (°C)</u>	<u>Measured Inner Can Temp. (°C)</u>	<u>Predicted Inner Can Temp. (°C)</u>
74.3	443	453	315	312
48.0	341	342	242	240
24.5	171	175	114	110

FIGURE III-19THERMAL MOCK-UP HEAT SOURCE TEST

HELIUM ENVIRONMENT III - 152 TORR

The following thermal transport properties were applied in the above heat transfer analysis:

Capsule to graphite:

Effective emissivity = 0.154

Helium conductivity = 0.15 Btu/hr-ft-°F at 75 W level

= 0.14 at 48 W level

= 0.12 at 24.5 W level

Gap width = 0.050 inch

Graphite to Inner Can (Inconel):

Effective emissivity = 0.552

Helium conduction = same as above

Gap width = 0.250 inch

B. VENT DEVELOPMENT

1. Design

The objective of a fuel capsule vent system is to vent the helium that is produced from alpha decay within the fuel. To contain this helium for long term space power missions, the fuel capsule would either have to be abnormally thick to withstand the pressure or a significant void volume would be required within each capsule. The second function of the vent is to eliminate the passing of fuel particulates.

To date, twelve (12) helium filter vents of the Teledyne design have been fabricated and flow checked. This design, nearly identical to the vents used for the SNAP 19 Nimbus application, consists of a calcia stabilized zirconia rod within a Pt-3008 housing as shown in Figure III-20. Five vent assemblies were fabricated where the cylindrical vent was nominally 0.090 in. in length. Two of these assemblies were shipped to LASL in support of the 75 W(t) test capsule. The remaining vent assemblies fabricated were made with a vent length of 0.150 inch. The diameter of all vents are .037/.031 inch. One vent assembly of each length vent was shipped to Battelle Columbus Laboratories for evaluation of potential plugging with their test apparatus.

The vent receives a coating of pure platinum followed by machining to length and then inserted into the housing with a press fit. The assembled filter is finally diffusion bonded by means of a vacuum furnace (1100-1400°C at $< 5 \times 10^{-4}$ micron pressure). Figure III-21a shows the cross section (50 x) of a vent assembly, whereas, Figure III-21b is a 500 x micrograph which clearly illustrates the grain structure of the diffusion bonded vent/housing interface.

FIGURE III-20

LCHPG FILTER VENT (75 W(t) CAPSULE)

FIGURE III-21
HELIUM VENT MICROGRAPHS

FIGURE III-22
FILTER PLATING TOOL

FIGURE III-23

FILTER VENT ALIGNMENT AND INSERTION TOOL

The puncture disc is also fabricated from Pt-3008 and is welded into the assembly prior to the vent assembly-capsule end cap weld. At this point in the heat source assembly, the puncture disc has an offset hole of .017/.013 in. in diameter. Following piece part assembly, the vent is flow checked to determine whether or not the weld heat has any detrimental effect. The hole in the disc is then welded closed prior to capsule assembly to allow for capsule surface decontamination. Following capsule assembly and decontamination the puncture disc is punctured with a small tool.

Reference documents and drawings to manufacture the vent assembly are tabulated below.

LCHPB0130006	Filter Vent
LCHPB0110011	Puncture Disc - Filter Vent
LCHPB0120012	Element - Filter Vent
NSK 1426	Test Elements - Filter Vent
LCHPB0140004	Plating Tool
LCHPB0140005	Alignment and Insertion Tool
LCHPB0140006	Cold Flow Test Fixture
LCHPB0140008	Element Holders - Capsule Vent

All 25 W(t) fuel capsules will include the LASL designed vent which consists of pressed and sintered alternating layers of woven wire (Pt-30 Rh) mesh and Pt-Rh powder. Figure III-24 provides 50 x and 100 x photomicrographs of the vent surface. This vent configuration is particularly attractive for fuel capsules of flat end closures due to the vent's thin wafer geometry.

100X

50X

FIGURE III-24

LASL VENT DESIGN (FOR 25 W(t) FUEL CAPSULES

An experimental apparatus has been developed at BCL to evaluate vents (either selective or nonselective). Over a relatively short period of time, vent plugging can be studied from vapor deposition and migration mechanisms with an emphasis on phenomena kinetics. The ultimate goal of the experimentation is to predict long term behavior.

The remaining seven vent assemblies not committed to the tests previously discussed will receive various thermal environments simulating reentry heating of various temperature histories. These vents will then be helium flow checked and sectioned for metallurgical evaluation.

2. Vent Flow Tests

Following fabrication, twelve (12) vent assemblies (dwg. LCHPB0130006) have been helium flow tested per LCHPB0130100, "Capsule Vent Cold Flow Tests." As discussed in the previous section the initial five (5) vent assemblies were manufactured per the above referenced drawing where the ZrO_2 vent was nominally 0.090 in. in length. The remaining seven (7) vent assemblies incorporated a ZrO_2 vent of 0.150 in. in length. All vent housings were fabricated from Pt-3008 rod supplied by ORNL.

The flow tests were conducted using a filter vent back pressure of 1.0 atmosphere air and an upstream helium pressure of 20 psia. A flow test apparatus schematic is provided by Figure III-25 where a bubble-o-meter is utilized.

Two additional tests were set up for vent S/N 6 where a known mass of helium was vented at backpressures of 1.0 atmospheres air and vacuum. By monitoring upstream pressure decay from a fixed volume with time the helium mass flow in scc/sec could be plotted as a function of vent upstream pressure and pressure drop across the vent. From this data it was seen that the helium flow is proportional to the upstream pressure P_1 and ΔP such that

$$Q \text{ (flow rate)} \cong 0.2 P_1 \Delta P \times 10^{-2}$$

LEGEND

- A - Vent Test Fixture (LCHPB0140006-009)
- B - Vent Assy (LCHPB0130006-009)
- 1 - Pressure Regulator (0-60 PSIG)
- 2 - Filter (3-10 Micron)
- 3A - Pressure Gage (0-60 PSIG)
- 3B & 3C - Pressure Gages (0-100 PSIA)
- 4A thru 4E - Shut Off Valves
- 5 - Bubble-O-Meter
- 6 - Vacuum Pump
- 7 - Sample Cylinder/ Reservoir

FIGURE III-25

COLD FLOW TEST DIAGRAM

for flow rates up to 30×10^{-2} scc/sec. At higher flow rates, say up to 80×10^{-2} scc/sec,

$$Q = 0.1 P_1 \Delta P \times 10^{-2}$$

Table III-4 presents the room temperature helium flow rate data for the twelve vent assemblies.

From the flow rate data at 1.0 psia to 14.6 psia, the average flow rate for the 0.150 in. vent is 2.07×10^{-2} scc/sec, whereas for the 0.090 length the average flow rate is nearly identical to the inverse ratio of length. However, individual vent flow rate deviations from the mean are recognized.

Figure III-26 illustrates the vent flow test apparatus at LASL. The associated schematic diagram is presented in Figure III-27. This laboratory equipment has been used in the development of the LASL vent and is employed for measuring helium flow rate through vents for pre- and post test activity.

C. GRAPHITE OXIDATION EXPERIMENTS

During the last month of this reporting period, specimens of a 3-D carbon/carbon composite and POCO AXF Q1 will be tested at various temperatures to obtain oxidation rate in air and to identify outgassing products. Two procedures will be incorporated. One method applies a Marshall tube furnace where oxidation rate will be determined as a function of temperature, specimen orientation (since the composite is highly orthotropic), pressure and air flow rate. The second technique employs a DuPont 990 Thermal Analyzer and a 951 Thermogravimetric Analyzer.

TABLE III-4

<u>Vent S/N</u>	<u>Flow Rate x 10⁻² scc/sec</u>		<u>Fuel Capsule ΔP (psi) for steady state helium release of 0.3 x 10⁻⁵ scc/sec.</u>
	<u>1.0 psid to 14.6 psia</u>	<u>1.0 psid to vacuum</u>	
2	4.2	0.29	0.03
3	4.4	0.30	0.03
5	1.9	0.13	0.05
6	3.6	0.24	0.04
8	2.5	0.17	0.04
10	2.1	0.14	0.05
12	1.8	0.12	0.05
13	2.4	0.16	0.04
14	1.5	0.10	0.05
15	2.9	0.20	0.04
21	2.0	0.14	0.05
24	2.2	0.15	0.04

NOTES:

- (a) S/N 2 was sectioned for metallography.
- (b) S/N 5 and 15 were shipped to LASL in support of heat source module tests.
- (c) S/N 6 and 21 were shipped to BCL for further testing.
- (d) Remaining units designated for additional flow checks after exposure to temperature/time.

FIGURE III -26

LASL VENT FLOW TEST APPARATUS

FIGURE III-27

SCHEMATIC DIAGRAM OF THE LASL VENT-FLOW TEST APPARATUS

The test specimens shown in detail in Figure III-28 are fabricated from a billet of 3-D carbon/carbon (PN 483B1) obtained from Fiber Materials, Inc., Biddeford, Maine). This particular billet is manufactured from layups or weaving of Thornel 400 yarns with two (2) yarns per bundle in the X, Y and Z directions. The Z-spacing per bundle is 0.030 inch. This matrix was then impregnated with resin followed by carbonization and graphitization processing. The resulting density was 1.90 gm/cc. Prior to testing the specimens they are outgassed in a vacuum per procedure specification, PS0200008, with the exception that the maximum temperature is 1250°C with a 3 to 3 1/2 hour hold time.

The Marshall tube furnace and associated test hardware is shown in the schematic of Figure III-29. The apparatus includes a tube of Vycor which permits maintenance of a pressure as low as one torr during operation at temperature. A regulator permits air flow through the system. For this series of tests the following temperature, pressure and air flow ranges will be applied:

pressure: 0.1 - 1.0 atmospheres

temperature: 900 - 1700°F

air flow: 0.1 - 4.0 liters/minute

The specimens will be measured and weighed prior to and after testing.

The thermal analyzer including a programmer and recorder and the thermogravimetric analyzer (TGA) is an extremely sensitive instrument with the capability of measuring weight variation of a microgram or less. The small cylindrical specimens of Figure III-28 are suspended from gold wire loops within the apparatus. Air flow velocity and temperature are controlled to the desired levels. The anticipated accuracy is as follows:

FIGURE III-28
3D CARBON-CARBON OXIDATION TEST SPECIMENS

FIGURE III-29
TUBE FURNACE TEST SETUP

Temperature:	$\pm 1\%$
Pressure:	± 10 mm
Air flow rate:	$\begin{matrix} + 5 \\ - 0 \end{matrix}$ cc/min.

D. HEAT SOURCE THERMAL MOCKUP

During the next phase of the LCHPG program a thermal mockup test chamber will be designed and manufactured to facilitate full scale heat source testing in various operational environments. The apparatus will provide direct measurement of temperatures and temperature gradients through heat source components for steady state operations. This testing capability will be instrumental in obtaining temperature drops across gaps (either from design and manufacturing tolerances and/or differential thermal expansion), evaluating true heat transfer paths including the combination of radiation and conduction, and evaluating the behavior of potential capsule coatings. This capability may be most valuable with a vacuum environment where regardless of a gap dimension some percentage of heat transfer must be attributed to conduction from local contact of components. This is a difficult assignment if only analytical procedures are available. The test capability will therefore include both vacuum and inert gas environments.

The design of the apparatus conceptually illustrated in Figure III-30, will be configured to receive a significant number of thermocouples and associated measurement devices. Heat source concepts and configurations will then be tested per the objectives stated above.

LEGEND

1. CONTAINER
2. COVER
3. O-RING SEAL
4. BOLT & NUT
5. HEADER
6. TUBE
7. VACUUM PUMP CONNECTOR
8. CAPSULE & HEATER
9. HEAT SHIELD
10. INSULATING TUBE
11. INSULATING BEADS
12. LOWER INSULATION
13. UPPER INSULATION
14. SET SCREW
15. HEATER POWER LEADS
16. T/C WIRE

FIGURE III-30
THERMAL MOCKUP

E. ACKNOWLEDGEMENT

The major contributions of LASL and ORNL personnel to the planning and technology leading to a successful heat source test program is gratefully acknowledged.

F. BIBLIOGRAPHY - DRAWINGS AND MEMOS

BIBLIOGRAPHY - DRAWINGS

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LCHPB0130003	Assemblies - 50W(t) Heat Source Demonstration Modules
LCHPB0130002	Assemblies - 25W(t) Heat Source Demonstration Modules
LCHPB0130009	Assemblies - 25W(t) Fire and Impact Test Modules
LCHPB0150000	Test Fixtures - Details and Assemblies, Heat Source Demo Modules
LCHPB0130006	Filter Vent - 75W(t) Fueled Capsule
LCHPB0130011	Puncture Disc - Filter Vent
LCHPB0130012	Element - Filter Vent
LCHPB0140004	Plating Tool, Capsule Vent
LCHPB0140008	Element Holders - Capsule Vent
LCHPB0140005	Alignment and Insertion Tool - Filter Vent
NSK1426	Test Elements, Filter Vent
LCHPB0140006	Cold Flow Test Fixture - Filter Vent
LCHPB0130007	Fillers, End - 75W(t) PPO Fueled Capsule
LCHPB0130008	Miscellaneous Details - Heat Source Demonstration Modules
LCHPB0150002	Heater Test Module Assembly
LCHPB0150001	Heater Test Capsule Assembly
LCHPB0130004	Assembly - 75W(t) Heat Source Demonstration Module
LCHPB0130400	Test Heat Source Module Specification - LCHP Program
LCHPB0130100	Capsule Vent Cold Flow Tests
LCHPB0130001	Fueled Capsule Assembly, 75W(t)
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