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Heat Source Component Development Program

QUARTERLY REPORT FOR JANUARY-MARCH, 1977

Report Date: April, 1977

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Battelle

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HEAT SOURCE COMPONENT DEVELOPMENT PROGRAM

Quarterly Report for January-March, 1977

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Prepared for the Division of Nuclear Research and Applications,
U.S. Energy Research and Development Administration
Under Contract No. W-7405-eng-92, Task 94

Report Date: April, 1977

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REA

FOREWORD

This is the third in a series of quarterly reports describing the results of several experimental programs being conducted at Battelle-Columbus to develop components for advanced radioisotope heat source applications. The heat sources will for the most part be used in advanced static and dynamic power conversion systems. These reports replace the informal monthly technical letter reports previously prepared and are being utilized so that more cohesive presentation of results can be achieved. In addition, a series of summary management monthly reports was initiated in July of 1976 to permit NRA assessment of contractual progress. Previous quarterly reports are identified as BMI-X-672 October 1976, and BMI-X-676 January 1977.

The activities covered in this new series of reports are conducted under Contract No. W-7405-eng-92, Task 94. The specific components development efforts which are described herein are: improved selective and nonselective vents for helium release from the fuel containment; an improved reentry member and an improved impact member, singly and combined. The unitized reentry-impact member (RIM) is under development to be used as a bifunctional ablator. Finally, thermochemical supporting studies are reported.

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HEAT SOURCE COMPONENT DEVELOPMENT PROGRAM

IMPROVED IMPACT MEMBER

(W. H. Duckworth - Task Leader)

Introduction

The impact-member work is directed to gaining needed knowledge and understanding of the response of materials and designs to heat-source impact conditions, and applying the technology gained to the selection or development and demonstration of preferred impact-member materials and designs that will insure fuel containment in the event of earth impact.

Considerable attention was given during the quarter to designing a program on impact protection materials and measures to be incorporated in LASL's GPHS module design. Initial concepts for the direction of effort on the problem were evolved in meetings with LASL personnel, and a tentative work plan has been developed. Briefly, the plan calls for execution of four major tasks involving design and analytical studies, materials development and testing, and impact performance evaluations, utilizing the technology acquired in our studies for ERDA-NRA of the impact member problem.

Summary

During the quarter, progress continued on assessing and interpreting properties of carbon-base materials pertinent to their performance in impact-member service. Specifically, a special apparatus was designed and built for investigating the constraint provided by the materials to lateral fuel deformation, and another special apparatus was completed and debugged for obtaining load-deformation data at loading rates anticipated in the event of earth impact. Performance data for selected materials will be obtained from both apparatus next quarter.

Further progress was made on establishing the applicability of the HONDO computer program for dynamic analyses in support of our work on impact-member materials. In addition, work was completed on programming a mathematical response model of a laminar carbon composite for insertion in the HONDO program.

Work This Period

Development of Preferred Impact-Member Materials (G. K. Bansal)

In the work to date on impact-member materials, attention has been given to evaluating and interpreting the tendency of carbon-base materials to crush and absorb energy. It is recognized that the tendency to crush will be enhanced and the amount of energy absorbed during impact could increase with tensile constraint of lateral fuel deformation afforded by the impact member. It is possible that such constraint is an important factor in the good performance shown by golf-ball wound carbon composite impact shells in service-type impact tests, although the major factor is thought to be their high energy absorption when crushed.

Accordingly, attention is now being given to evaluating and interpreting the tensile constraint of golf-ball wound carbon composite impact members. Previously, to evaluate crushing strength and energy absorption, the testing method used consisted of applying a crushing load to the impact member with a hardened steel platen. The spherical surface of the impact-member specimen was supported by a mandrel, usually of hardened steel, although various "soft" mandrel materials also have been used. A detailed description of the testing method and the data obtained has been reported in the previous quarterly reports on this program.

During this period, a test method has been developed for evaluating tensile resistance of the impact-member specimens, to provide data for selection of preferred materials systems. This test method consists of applying an internal pressure to the spherical specimen and determining the displacement of the inside and outside surfaces. The test set-up is shown in Figure 1. In this arrangement, the primary load is supplied by the elastomer to the inside surface. Although the specimen is supported by the fixture, no compressive load is applied due to the four spacers which separate the top plate from the base. A lateral support ring provides lateral restraint at the base of the specimen. As the load is applied, the plunger compresses the elastomer insert which essentially acts as a fluid and applies a uniform pressure to the internal surface.

The displacement of the inside surface is measured through a small hole by the movement of a 1-1/6-in. diameter rod. A thin disk

(a) Displacement measurement of external surface relative to the fixture.
 (b) Displacement measurement of internal surface relative to external surface.

FIGURE 1. TEST SET-UP UTILIZING INTERNAL PRESSURIZATION

is attached to the rod and conforms to the inside surface. The movement of the external surface is monitored by a tube concentric with the rod which rests on the exterior surface. This approach allows the measurement of the movement of the external surface relative to the fixture and also the movement of the internal surface relative to the exterior of the impact member.

This test apparatus has been fabricated and initial experiments conducted to verify the feasibility of the approach. The internal pressure-displacement curves for a standard golf ball would impact member (Thornel 50 fiber) are shown in Figure 2. The curve showing the displacement of the external surface indicates that after reaching an internal pressure of approximately 3000 psi, no additional pressure build-up could be obtained and the displacement continued until sufficient deformation had taken place to free the diameter of the impact member from the lateral support ring. The curve which indicates the compression or thinning of the impact member wall during test shows a generally uniform rate of thinning until an internal pressure of about 2700 psi was reached. Whether this was the actual limit beyond which the wall would not thin more or a malfunction in the displacement pick-up will not be known until additional tests are performed. The portion of this curve which is in question is shown by the dashed line.

During the next period tests will be conducted on a number of the experimental and standard impact member specimens previously evaluated by other test techniques. A listing of these impact members is given in Table 1.

Impact Analysis (L. E. Hulbert)

In support of the development of improved materials for impact protection of the RTG fuel capsule, BCL has been investigating the applicability of the HONDO computer program. This program was developed at Sandia Laboratories by S. W. Key to analyze dynamic axisymmetric and plane strain problems.

HONDO uses the explicit time integration approach with "constant strain" quadrilateral elements for the discretization of the model. With

FIGURE 2. INTERNAL PRESSURE - DISPLACEMENT CURVES FOR A STANDARD GOLF BALL WOUND IMPACT MEMBER

TABLE 1. CARBON COMPOSITE IMPACT-MEMBER SPECIMENS TO
BE EVALUATED FOR TENSILE CONSTRAINT

Fiber Type	Fiber Properties		Fiber Diameter, μm	Precursor	Specimen Density, g/cc
	Longitudinal Modulus, $E, 10^6 \text{ psi}$	Tensile Strength, $\sigma_f, 10^3 \text{ psi}$			
1. Standard, (Therme1 50 WYG130)	57	315	5-9	Rayon	1.18
2. Standard, decrease tension	57	315	5-9	Rayon	1.17
3. Standard, increase tension	57	315	5-9	Rayon	1.26
4. Graphite yarn WYB85	6	90	8.9	Rayon	1.02
5. Thorne1 75 WYL160	78	380	6	Rayon	1.27
6. Thorne1 300 WYB90	33	385	3.7	PAN	1.19
7. Thorne1 75 WYL160 (25%) and Graphite yarn WYB85 (75%)	Mixture -- see above			Rayon	1.13

these two features, it was felt that HONDO could give an independent check of the results obtained by LASL with the modified NONSAP code. In addition the HONDO code has some very useful material options for the modeling of the carbon-carbon composite materials.

A comparison of the HONDO and NONSAP predictions of the impact of a copper ball on a flat steel surface was initiated in 1976. Initial results of the HONDO analyses predicted significantly higher strains than when measured experimentally or predicted by the NONSAP program. A low key investigation into the reasons for the discrepancy has been carried out over the intervening time. The investigation included a study of the effects of element size (model refinement), boundary conditions at the impact face, and the effect of the assumed stress-strain curve.

In the course of the investigation it was learned that a version of HONDO (called HONDOV) incorporating slide-line capabilities had been developed at the Lawrence Livermore Laboratories by J. O. Hallquist. This capability permitted a seemingly more realistic interpretation of the impact boundary conditions than the "Winkler spring" model used in the original HONDO code. In addition, it was clear that the slide line capability was necessary for the analysis of RTG's with carbon-carbon impact members. An additional improvement in the HONDOV code was the incorporation of a triangular element which enhanced the applicability of HONDO to complex shapes.

Although HONDOV has not been publicly released, it was possible (with the help of the ERDA Contract Monitor) to obtain a copy of HONDOV and instructions in its use.

Work in this quarter involved a study of the application of this new HONDO code (HONDOV) to the analysis of the copper ball impact problem. Analyses included comparison of the effects of changing the impact face boundary conditions and the effect of further refining the finite element model using the enhanced modeling flexibility provided in HONDOV. In addition, Dr. Key (HONDO author) agreed to look at the comparisons between HONDO and NONSAP.

As a result of these investigations, it was determined that the primary reason for the differences in the results obtained with HONDO and NONSAP was the difference in the material model used. HONDO incorporated a bilinear stress-strain curve while a curvilinear stress-strain curve was

incorporated in the NONSAP code at LASL. Moreover, the stress-strain curve used at BCL was an early version used at LASL which underpredicted the strain hardening measured in copper at higher strain values. Using a stress-strain curve more nearly approximating the actual measured stress-strain curve more nearly approximating the actual measured stress-strain curve gave maximum hoop surface strains of 18 percent which are in agreement with the values measured and calculated at LASL. Although the detailed shape of the stress distribution is not yet in complete agreement with the LASL results, it appears now that this is due to the remaining discrepancy between the bilinear stress-strain curve representation and the curvilinear curve used in NONSAP. This will be cleared up by adding the nonlinear stress-strain curve capability to HONDO and rerunning the problem.

The investigation demonstrated that the relative size and spacing of the finite elements in the model can have significant effects on the predicted results. Although this is well-known for all finite element analyses, it appears that element size effects in dynamic problems may be of a different character than for static problems. In effect, the finite elements used in HONDO tend to be too "stiff". Moreover, the bigger they are, the stiffer they are. Thus, when models were used in which very small elements were used at the impact face, backed up by much larger elements, the relatively greater "stiffness" of these larger elements forced the smaller elements to overstrain. Using a fairly uniform mesh such as used by LASL alleviates this problem. However, this effect will certainly have to be carefully accounted for in analyzing the impact of the GPHS modules. Because of the more complex shape of these proposed fuel capsules, it will have to be modeled with elements of widely varying size.

Development of a Response Model for C/C Composites (F. A. Simonen, Battelle-Northwest)

A mathematical response model allowing for the crushing and fracture of a transversely cross-piled C/C laminate was reported in November, 1975. This response model has been programmed for insertion in the HONDO program. For completeness, this section will describe the response model and then describe the changes in HONDO for implementing the model. These changes, together with those additional features of HONDOV described earlier, will enable the application of HONDOV to the two-dimensional analysis of RTG fuel protected by C/C overwraps.

The Mathematical Response Model for Carbon/Carbon Composites.

Figure 3 shows some of the features of an analytical model to describe the load bearing characteristics of a two-dimensional carbon-carbon composite material. This model is intended to introduce a highly anisotropic behavior due to relatively high modulus and strengths in the direction of the fibers of the reentry impact member (RIM) material.

The approach taken in Battelle's model is to superimpose the two-dimensional fibrous type of response on an otherwise isotropic media. The large deflection crush-up response will be described by matching existing equations in HONDO for plastic and/or crushable materials to data from experiments on the RIM and other carbon-carbon materials. The behavior of the material under hoop or transverse stress loading will be strongly influenced by the fiber characteristics. At sufficiently large strains, the response model will simulate the loss or fiber continuity through fracturing of fibers. At large deformations, complete fracturing of the fibers in a given element will result in deformation by flow and compaction processes only.

As indicated by Figure 3, a random, directional transverse lay-up of fibers can be approximated by a laminated plate of many plies at random orientations. As the thickness of each ply decreases to zero and the number of plies approaches infinity, the behavior of the laminated plate will approach that of the random weave, two-dimensional composite.

As a first approximation, the behavior of a single ply, as shown in Figure 3b, is taken as

$$\sigma_{\alpha} = E_o \epsilon_{\alpha} \quad ,$$

$$\sigma_{\beta} = 0 \quad ,$$

where E_o is the fiber modulus times the volume fraction of the fibers in the composite. If ϵ_x and ϵ_y are the strains in the ply related to the x-y axes and the stresses in the ply related to these same axes are

(a) Laminated Plate Representation of Two Dimensional Composite Material

(b) Stress in Single Ply

FIGURE 3. FIBER MODEL OF TWO DIMENSIONAL CARBON-CARBON COMPOSITE

$$\sigma_x = E_o \cos^2 \alpha \left[\epsilon_x \cos^2 \alpha + \epsilon_y \sin^2 \alpha \right] ,$$

$$\sigma_y = E_o \sin^2 \alpha \left[\epsilon_x \cos^2 \alpha + \epsilon_y \sin^2 \alpha \right] ,$$

$$\sigma_{xy} = E_o \sin \alpha \cos \alpha \left[\epsilon_x \cos^2 \alpha + \epsilon_y \sin^2 \alpha \right] .$$

If one considers $N + 1$ plies stacked at equally space angular orientations, the overall response can be computed as a summation as follows:

$$\sigma_x = \frac{E_o \Delta t}{N + 1} \sum_{n=0, N} \cos^2 \left(\frac{n\pi}{N} \right) \left[\epsilon_x \cos^2 \left(\frac{n\pi}{N} \right) + \epsilon_y \sin^2 \left(\frac{n\pi}{N} \right) \right] ,$$

where Δt = thickness of each ply, and $(N + 1) \Delta t = 1.0$.

Similar equations hold for the stress components σ_y and σ_{xy} . As one takes the limit as $N \rightarrow \infty$, it can be shown that the above equation gives

$$\sigma_x = \frac{E_o}{\pi} \left[\epsilon_x \int_0^{\pi} \cos^4 \theta d\theta + \epsilon_y \int_0^{\pi} \cos^2 \theta \sin^2 \theta d\theta \right] .$$

Evaluation of the definite integrals of the trigonometric expressions can be made. Carrying out the derivations for all stress components results in the following stress-strain equations for elastic behavior.

$$\sigma_x = \frac{3}{8} E_o \epsilon_x + \frac{1}{8} E_o \epsilon_y ,$$

$$\sigma_y = \frac{1}{8} E_o \epsilon_x + \frac{3}{8} E_o \epsilon_y ,$$

$$\sigma_{xy} = 0 .$$

The effective elastic modulus and Poisson's ratio of the composite can be shown to be

$$E = \frac{1}{3} E_o ,$$

$$\nu = \frac{1}{3} .$$

The derivations have also been extended to account for fracture of fibers at those orientations in which the strain component exceeds a strain limitation of the individual fibers. Thus, the gradual breakup of the fibers can be taken into account. However, numerical integration is required to calculate the effective properties of the material. For example, the stress component, σ_x , is given by

$$\sigma_x = \frac{E_o}{\pi} \int_0^{\pi} \cos^2 \theta f(\epsilon_x, \epsilon_y, \theta) d\theta \quad ,$$

where

$$f = \delta \left[\epsilon_x \cos^2 \theta + \epsilon_y \sin^2 \theta \right] \quad ,$$

$$\delta = \begin{cases} 1, & \text{if } \epsilon_x \cos^2 \theta + \epsilon_y \sin^2 \theta < \epsilon_f \quad , \\ 0, & \text{if } \epsilon_x \cos^2 \theta + \epsilon_y \sin^2 \theta \geq \epsilon_f \end{cases}$$

ϵ_f = strain to fracture of individual fibers.

Figure 4 shows results of numerical calculations based on these equations. The composite was approximated as 20 plies, with

$$E_o = 17,000,000 \text{ psi}$$

$$\epsilon_f = 0.005 \quad .$$

The predicted stress-strain characteristics for various values of strain ratio ϵ_x/ϵ_y are shown in Figure 4. The initial response is linear elastic and remains so until fracturing of fibers at critical orientations occurs. Thereafter, a gradual decrease in load-bearing capacity occurs as more and more fibers fracture. The numbers along the calculated curves indicate the percent of fibers that have fractured in the model at a given strain level. It is significant that the curves show a predicted capability of the composite to maintain some load-bearing capacity at uniaxial strains far in excess of the strain limitation of individual fibers.

FIGURE 4. STRESS-STRAIN BEHAVIOR PREDICTED BY FIBER MODEL OF TWO-DIMENSIONAL CARBON-CARBON COMPOSITE

Incorporation of the fiber behavior into HONDO requires equations which allow the two-dimensional fiber-type material to be arbitrarily oriented with respect to the axis of revolution. This orientation may be defined as α in the initial unstrained configuration and $\Omega = \alpha + \theta$ in the deformed configuration. The angle θ is the rotation, which can be calculated from the deformation gradient as

$$\theta = \text{Arctan} \left[\frac{1}{2} \left(\frac{\partial z}{\partial R} - \frac{\partial r}{\partial Z} \right) \right].$$

The partial derivatives are components of the deformation gradient and are computed numerically within the HONDO computer code.

In order to apply the stress-strain equations to an anisotropic material, the deformation gradients in the rotated principal material directions are required. Of primary concern is the quantity $\partial r^1 / \partial R^1$, where r^1 and R^1 correspond to axes in the rotated coordinate system. The chain rule for partial differentiation gives

$$\frac{\partial r^1}{\partial R^1} = \cos \Omega \left[\cos \alpha \left(\frac{\partial r}{\partial R} \right) + \sin \alpha \frac{\partial z}{\partial R} \right],$$

where the derivatives on the right-hand side are computed numerically in the present version of HONDO. The quantity $\partial r^1 / \partial R^1$ is a measure of the deformation within the direction of the fibers. The comparable measure in the cross fiber direction is

$$\frac{\partial z^1}{\partial Z^1} = \cos \Omega \left[-\sin \alpha \left(\frac{\partial r}{\partial Z} \right) + \cos \alpha \left(\frac{\partial z}{\partial Z} \right) \right].$$

The model of fiber behavior in a carbon-carbon composite will permit the representation of carbon-carbon materials in HONDO in a relatively straightforward manner. The model requires a minimum of material property data in the form of basic fiber characteristics. The fiber model is intended only to represent the hoop strength and stiffness characteristics of the composite material. Energy absorption through crush-up type mechanisms will be represented by more conventional isotropic constitutive equations. Possible coupling between the two superimposed responses has not yet been formulated. This coupling could be in the form of fiber fracture due to transverse deformation as measured by the quantity $\partial z^1 / \partial Z^1$.

Carbon-Carbon Response Model Implementation in HONDO. In the computer program HONDO the fiber stress-strain behavior is obtained by relating the second Piola-Kirchhoff stress to the Green-St. Venant strain. The corresponding Cauchy stress components are then computed for the equilibrium calculations in HONDO. This appears to be an acceptable procedure to formulate constitutive equations for the fiber type material of interest. Strains in the fibers are relatively small and are not influenced by volumetric effects. The formulation permits large rotations and provides a suitable scheme to account for changes in element cross-sectional areas due to large strains transverse to the fiber plane.

Subroutine SE9. The subroutine SE9 is a new routine for the program HONDO which calculates element stresses due to deformation of carbon fibers. As presently coded, a total of 20 plies of fibers are used to approximate a random layup. Due to symmetry of the deformation state in the axisymmetric analysis, only ten angular integration points are used in the calculation. The elastic modulus of the equivalent unidirectional layup is presently set at 17×10^6 psi within the subroutine SE9. Also, the fiber fracture strain is present at 0.005 in./in. Both of these values correspond to the values estimated for purposes of the November 1975 progress report, and should be better defined in future work.

Subroutine SE2. This subroutine forms the basis for the transverse behavior of the two-dimensional composite. Details of the response model used in SE2 are described in the Sandia report on the computer program HONDO. Specification of material number nine on problem input cards automatically activates the crushable material option of subroutine SE2 in conjunction with the fiber model of subroutine SE9. A revised source deck for SE2 will be supplied to BCL which includes updates made in accordance with a recent notice from Sam Key of Sandia Laboratories.

Subroutine SOLVE. The revised source deck of the subroutine SOLVE incorporates call statements to subroutine SE9. It also carries out the superposition and partition of the fiber and crushable material aspects used to simulate the carbon-carbon response. Fiber stresses and fracture indication

switches are contained in the arrays of a new common block BKF. These arrays are presently coded to allow for a maximum of 200 elements. The subroutine SOLVE controls the initialization of the stress and fracture arrays. In addition, the restart tape configuration has been modified to include the new stress and fracture arrays.

Subroutine DATAIN. The changes in the subroutine DATAIN provide for the additional material type number nine. Also, columns 31-40 on the element card indicate the orientation of the plane of the composite material relative to the R-Z coordinate axes. An angle of 0 degrees indicates that the plane is normal to the Z-axis, and an angle of 90 degrees indicates that the plane is normal to the R-axis.

Subroutine FORCE. The revised subroutine FORCE contains a one-card correction to the internal energy calculation procedure. This portion of the routine was found to give incorrect results on certain test problems.

Testing of Subroutine SE9. Extensive numerical results were generated by calling the subroutine SE9 from a small driving program. This was done to eliminate as many errors from the routine as possible before coupling it to the main finite-element program. In one set of trial calculations, the results presented in Figure 6 of the November 1975 progress report were reproduced. Following this, a spectrum of deformation states were provided to the subroutine as input. These states included large rotations and large values of strain transverse to the direction of the composite. Also, the inclination of the material direction was varied. The subroutine SE9 was found to give reasonable and consistent results for these test cases.

Analyses of the Transverse Compression of the RIM Material. An enclosed set of plotted results (Figures 6 through 14) describes a test problem involving uniaxial compression of the RIM material transverse to the plane of the composite. In the dynamic analysis, a 0.5-in.-thick sample (Figure 5) was assumed to be impacted by a massive object at a velocity of 300 ft/sec. The volumetric behavior of the RIM material was specified in accordance with Battelle compression test data from small samples. While reasonable values

FIGURE 5. EXAMPLE PROBLEM - PRESSURIZED
SPHERE OF RIM MATERIAL

FIGURE 6. RADIAL EXPANSION OF PRESSURIZED SPHERE OF RIM
MATERIAL $R_i = 1.0$ psi, $R_o = 1.5$ psi

FIGURE 7. HOOP STRESS IN PRESSURED SPHERE OF RIM MATERIAL, $R_i = 1.0$ IN. $R_o = 1.5$ IN.

FIGURE 8. RADIAL EXPANSION OF PRESSURIZED SPHERE OF RIM MATERIAL
 $R_i \approx 1.0$ psi, $R_o = 1.5$ IN.

FIGURE 9. HOOP STRESS IN PRESSURIZED SPHERE OF RIM MATERIAL
 $R_i = 1.0$ IN., $R_o = 1.5$ IN.

FIGURE 10. EXAMPLE PROBLEM - TRANSVERSE COMPRESSION OF RIM MATERIAL

FIGURE 11. TRANSVERSE COMPRESSION OF RIM MATERIAL WITH LATTERAL RESTRAINT

FIGURE 12. DISTRIBUTION OF TRANSVERSE COMPRESSIVE STRESS IN RIM MATERIAL WITH LATTERAL RESTRAINT

FIGURE 13. TRANSVERSE COMPRESSION OF RIM MATERIAL WITHOUT LATTERAL RESTRAINT

FIGURE 14. DISTRIBUTION OF TRANSVERSE COMPRESSIVE STRESS IN RIM MATERIAL WITHOUT LATTERAL RESTRAINT

of elastic shear modulus, yield strength, and bulk unloading modulus were assumed, further consideration needs to be given to these parameters before additional calculations are performed. The demonstration calculations were performed for conditions of both lateral restraint and lack of lateral restraint.

Instrumented High Loading Rate Test
for Impact-Member Materials
(B. D. Trott)

The objective of this test is to develop force-deformation data on present and future impact materials for use in assessing rate effects to 300 fps on energy-absorption mechanisms. It is planned that the test will be conducted at room temperature to provide relative rankings of the materials. The data obtained from this test will be correlated with the "static" force-deformation tests being conducted at both room and elevated temperatures.

The planned test is not simply an ultra-high strain rate test, but rather an impact simulation test which will duplicate the strain rates occurring at simulated ground impact of a reentering heat source. As such, impacts in the range of expected reentry impact velocities will initiate the generation of force-deformation data. During this initial phase of the impact, strain rates are very high and fast response instrumentation is required, and unwanted stress wave reflections must be avoided. During the final phase of the impacting body, relative motions cease and lower strain rates occur. To date, all high strain rate tests of impacting materials have been performed on a pass-fail basis only due to the unavailability of such a sophisticated apparatus in this country. The ability to perform such tests should be of great significance to future NRA activities.

Work on this new test was initiated in the fall of 1976. In the interim equipment to conduct the test was designed and fabricated. The motion transducer originally conceived and built failed because of an unanticipated triboelectric effect. During the latter part of the previous reporting period, work was initiated to develop an optical motion transducer. The optical transducer to be described in this report has been calibrated and shown to work satisfactorily in a dummy shot with fabric-reinforced plastic.

The optical motion transducer is shown schematically in Figure 15. Light from the tungsten ribbon filament lamp (1) which provides a relatively large (~ 2 mm x 10 mm), uniformly illuminated source is collimated and passed through a slot milled through the launcher barrel. The light beam entering is partially interrupted by the rear of the projectile. The light emergent from the slot is refocused to image the tungsten filament in the plane of the photodiode sensor. The electrical signal from the photodiode is amplified and sent to recording oscilloscopes. The output of

FIGURE 15. SECTION VIEW OF IMPACT GEOMETRY SHOWING OPTICAL MOTION TRANSDUCER

(1) Tungsten Ribbon Filament Lamp	(7) Projectile Skirt (Al)
(2) Collimating Lens	(8) Projectile Face (Steel)
(3) Slot thru Launcher Barrel	(9) Test Specimen
(4) Launcher Barrel	(10) Replaceable Tup
(5) Focusing Lens	(11) Force Transducer Bar
(6) Post Photodiode	

the photodiode was shown to be very accurately linear versus projectile position over the central one inch of the milled slot in static calibrations, and a test firing of a projectile with no target confirmed the linearity of output under dynamic conditions. A good clean signal was obtained. In several calibration checks, the slope of the transducer output remained constant to within 0.3 percent.

In a dummy shot using a fabric-filled phenolic test specimen, the motion and load transducers gave clean signals and showed the need for an accurate fiducial mark on the transducer outputs to show the instant of projectile contact for data reduction purposes. After some experimentation a satisfactory trigger circuit was developed which places a small, short-duration fiducial pulse on the load transducer output, without further effecting the load output curve which occurs later ($\sim 26 \mu\text{sec}$) in time.

Also during this reporting period a data reduction system was developed and programmed for computer-based data reduction. The program was checked out with a dummy data set, debugged and readied for operation.

During the next reporting period it is expected that a number of dynamic tests will be performed.

Energy Absorption By Bulk Graphite (G. K. Bansal and W. H. Duckworth)

The earlier data reported by LASL from impact tests of copper balls contained in shells of Carbocell, golf-ball wound Thornel 50 composite, and bulk graphite revealed less deformation in the copper ball encased in bulk graphite than in either of the two carbon-carbon composites. The indicated good performance of the bulk graphite was postulated by BCL* to be associated with strain hardening characteristics of the copper in the dynamic test, coupled with formation of a graphite "plug" at the impact face early in the impact event. The plug is believed to be entrapped during the impact event, and subsequently to absorb a significant amount of kinetic energy by crushing.

* Heat Source Component Development Program Quarterly Report BMI-X-676, p 25 January 1977.

To examine whether the entrappment of the plug and subsequent crushing could be achieved in a static test with a relatively soft and strain-hardenable fuel simulant, a copper ball, 3/4 inch diameter and of hardness RF-10, was forced against a flat plate, 2 x 2 x 0.5 inch, of ATJ-S graphite. The ball deformed approximately 10 percent along the load axis, but no entrappment of a "plug" nor crushing were obtained. The graphite specimen fractured catastrophically, as was the case in static tests with a hard fuel simulant (steel)**. It is, therefore, indicated that the good performance of the graphite shell in LASL tests is associated with dynamic effects as postulated.

* Heat Source Component Development Program, Quarterly Report BMI-X-673, p 9
October 1976.

IMPROVED REENTRY MATERIALS
(I. M. Grinberg - Task Leader)

Introduction

The objective of this program is to expand and improve on the understanding of the reentry behavior of carbon-carbon composites for isotope heat sources and then to select or develop an improved material for this application. If possible, this material will serve the function of impact protection as well, so that the overall goal of a bifunctional material development can be achieved.

Task activities are being conducted such that the recommended heat shield or bifunctional material can be applied to the General Purpose Heat Source (GPHS) which is being designed by LASL.

Shown below are the major efforts being conducted as part of this task along with the specific objectives of each activity.

- Evaluate Bifunctional Component Configurations. The objectives of this activity are to determine the response of bifunctional component configurations for thermal, ablative, and thermal stress performance and to identify critical factors associated with the use of graphite and carbon/carbon materials for heat source heat shield/bifunctional members.
- Surface Chemistry Studies. The overall objective of this effort is to develop an improved criterion for specifying the heat shield/bifunctional member (ablator) thickness. The immediate objective of the work currently in progress is to determine the boundary layer surface chemistry on candidate heat shield members at simulated reentry conditions.
- Develop Carbon/Carbon Composite Failure Criteria. The objectives of this effort are to formulate an approach to predict the thermal stress resistance of C/C composites for isotopic heat source heat shield/bifunctional members and to define suitable failure criteria for these materials.

These activities as well as those to be implemented are being reviewed to ensure their relevancy and timeliness for input to the GPHS Technology Readiness milestone of March, 1978.

Summary

Efforts during this quarter in this Improved Reentry Materials task were primarily directed toward the bifunctional component configuration and surface chemistry component configuration subtask) were redirected to identify those efforts required to generate design information on heat shield or bifunctional member materials for the GPHS, including the preparation of a preliminary work plan and schedule for the supporting heat shield technology. Analyses were also conducted which illustrate the importance of graphite surface chemistry on heat shield ablation and metal containment impact for GPHS shape modules. These results are compared with results of analyses previously conducted for spherical shape heat source modules.

Further progress on the surface chemistry study subtask is reported, primarily in regard to the achievement of the required material surface temperature level to simulate subsonic minimum γ reentry and in the equipment setup.

Work This Period

Evaluate Bifunctional Component Configurations (I. M. Grinberg, et al)

Activities in this subtask were redirected to focus on the identification of those efforts that would provide LASL with the required design information regarding the selection of graphite or carbon-carbon heat shield or bifunctional member materials for the GPHS module. Accordingly, the activities associated with the four subtasks:

- development of thermal/ablation models for use in configuration and thermal stress studies
- configuration sensitivity studies to identify preferred materials/heat source shapes

- two-dimensional thermal/ablation analyses for input to thermal stress studies
- two-dimensional thermal stress studies,

as documented in the last quarterly report,^{*} were not conducted. Rather, technical and planning activities were performed in response to the information LASL is expecting from BCL for technology support of their GPHS development. Identification of the deliverables is based on the letter of February 3, 1977, from S. E. Bronisz of LASL to W. M. Pardue of BCL, and discussions with E. Snow and R. Zocher of LASL at BCL on March 15-16, 1977.

From the viewpoint of reentry thermal/thermal stress protection, the design information to be provided by BCL for the GPHS consists of a ranking of graphite and carbon-carbon composites according to

- thickness/reentry protection considering
 - (a) ablation
 - (b) subsurface damage, if any
- thermal properties
- production availability
- cost

In addition, Battelle will assist LASL in the reentry design analysis of GPHS modules, i.e., selection and evaluation of material and design concepts.

A preliminary plan formulated to provide the desired information was presented by BCL for discussion at the March 15-16 meeting. This plan is being reviewed by BCL to ensure responsiveness to the GPHS performance requirements and schedule and will be further discussed with LASL at a meeting to be held during the next quarter.

Limited material property sensitivity studies were conducted to identify the seriousness of the design problems to be encountered in GPHS conceptual designs, e.e., effect of graphite or carbon-carbon surface chemistry on peak and impact metal containment temperatures, need for insulation or anisotropy in heat shield properties to maintain metal containment above embrittlement temperature at impact for iridium alloy clad. These preliminary results, along with the results of analyses previously conducted for a spherical heat source module, are given below and compared.

^{*} Heat Source Component Development Program, Quarterly Report BMI-X-676, p 25 January 1977.

Heat Shield Material Property Sensitivity Study (G. R. Whitacre).

The objectives of the heat shield sensitivity study are to (1) identify those material properties that influence the heat shield response most strongly and thereby affect the design and overall performance of the heat source module, and (2) rank materials for their application as heat source heat shield bifunctional members. The initial phase of the study was conducted in 1975 and was reported in the June, 1975, Informal Progress Report.* These results aided in the development of a test matrix plan and the work currently in progress. For example, the need for experimental work on the surface chemistry of graphitic ablators was established (currently in progress). During the current reporting period, these property sensitivity results were reexamined considering the changes and constraints imposed by the GPHS requirements.

The shape of the GPHS module (parallelepiped) results in lower impact velocities and longer reentry times than the spherical heat source module used in the initial sensitivity study. A disk-shape axisymmetric heat source model reentering face-on was used to approximate the GPHS class of shapes for the new property sensitivity analysis. This model will be refined further in the heat shield screening study to obtain more accurate temperature histories and distributions. Before discussing the current results, however, it is appropriate to summarize the results of the initial sensitivity study performed with the spherical thermal model.* This will help provide the basis for a direct comparison of the effect of shape change, i.e., sphere to brick or block on material performance during reentry and important parameters at impact.

Summary of Property Sensitivity Study With Spherical Model.

Figure 16 shows the idealized 150-watt spherical heat source thermal model used in the initial material property sensitivity study. For this study, a one-dimensional thermal and ablation analysis was performed with stagnation point conditions supplying the heat input. The heat shield thickness was adjusted to be twice the predicted recession for the maximum recession trajectory which is the minimum reentry angle for nonskip case. Table 2 lists the conditions used in the initial sensitivity study.

* Letter to Mr. Arnold Litman dated July 21, 1975; Informal Progress Report for June, 1975, on Task 67 (Impact Member) and Task 68 (Reentry Materials), Contract W-7405-eng-92, Battelle's Columbus Laboratories.

FIGURE 16. 150-WATT SPHERICAL-HEAT-SOURCE THERMAL MODEL

TABLE 2. CONFIGURATION AND REENTRY CONDITIONS USED
FOR HEAT SHIELD SENSITIVITY ANALYSIS

150-Watt Spherical Heat Source with Spherical Heat Shield

$^{238}\text{PuO}_2$ Spherical Fuel Source O.D. = 1.710 in.

3.50 Watts/cc

Iridium Encapsulation Member 0.02 in. thick

T50 Sphere Impact and Insulation Member 0.46 in. thick

Heat Shield Thickness Adjusted to be Twice Recession

Radiation Gaps Between Members

Initial Temperature Distribution Steady State With

Hot Junction Temperature 950 C

RTG Breakup at 271 Kft for Low-Angle Trajectories

RTG Breakup at 200 Kft for Steep Angle Trajectory

Reentry Initial Conditions at 400 Kft

	<u>V ft/sec</u>	<u>degrees</u>
Minimum γ (for nonskip)	36,000	-5.3
Orbital Decay	25,600	-0.1
Steep Angle	36,000	-89.9

Ballistic Coefficient W/C_D A

Weight Determined by Configuration and Density

Frontal Area Determined by Outer Radius

C_D for Spherical Configuration

Figure 17 illustrates the importance of the two factors related to surface chemistry which have a significant effect on heat shield recession during reentry. These factors are (1) boundary layer, diffusion-controlled, mass loss (factors of heat of combustion and the corresponding diffusion oxidation rate) and (2) reaction rate-controlled mass loss (factors of activation energy and collision frequency). Results of analyses conducted to determine the effect these factors have on heat shield recession for the spherical heat source configuration are given in Table 3. Also shown in Table 3 are the representative recession and containment member temperature results obtained when "other" effects (e.g., aerodynamic effects, materials properties) were varied.

Considering the diffusion phenomena, the results of the first three cases listed in Table 2 illustrate the conservatism in predicted heat shield recession resulting from the inconsistent assumption regarding the values of the heat of combustion and the diffusion rate. For example, the diffusion rate used in these cases is consistent with the formation of only CO at the heat shield surface, whereas the values used for the heat of combustion for Cases 1, 2, and 3 are consistent with 33, 50, and 100 percent, respectively, CO₂ formation at the material surface. The maximum recession for these cases can be compared with the results of analyses for Cases 4-7 for which consistent assumptions were made regarding the values of the heat of combustion and the oxidation diffusion rate. It can be seen that the peak recession for Cases 1-3 are higher than for Cases 4-7. The dramatic increase in recession for Case 2 occurs because the material surface remains sufficiently hot to burn all the way to impact. The uncertainty associated with the actual behavior of graphitic materials during subsonic flight of a low γ reentry, as typified by the shaded region in Figure 17, has prompted the use of conservative but inconsistent assumptions regarding the treatment of graphite surface chemistry in the preliminary sizing and design of RTG heat source modules.

Also of relevance in terms of the diffusion effect for the spherical heat source configuration is the relative insensitivity of the heat shield recession to the magnitude of CO/CO₂ formed at the wall when consistent assumptions are made regarding the values of the heat of combustion and the diffusion rate. The results are illustrated by Cases 4-7. Apparently, the effect of the reduction in diffusion rate on decreasing the recession

FIGURE 17. GRAPHITE ABLATION

TABLE 3. SENSITIVITY RUNS WITH 1-D SPHERE CONFIGURATION

Case No.	CO ₂ , Percent	Diffusion Factors		Type of Kinetics	Kinetics Factors		Gap	Maximum Recession, in.	Iridium Temperature, C	
		Heat of Combustion, Btu/lb	Diffusion Oxidation Rate, lb/ft ^{3/2} sec atm ^{1/2}		Activation Energy, Kcal/mole	Collision Frequency, lb/ft ² sec atm ^{1/2}			Peak	Impact
<u>Diffusion Effects</u>										
1	--	6,486	0.00635	POCO*	28.05	217.5	Vacuum	0.182	2309	1594
2	--	8,036	0.00635	POCO	28.05	217.5	Vacuum	0.285	2276	1709
3	--	14,100	0.00635	POCO	28.05	217.5	Vacuum	0.296	2281	2042
4	0	3,950	0.00635	POCO	28.05	217.5	Vacuum	0.169	2314	1551
5	33	6,486	0.005115	POCO	28.05	217.5	Vacuum	0.143	2323	1566
6	50	8,036	0.004572	POCO	28.05	217.5	Vacuum	0.133	2325	1581
7	100	14,100	0.003175	POCO	28.05	217.5	Vacuum	0.154	2318	2003
8	0	3,950	0.00635	POCO	28.05	217.5	He	0.171	2296	1174
<u>Kinetic Effects</u>										
9	0	3,950	0.00635	"Slow"	42.3	4.47 x 10 ⁴	Vacuum	0.178	2310	1561
10	0	3,950	0.00635	"Moderate"	44.0	9.65 x 10 ⁵	Vacuum	0.298	2266	1686
11	0	3,950	0.00635	"Fast"	44.0	6.73 x 10 ⁸	Vacuum	0.298	2266	1687
<u>Other Effects</u>										
12	0	3,950	0.00635	POCO	28.05	217.5	Vacuum			
With Laminar Subsonic Flow										
13	0	3,950	0.00635	"Moderate"	44.0	9.65 x 10 ⁵	Vacuum	0.353	2251	1664
With Laminar Subsonic Flow										
14		With Emissivity Reduced to 0.5						0.206	2436	1706
15		With Thermal Conductivity Reduced to 1/10 POCO Values						0.168	2004	1509
16		With Thermal Conductivity Increased to 5 x POCO Values						0.169	2346	1554

γ = -5.3 degrees

* POCO parameters indicate slow kinetics.

rate is essentially compensated by the effect of the increase in the heat of combustion.

The second surface chemistry factor which has a significant effect on recession is the kinetic behavior of the oxidation. The analysis of Cases 1-8 in Table 3 were made with the kinetic parameters of activation energy and collision frequency characteristic of POCO AXF-5Q graphite.* Although the individual parameters are different, the net effect is essentially the same as the case when typical values called "slow" graphite kinetics are used, see Case 9 in Table 3. However, when the graphite kinetic parameters are increased to values called "moderate" graphite kinetics by Scala,** see Case 10, the recession increases because burning continues all the way to impact. Further increase to "fast" kinetics (Case 11) results in no additional recession since the surface is already remaining in the diffusion controlled regime all the way to impact with the moderate kinetics.

The majority of the sensitivity analyses for the spherical heat source module were made assuming that the subsonic flow was turbulent. Subsequent to conducting these analyses, test results reported by GE demonstrated that the heated heat source module surface would delay boundary layer transition such that the boundary layer would remain laminar essentially to impact. For most cases considered in Table 3, this change would cause no significant difference as demonstrated by the results with laminar subsonic flow using POCO kinetics (Case 12). However, when combustion occurs all the way to impact using moderate kinetics (Case 13), the recession is significantly greater. This is caused by the longer reentry time resulting from the higher laminar drag coefficient. The surface chemistry considerations should thus be even more significant for the GPHS-type shapes which have even higher drag coefficients than the spherical shape. The brick- or disk-shaped GPHS modules will experience longer reentry times and lower impact velocities than will the spherical shape heat source module (or the cylindrical MHW HSA). Surface chemistry phenomena/sensitivity analyses for a disk configuration is discussed later in this report.

There is an additional property-sensitive result which is not shown in Table 3 but can be stated quite simply. Recession is practically

* Knight, D. D., and Knechtel, E. E., "Graphite Oxidation at Low Temperature in Subsonic Air," AIAA Paper No. 73-735, Presented at the AIAA 8th Thermophysics Conference, Palm Springs, California, July 16-18, 1973.

**Scala, S. M., "The ablation of Graphite in Dissociated Air, Part I. Theory," IAS Paper 62-154, June, 1962.

inversely proportional to the heat shield density. A lower density ablator does reduce the W/A^* of the heat source module and thereby decreases the severity of the reentry environment. This could decrease the required weight of ablator slightly. However, the larger required module diameter will increase the surrounding RTG volume and weight such that the overall RTG weight might actually increase. In addition, these analyses did not consider the effect demonstrated in tests that a low-density heat shield material may experience significantly greater mass loss due to surface roughness effects or reduced erosion resistance.

Predicted peak and impact metal containment** temperatures for the spherical module are also shown in Table 3. Because of the one-dimensional analysis and the use of stagnation-point heating, the predicted temperature levels are not accurate. However, the trends and sensitivity of the temperature to parameter variations should still be valid. For the spherical heat source module, the peak clad temperature is relatively insensitive to all the variations shown in Table 3 except for the drastic change of reducing the surface emissivity to 0.5. (Actually, this low value of emissivity is a special case which applies only to pyrolytic graphite and then only in the low-temperature range.) The only other sensitivity of peak clad temperature found in the study was to heat shield thermal conductivity. Reducing the thermal conductivity value to one-tenth that of POCO reduced the peak clad temperature by 300 C. Increasing the thermal conductivity value had little effect, primarily because the thermal resistance of the impact/insulation member became controlling.

The metal containment temperature at impact is much more sensitive to the various properties and conditions, for example, the clad temperature at impact is very dependent on the heat transfer in the gaps. The most probable condition is now considered to be helium-filled gaps, although most of this initial study was done with vacuum gaps and heat transfer only by radiation across the gaps. The heat of combustion has a significant influence on the clad temperature at impact. In addition, reducing the heat shield emissivity to 0.5 also results in a higher metal containment impact temperature. The thermal conductivity of the heat shield material had little effect on the clad temperature at impact.

* W = heat source module weight, A = heat source module frontal area.

**"Metal containment" and "clad" are used interchangeable in this report.

Following the one-dimensional spherical property sensitivity study, a shape sensitivity study was conducted which is reported in detail in the Quarterly Report for July-September, 1976^{*}. As part of that study, the spherical heat source module was reanalyzed using a two-dimensional (2-D) thermal analysis assuming stable reentry. This thermal model results in much more accurate temperature predictions than does the one-dimensional assumption. Several other changes were made in the analysis procedure at that time. These consisted of the following: (1) the thickness of the impact/insulation members was adjusted so that the energy absorption capability matched the predicted impact velocity using an iterative procedure, (2) the heat shield thickness was adjusted to be twice the predicted maximum recession as before in the analysis reported herein, and (3) laminar subsonic flow with its higher drag coefficient was established as the standard condition. These changes resulted in a very slight change in maximum recession in comparison with the baseline one-dimensional case for 0 percent CO₂, 0.153 in. compared to 0.169 in. As expected, there was a significant change in predicted peak and impact clad temperature for the baseline case due to the two-dimensional heat conduction effects, with the peak iridium temperature decreasing from 2296 to 1920 C and the clad impact temperature decreasing from 1174 to 988 C. This temperature is believed to be sufficiently high for improved iridium alloys to maintain adequate ductility and resist fracture during the impact event.^{**} In the absence of He in the gaps (vacuum gaps), the peak iridium temperature would be 1968 C and the impact temperature would be 1336 C for the 2-D thermal model.

Sensitivity Runs with Disk Configuration (GPHS-like). The shape sensitivity study conducted during this reporting period considered a disk heat source module configuration with a height/diameter ratio of 0.68 which is an approximate two-dimensional representation of preliminary GPHS module designs. This heat source module was analyzed for heat shield recession response and first order thermal response for the $\gamma = -5.3^\circ$ trajectory assuming a face-on stable orientation to impact. This orientation is the

* "Heat Source Component Development Program", Quarterly Report for July-September, 1976, BMI-X-672, October 1976.

**General Purpose Heat Source Program, Open Contractors Briefing, January 25, 1977, Attachment II.

probable attitude in hypersonic flow, however, the most probable configuration in subsonic flow is tumbling about a fixed axis which is perpendicular to the air stream. The face-on analysis should be suitable for preliminary property sensitivity determinations.

Figure 18 shows the disk configuration thermal model used in the sensitivity analysis. This model which was originally used in the shape sensitivity analysis^{**}, has the properly adjusted RIM (reentry impact member) impact thickness and POCO heat shield thickness. The predicted impact velocity is 166 ft/sec. For the material property sensitivity study conducted during the current reporting period the thickness were not readjusted from the baseline case as properties change because this was found to be a second-order effect in the previous analysis using the one-dimensional response model. In the two-dimensional analysis, this iterative process would become time consuming and quite expensive and add little to the accuracy and applicability of the results. This module contains 150 watts_{th} of fuel with 3.50 watts/cc in a single disk-shaped central core. It is recognized that this model is not an adequate representation of the preliminary GPHS fuel array and geometry. This two-dimensional model will be refined and compared to a limited number of three-dimensional analyses to define an adequate two-dimensional thermal model for the GPHS in support of the heat shield screening efforts. A more accurate representation of the GPHS configuration is essential because of the need to determine the metal containment temperature at impact and the thermal stress performance of the heat shield member. For the heat shield property sensitivity analysis which is concerned with the comparative performance of heat shield materials (primarily recession), the model shown in Figure 18 with the proper fuel loading should be adequate for screening purposes.

Table 4 shows the results of the property sensitivity analyses with the two-dimensional disk configuration of Figure 18. The significant effects of surface chemistry which were demonstrated with the one-dimensional spherical module were rechecked with the two-dimensional disk configuration. Because of the higher drag and longer reentry times resulting from the GPHS brick-like shape, (especially at subsonic velocities) surface chemistry effects are important for this configuration.

It is anticipated that either special insulation members or anisotropy in the heat shield thermal conductivity will be required for the GPHS module to ensure adequate thermal protection of the metal containment member during reentry. Alternatively, the impact member can also serve as a bifunctional insulator-impact member. In the preliminary analysis reported herein, for the GPHS module shape, the low-conductivity direction for the reentry impact member (RIM) material was taken as the radial direction (direction r in Figure 18). A more efficient approach is to have the low-conductivity direction in the axial direction, especially for face-on reentry. For example, pyrolytic graphite should be considered as a special insulation member not only because of its desirable conductivity characteristics but also because of its unique low emissivity at low temperatures. The low emissivity would reduce radiation heat losses from the heat source module and (possibly) maintain the metal containment member above the embrittlement temperature at impact. Finally, it might be necessary to rely on the relief afforded by using the tumbling mode of subsonic reentry rather than the limiting case of face-on reentry. In this case, it would be necessary to establish that the stable face-on mode could not occur in subsonic reentry.

In summary, the sensitivity of ablation characteristic to surface chemistry effects has been confirmed for disk-shaped heat source modules approximating GPHS shapes. Proper temperature control of the metal containment member temperature through choice of materials coupled with unique designs appears to be a major problem area for parallelepiped or disk-shaped heat source modules.

Thermal Stress Studies (L. E. Hulbert)

Work on this subtask has been carried out at a very nominal level awaiting the results of the design review being performed at Los Alamos Scientific Laboratories. It was learned on a visit by L. E. Hulbert to LASL on January 12, 1977, that the GPHS configuration might be patterned after the LCHPG design. This was confirmed on a visit to BCL on March 10-11, 1977, by Ed Snow and Roy Zocher of LASL. Since the geometry of the reentry member has a fundamental influence on the thermal stresses and on the approaches used to model these stresses, a reevaluation of the approach to

model these stresses, a reevaluation of the approach to the thermal stress screening of materials for the reentry member was carried out. As a result, a new work plan is being drafted for this effort.

Surface Chemistry Studies

(A. A. Boiarski)

Introduction. The importance of the Surface Chemistry Experiment was described in detail in the previous Quarterly Report BMI-X-676. As indicated there, mass loss in the transition from the kinetically-controlled to the diffusion-controlled regime is highly dependent upon the ratio of CO to CO₂ at the material surface. Hence, the present surface chemistry experiment is an attempt to better define the graphite surface oxidation utilizing the laser Raman scattering technique to measure the concentrations of CO and CO₂ near the hot graphite wall. The experimental setup was also described in the previous Quarterly Report. As indicated in this earlier description, an infrared thermal camera will be used to check the uniformity of the surface temperature on the sample.

Current Progress. During this quarter, several pictures were taken with the infrared thermal imaging camera (i.e., AGA camera) of the inductively heated sample to determine the surface temperature gradient. A representative photograph is shown in Figure 19. The significant aspect of this picture is the small white spot at the center of the sample. The mean temperature of this region was measured with an infrared pyrometer which gave a value of 1530 C. The temperature span within this center spot was computed to be less than 6 C.

This isothermal zone is roughly circular with a nominal diameter of 1/4 in. The laser beam will be focused to the vicinity of this spot. The scattering volume (i.e., measurement region) will be less than 0.02 in. diameter by 0.04 in. long so it will be well within the isothermal zone. Thus, the maximum spatial variation in the surface temperature during the measurement will be less than 6 C or within 1/2 percent of the nominal centerline value. At a radius of 0.45 in. the sample was 86 C hotter than the central spot and the sample edge temperature was approximately 220 C higher than the central spot temperature. These temperature differences will

FIGURE 19. TEMPERATURE UNIFORMITY MEASUREMENT ACROSS GRAPHITE SAMPLE USING AN AGA THERMOVISION CAMERA

cause no difficulties in the present experiment. In fact, it is beneficial in the present experiment. In fact, it is beneficial that the edges are hotter than the center since increased ablation in those outer areas allows for better focusing of the laser beam near the surface at the sample centerline. If the reverse were true (i.e., edges cooler than center), the sample could become saucer-shaped and the edges would prevent the laser from being focused near the surface at the centerline.

Additional pyrometer and infrared pictures were obtained at various center-line temperatures from 1060 - 1600 C. This range covers the region of negligible mass loss to 0.6 of the diffusion-limited mass loss. Further induction power was available and higher temperatures could be obtained if necessary. However, theoretical estimates indicate that the above range is adequate to define the surface chemistry phenomena occurring during the graphite oxidation reaction.

The Optical Multichannel Analyzer (OMA) was received in early January. It has been checked out and mounted in the spectrograph. Both Copper Hollow Cathode and Mercury Pen-Ray Lamp spectra have been obtained with the OMA to adjust the focus and orientation of the receiver screen. A system to trigger the ruby laser using internal OMA signals was devised so that proper synchronization occurs between the laser firing event and the gating of the OMA Vidicon/Intensifier tube.

THERMODYNAMIC STUDIES
(C. A. Alexander and J. S. Ogden)

Introduction

The purpose of this task is to supply supportive information on the thermodynamic behavior of components within actual or potential heat source designs by basic studies of mechanistic nature.

Summary

Indications have been obtained that previously unobserved gaseous molecules exist in the iridium- $^{238}\text{PuO}_2$ system that might lead to unexpected long-term deleterious effects. Individual component of the total system have been analyzed, to determine the total contribution toward vapor species and vapor transport of the components in the iridium $^{238}\text{PuO}_2$ system.

Work This Period

Additional experiments and analysis of previous data to establish thermodynamic data upon which to predict reaction potentials when the total system is considered, have been carried out this period. The total system is made up of the fuel, the fuel impurities, the cladding, the cladding impurities, the GIS, and the gases which make up the atmosphere surrounding the system. As of this time the surrounding atmosphere needs to be defined, e.g., what effect does the cleaning procedure have on the gaseous species and do these species contribute to the vapor transport of material which might close a vent? The experiments conducted to determine reaction products within the total system have been carried out in Knudsen cells made of BeO , Y_2O_3 , Pt and Ir with no noticeable difference in the vapor species. The same mixture of U_3O_8 , fuel impurities as oxides and iridium powder was used in each crucible. One difference that was noted was that platinum does not form a complex molecule with sodium like the iridium does. Table 5 shows the typical impurities in the $^{238}\text{PuO}_2$ fuel, as reported in BMI-X-672. Table 6 shows the impurities found in the iridium used for fabrication of the vents. Table 7 shows the degree of vaporization contribution

by each of the impurities. These pressures are measured from a Knudsen cell where equilibrium is maintained (if there is enough material present) until the sample is depleted of that constituent. For the lead and copper the time required for depletion from the sample is a matter of a few minutes. Naturally, the depletion time depends upon the concentration and the volatility of each component.

TABLE 5. TYPICAL IMPURITIES IN $^{238}\text{PuO}_2$

Impurity	ppm
Al	18
Ca	31
Cr	46
Cu	3
Fe	120
Na	11
Ni	37
Pb	16
Si	82

TABLE 6. IMPURITIES IN THE Ir

Impurity	ppm
Fe	10
Ni	5
Al	10
Cu	5
Co	10
Si	10
Mn	5
Cr	30
Ca	5
W	1000
No other elements at detection limit	

TABLE 7. PARTIAL PRESSURES OF IMPURITIES WITH
 $PO_2 = 10^{-7}$ ATM AT 1700 K

Vaporizing Species	Pressure (atm)
Pb	10^{-1}
PbO	10^{-1}
Na	10^{-5}
NaO	10^{-7}
Cu	10^{-4}
Ni	10^{-6}
Fe	10^{-7}
Cr	10^{-7}
WO ₃	10^{-6}
Na ₂ WO ₄	10^{-6}
W ₂ O ₆	10^{-6}
Na ₂ IR ₄ O ₄	10^{-9}
IrO ₃	5×10^{-10}
Al	
Ca	no vapor species detected
Si	

The vaporization data listed in Table 7 was used as a guide to determine the frit material which would be acceptable to use in the selective vent (without adding to the problem of material transport and vent plugging). Thus Anorthite $CaO \cdot Al_2O_3 \cdot 2SiO_2$ was chosen as a material which would not cause material problems in the system.

The inside of the iridium crucible lid was analyzed using the Cameca Secondary Ion Mass Spectrometer. The purpose of this analysis was to determine if sodium was penetrating into the iridium (like one would expect when the sodium intensity would decrease with time at a given temperature). Figure 20 shows the depth profile of the sodium into the iridium lid of the crucible.

FIGURE 20. SODIUM DEPTH PROFILE INTO IRIDIUM

The penetration appears to be about 8000 Å. This amount of sodium penetration into the iridium would account for the decrease in sodium activity as theorized in BMI-X-676 and is very possibly the mechanism which leads to the formation of the complex molecule Na_2IrO_4 . The iridium crucible contained the material at 1425 C for a total of 22 hours. Figure 21 shows an electron probe scan of the surface area where the depth profile was made. The sodium is spread somewhat evenly over the surface, indicating that there has not been any concentration of the sodium in grain boundaries. This analysis also shows that the solution of the sodium into the iridium probably does not affect the strength of the iridium.

FIGURE 21. SURFACE SODIUM PICKED UP BY THE
IRIDIUM AS SHOWN BY ELECTRON PROBE

HELIUM VENT DEVELOPMENT
(C. A. Alexander and M. P. Rausch)

Introduction

The objective of this program is to develop helium vent systems capable of long-term unobstructed helium passage while containing particulates and restricting the egress and ingress of other gases through the primary fuel containment member. The approach is to use a parametric study of the meaningful variables determined by analytical examination of previous efforts, theoretical vent modeling, and basic vapor transport relations that have been developed at BCL in earlier studies.

Summary

Studies of selective vents are continuing: Pt vents have been eloxed, cleaned, annealed, and cold formed. The selective filter of anorthite will be applied and vents will be bonded at 1400 C in the new hot press facility.

Work This Period

Work this period was directed toward the fabrication of a selective vent.

An (additional) 100g batch of anorthite ($\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) was prepared by combining (weight percent);

- 31.06 CaCO_3
- 31.68 Al_2O_3
- 37.27 SiO_2

About 30g of the batch were loaded in a platinum crucible and fired in air in a molybdenum-wound alumina tube furnace to 1520 C. After 30 minutes at 1520 C the melt was removed from the furnace and quenched to room temperature. The appearance of the batch as it was withdrawn from the hot furnace indicated that the materials had melted.

A visual comparison of melt #1 and #2 materials showed striking differences between two. Microscopic examination of the first batch (fired at 1600 C for 20 minutes) revealed that the material had completely melted during processing and that it remained in a glassy state after its room temperature quench*. Examination of the second batch showed that a large amount of recrystallization had occurred upon quenching. Apparently the materials had not become sufficiently molten to form a homogeneous glass at the lower processing temperature of 1520 C. Material from both melts was crushed and sieved to minus 400 mesh. The glass formed at 1600 C was very high strength and considerably harder to crush than was the lower temperature melt. Powder mounts were prepared for each batch and X-ray diffraction (XRD) analyses of each melt were performed. The analysis showed that batch #2 had reacted completely to form anorthite. No residual silica (SiO_2), calcia (CaO), or alumina (Al_2O_3) was observed. XRD was not useful for species identification of melt #1 as the pattern obtained was characteristic of a glass and did not reveal any long range crystalline ordering. In general batch processing results show that the $\text{CaCO}_3/\text{SiO}_2/\text{Al}_2\text{O}_3$ mix forms a highly viscous glass at 1520 C which readily recrystallizes to anorthite when quenched rapidly. At higher temperatures a homogeneous melt is achieved which, when cooled, results in a high strength glass. From processing results anorthite appears to be a promising candidate for use as a selective vent filter material for temperatures approaching 1500 C.

Also this period, eight vents have been machined by a spark discharge technique from 5 and 10 ml platinum-rhodium foil. The vent pieces were cleaned as follows:

- (1) Detergent wash (hot, cold, then hot)
- (2) Deionized water rinse
- (3) Ethanol rinse
- (4) Chemically clean in a 7.7 normal nitric acid solution
- (5) Deionized water rinse.

* Heat Source Component Development Program, Quarterly Report BMI-X-676, January 1977.

The vents were then loaded into the tungsten element furnace and annealed for 2 hours at 900 C. The pieces have been cold formed to spherical curvature.

The electronics to the hot press (completed last quarter) have been connected and the RF generator was tested and is functioning properly. A new inductor coil was designed to accomodate the susceptor of the new press. An iridium nonselective vent (ThO_2 -Ir particulate filter) was prepared and is scheduled to be bonded in the new facility very soon. The selective filter will soon be added - the crushed anorthite will be mixed with a binder and applied by slurry impregnation. Vents will be bonded at 1400 C in the new hot press facility. Preliminary tests for bonding platinum and anorthite are currently underway.

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