Heat Source Component Development Program

QUARTERLY REPORT FOR APRIL-JUNE, 1977

Report Date: July, 1977

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Battelle

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HEAT SOURCE COMPONENT DEVELOPMENT PROGRAM

Quarterly Report for April-June, 1977

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Prepared for the Division of Nuclear Research and Applications,
U.S. Energy Research and Development Administration
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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, BMI-X-676 January 1977, and BMI-X-679 April 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. The development of a unitized reentry-impact member (RIM) has been stopped and the efforts are being redirected to the evaluation of materials that could be used in the near term for the module housing of the General Purpose Heat Source (GPHS). This redirection will be particularly felt in the selection of (improved) materials for reentry analysis and in the experimental evaluation of materials in impact tests. Finally thermochemical supporting studies are reported.

TABLE OF CONTENTS

	<u>Page</u>
IMPROVED IMPACT MEMBER	1
Introduction	1
Summary	1
Work This Period	3
Development of Preferred Impact-Member Materials . .	3
Instrumented High Loading Rate Test for Impact-Member Materials	8
IMPROVED REENTRY MATERIALS	33
Introduction	33
Summary	34
Work This Period	35
Evaluate Bifunctional Component Configurations	43
Surface Chemistry Studies	45
THERMODYNAMIC STUDIES	53
Introduction	53
Summary	53
Work This Period	53
HELIUM VENT DEVELOPMENT	56
Introduction	56
Summary	56
Work This Period	56

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.

Additional attention this quarter was given to the planning of activities (and discussions with LASL and ERDA/DNRA staffs) that will directly support LASL's GPHS program in regard to the selection and evaluation of materials for the module capsules that would provide impact protection to the GPHS's fuel capsules. It is anticipated that these discussions will result in a redirection of efforts so that the results of experiments will be directly applicable to the materials being considered in the GPHS conceptual and trial designs.

Summary

During the quarter, the work on development of impact member materials included the examination of the role that tensile constraint can play in the impact protection of the fuel capsule and the examination of result from the high rate impact tests conducted on the Battelle-developed apparatus.

The activities that are examining the role of tensile constraint were directed primarily to the modification of the apparatus and test procedure that was described in the last quarterly report and to the evaluation of two different impact member materials. The interpretation of the results of these tests and tests of additional materials (that will

be of direct interest to the GPHS program) will be made at the end of the test series. However, significant differences have been noted in the test results from the modified apparatus.

The activities that have been developing force-deformation data on present and future impact (member) materials have been directed to the conduct of tests at 150 ft/sec and 285 ft/sec impact velocities with hardened steel and annealed copper mandrels, to the development of analyses for interpretation of instrumentation and to examination of the results from these tests and analyses. Difficulties have been encountered in the position indicator instrumentation; however, resolution is expected during later tests.

The results of tests of ATJ-S graphite materials identified the fact that the trapping of material between the projectile and the annealed copper mandrel does occur but the extent of this trapping and the attendant energy absorption varies with velocity. One estimate (21%) of the momentum involved in the flying ATJ-S particles at impact was also made.

Tests of the MIW GIS (golf-ball wound) material clearly showed that the tensile strength of this material was effective in restraining the lateral deformation of the core it surrounds. Depending upon the specific nature of the surface impacted this could reduce the deformation of fuel claddings to reduce likelihood of rupture if impact occurred against a rough surface.

Work This PeriodDevelopment of Preferred Impact-Member Materials
(G. K. Bansal and J. H. Peterson)

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 previous quarterly reports on this program.

Last period, a test method was developed for evaluating tensile resistance of the impact-member specimens, to provide data for selection of preferred materials systems. This test method consisted of applying an internal pressure to the spherical specimen and determining the displacement of the inside and outside surfaces. Although the approach which is described in the last quarterly report appeared to be viable, additional testing indicated certain deficiencies and the test results may not be meaningful. These problems were primarily associated with the upward movement of the impact member from the base and subsequent extrusion of elastomer into the void produced which then provided an upward force on the bottom surface. A ring of annealed aluminum was placed in this area prevented the extrusion of the elastomer, but did not fully prevent upward movement of the impact member. The upward movement with the ring modification was a result of the bulging of the impact member when under internal pressure. To prevent this movement the test set-up was modified as shown in Figure 1.

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

FIGURE 1. MODIFIED TEST SET-UP UTILIZING INTERNAL PRESSURIZATION

FIGURE 2. INTERNAL PRESSURE-DISPLACEMENT CURVES FOR GOLF BALL WOUND IMPACT MEMBER 7B

Material is 25% Thornel 75 at exterior and 75% graphite yarn WYB 85 on interior.

FIGURE 3. INTERNAL PRESSURE-DISPLACEMENT CURVES FOR GOLF BALL WOUND IMPACT MEMBER 3B

Material is Thornel 50 wound with increased tension.

This modification incorporates the same displacement measurement set-up and the anti extrusion ring. The cut face of the impact member is impregnated with epoxy cement to a depth of about 0.10 in. and this face and the surface that contacts the top plate is cemented to the base and the top plate. The displacement of the inside surface is measured through a small hole by the movement of a 0.062 in. diameter rod. A thin disk 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.

The initial tests with this modified test fixture showed considerably different test results than those obtained previously. The internal pressure required to cause failure was considerably greater, and the external displacement did not begin until a considerable amount of thinning of the impact shell had occurred. Although this may not exactly duplicate the behavior of a full spherical shape under the internal pressurization resulting from fuel deformation, it does more closely simulate the effect of deformation than the previous test set-up.

The pressure-deformation curves for two impact member materials are shown in Figures 2 and 3. Interpretation of these data will not be made until the remaining tests of the special impact member materials are completed. These tests include a standard Thornel 50 impact member and impact members made from Thornel 75 and Thornel 300.

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, fast response instrumentation is required, and unwanted stress wave reflections must be avoided. During the final phase of the impact, 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 previous reporting period the design, construction, and calibration of a new optical motion transducer was completed, together with a preliminary, computer-based data reduction program. During the current period, several instrumented impact tests were conducted as reported here.

In this report we briefly describe the overall apparatus, the data reduction method developed for this test, the results of 3 instrumented tests run at near 150 ft/sec impact velocity, some difficulties encountered with the new motion transducer, their partial resolution, and preliminary

results of two instrumented tests run at near 285 ft/sec.

The overall launcher design is shown in the photographs of Figure 4, with the various parts labeled. The launcher and force transducer are rigidly held in alignment by the 10 in. H-beam used for support. Two 300 kv flash X-ray channels, fired at 155 kv, were used to obtain data (at two times) regarding the target break-up and mandrel deformation. The flash X-ray film cassettes were placed as close as practicable to the target to obtain maximum resolution, with appropriate shields to prevent film double exposure. The X-ray beams were accurately aligned normal to the impact axis at the front surface of target to allow quantitative measurements to be made from the X-rays.

The newly developed data reduction technique takes into account the times required for stress wave travel through the various parts. An impact event is shown schematically in Figure 5. The central portion of the figure shows the impact event depicted on a characteristic or distance-time plot. The left-hand plot shows the output of the position transducer, while the right hand plot shows the output of the load transducer. In practice, both of these outputs are recorded as dual traces on a single oscilloscope photo. The distances and times shown were intentionally distorted from true scale to better show the time-distance relationships. The motion is sensed by the amount of light passing through a milled slot in the launcher barrel as described in the previous quarterly report.* The load is sensed by strain gages bonded to the load bar as shown schematically in the lower portion of Figure 5. Note from the characteristic plot that the position and load transducer outputs are displaced in time from the target front surface by the required stress wave transit times, as indicated schematically by the dashed lines through the projectile, target, and load transducer bar. The front surface impact time is indicated by a small fiducial mark on the load-time curve produced by a capacitor discharge circuit used between the projectile and load transducer bar. The stress wave transit times through the projectile and force transducer bar are nearly constant, easily calculated values, near 12 μ sec for the projectile and 28 μ sec for the load transducer. In

* "Heat Source Component Development Program", Quarterly Report for January-March, 1977, BMI-X-679, April 1977.

a. View From the Accumulator End

b. View From the Force Transducer End

FIGURE 4. PHOTOGRAPHS SHOWING LAUNCHER DESIGN

c. View of the Impact Region

d. View Showing X-Ray Head Locations

FIGURE 4. (CONTINUED)

FIGURE 5. IMPACT EVENT

addition, the transit time through the load transducer was measured in close agreement with the calculation made in a separate experiment. The total initial stress travel time through the target and load bar is measured from the fiducial mark indicating impact to the time of first load appearance on the load-time record.

The initial stress travel time thru the specimen is then found by subtraction of the known stress travel time thru the load bar. The initial stress wave velocity thru the target is then obtained with the aid of the initial target thickness. In the subsequent data reduction, this velocity is assumed constant and the calculated stress wave travel time through the target is reduced proportionately as the target thickness is reduced by deformation. This step required shifting the output of the load transducer by a variable amount as the deformation progresses. In the final outputs, all times were corrected to the target front surface time as a reference.

As shown in Figures 4a and 4b, the interface between the target rear surface and load transducer bar is solidly supported by the long, hardened steel force transducer bar. Nevertheless, the large loads which occur in the impact event produce some motion of this interface. A simple analysis shows that the displacement of this interface at any time is proportional to the integral of the force-time pulse up to that time. The constants of proportionality are known from first principles, allowing the rear surface motion to be found as a function of time by progressive numerical integration of the recorded load-time pulse.

In practice, the data reduction technique consists of digitizing sufficient data points from the load and position curves to allow accurate linear interpolation between points. These data are input to a computer program, "DYNDEF", written for this purpose, together with other data pertinent to the experiment. The computer program makes the necessary time adjustments, applies the load and position calibration factors, calculates the force-time integral, target front and rear surface displacements and target deformation as a function of time. The curves shown in this report are outputs from this computer program.

During this report period, a total of five fully instrumented tests were conducted as summarized in Table 1. In these tests

TABLE 1. SUMMARY OF INSTRUMENTED IMPACT TESTS

Shot No.	Target	Support Mandrel	Velocity Impact ft/sec	Approx Max Load on Target lb	Remarks
21	ATJ Graphite	Hardened Steel	150.6	13,400	Load dropped to zero without stopping projectile. Projectile finally arrested by impact with steel mandrel generating a peak load of 54,300 lb resulting in deformation (<.01") of both projectile and mandrel.
22	MHW GIS	Hardened Steel	149.6	37,800	Projectile arrested after a maximum target deformation of 44.6%. No deformation of either projectile or mandrel. 14
23	ATJ Graphite	Annealed Copper	149.6	6,900	Initial gap, near 0.06" between target and mandrel. Load dropped to zero without stopping projectile. Projectile finally arrested by impact with Cu mandrel deforming only the mandrel. Peak final load probably greater than 70,000 lb - off scale.
29	ATJ Graphite	Annealed Copper	281.7	28,500 39,300	Initial maximum load on target, then dropping to 17,400 lb/min and rising again to 2nd maximum load. Projectile motion arrested at 2nd maximum load with contact against Cu mandrel.
30	MHW GIS	Annealed Copper	288.4	(78,000 - 88,000)	Load in one large pulse stopped projectile w/o metal-to-metal contact. Peak load-off scale. Values given represent approximate limits of extrapolation.

the carbon/carbon targets were complete spheres, except for the access plug removed from the MHW GIS targets. The ATJ graphite targets were machined to the same dimensions as the GIS.

A check of the deformations measured from the flash X-rays on Shots 21 and 22 with those calculated from the original data reduction based on static calibration of the motion transducer showed that a discrepancy existed. Subsequently, additional calibration shots were fired without targets which showed that the calibration of the motion transducer was effected by condensation of moisture in the driver gas behind the projectile. This moisture originated primarily from the moisture in the room air which was included in the gas pressure accumulator. Its condensation was produced by the rapid expansion and cooling of the gas during projectile acceleration. This condensation or fog partially obscured the light passing through the barrel slot of the optical transducer thus reducing the sensitivity of the transducer and producing a nonlinear response by an amount depending on the amount of condensation present which varied from shot-to-shot. Hence, rather than using the static transducer calibration, a partial dynamic calibration (during each of Shots 21 and 22) was obtained as follows.

Using the velocity of the projectile as measured by the trigger pins and electronic counters as described previously, the initial motion transducer sensitivity was obtained from the measured slope of the transducer output trace before impact with the target. The deformations measured from the two flash X-radiographs became point checks on this basic sensitivity at two times after impact.

In Shot 21, all three measurements agreed within the experimental error of ~ 0.005 inch under the assumption that the transducer output continued linearly over the whole experiment time (but was different from the static calibration). In Shot 22, the three measurements indicated a slightly nonlinear, progressively decreasing transducer sensitivity. However, a linear fit of the motion transducer output produced an approximate fit with indicated total errors of 0.025 inch or less, which is somewhat outside the limits of experimental error but should nevertheless

produce a reasonable representation of the impact event. Calibration at only two fixed points after impact of a nonlinearly responding transducer leads to some uncertainty in absolute accuracy of the reported results, however the objective of comparison of target materials was amply satisfied as will be shown in the presentation of the data.

Control of the moisture content of the driver gas by pre-purging of accumulator volume, together with preshot dynamic calibrations (no target firings) lead to a much firmer basis for the interpretation of the records of Shot 29 and 30 to be reported in detail in the next quarterly report.

The postshot appearance of the MHW GIS on the force bar after Shot 22 is shown in Figure 6. As shown, the deformation of the GIS was restricted nearly to the impacted zone. By contrast, the ATJ was shattered, leaving behind a bare mandrel, like that shown in Figure 7, taken after Shot 23 which also illustrates the relatively small copper mandrel deformation which occurred at an impact velocity of 149.6 ft/sec.

Similarly, the ATJ mandrel and GIS mandrel appearance after impacts at near 285 ft/sec are shown in Figures 8 and 9. Note the increased damage to both as compared to impacts near 150 ft/sec. Also notable is the flat front of the ATJ mandrel as shown in Figure 9 produced by the final projectile-mandrel contact after the ATJ shatter. The difference in damage to the copper mandrel and the degree of ATJ fracturing as a function of impact velocity are further illustrated in Figures 10 and 11. In each of these shots, the steel projectile face was undeformed. The small, near-round spot on the face of the projectile is a lenslet of graphite which was compressed at high pressure during the impact and remained adherent to the face of the projectile, though it was easily removable.

The postshot appearance of the MHW GIS target after impact at 288 ft/sec is further illustrated in Figure 12 to clearly show the nature of the fractured volume of composite which fell away after the impact. Note that the surface of the copper mandrel remains well below the remaining GIS material, further demonstrating that no mandrel-projectile contact occurred during this impact.

FIGURE 6. POST SHOT APPEARANCE SHOT 22 MHW GIS IMPACTED ON STEEL MANDREL AT 149.6 FT/SEC

FIGURE 7. POST SHOT APPEARANCE SHOT 23 ATJ GRAPHITE ON ANNEALED COPPER MANDREL AT 149.6 FT/SEC

FIGURE 8. POST SHOT APPEARANCE SHOT 29 ATJ GRAPHITE ON ANNEALED COPPER MANDREL AT 281.7 FT/SEC

FIGURE 9. POST SHOT APPEARANCE SHOT 30 MHW GIS ON ANNEALED COPPER MANDREL AT 288.4 FT/SEC

FIGURE 10. POST SHOT APPEARANCE SHOT 23 ATJ GRAPHITE ON ANNEALED COPPER MANDREL AT 149.6 FT/SEC

FIGURE 11. POST SHOT APPEARANCE SHOT 29 ATJ GRAPHITE ON ANNEALED COPPER MANDREL AT 281.7 FT/SEC

FIGURE 12. POST SHOT APPEARANCE SHOT 30 MHW GIS ON ANNEALED COPPER MANDREL AT 288.4 FT/SEC

FIGURE 13. POST SHOT APPEARANCE OF MANDRELS

<u>Shot</u>	<u>Target</u>	<u>Mandrel</u>	<u>Velocity, ft/sec</u>
22	MHW GIS	Steel	149.6
29	ATJ Graphite	Copper	281.7
30	MHW GIS	Copper	288.4

Deformations are shown in parentheses

FIGURE 14. DEFORMATION OF COPPER MANDRELS AFTER ~285 FT/SEC IMPACTS

FIGURE 15. DEFORMATION - TIME CURVE FOR SHOT 21 MHW GIS ON HARDENED STEEL MANDREL IMPACTED AT 150.6 ft/sec.

FIGURE 16. LOAD - TIME CURVE FOR SHOT 21.

FIGURE 17. LOAD - DEFORMATION CURVE FOR SHOT 21.
THIS CURVE IS DERIVED FROM THE DATA OF
FIGURES 15 and 16.

TABLE 2. SUMMARY OF MOMENTUM EXCHANGES

	Momentum, lb-sec	Projectile Calculated	Velocity, ft/sec Measured	Percent of Impact Momentum
<u>Shot 21</u>				
Initial Projectile Velocity	4.68		150.6	
Transmitted through ATJ during impact	<u>1.30</u>			
Difference	3.38			
Residual Projectile Vel. after impact with ATJ	<u>2.38</u>		76.7	
Momentum to ATJ Particles (a)	1.00	96.1 (b)		21.4
Projectile Velocity at Impact with Mandrel	2.18		70.2	
Transmitted through mandrel during impact	<u>3.6</u>			
Difference	-1.4	-45.		
Residual Projectile velocity after mandrel impact	<u>-2.04</u>		(-66)	
Error in Final Balance	- .64			14.0
<u>Shot 22</u>				
Initial Projectile Velocity	4.65		149.6	
Transmitted through GIS during impact	<u>5.38</u>			
Difference	-0.73	-21.		
Residual Projectile Velocity After Impact	<u>-1.36</u>	(-44)		
Error in Final Balance	-0.63(c)			13.5

(a) By difference

(b) Assuming all ATJ mass (152 gm.) accelerated to uniform velocity

(c) Negative sign indicates more momentum accounted for by transmission and rebound than present initially.

The relative deformations of the copper mandrel which occurred during ~ 285 ft/sec impacts on ATJ, Shot 29, and MHW GIS, Shot 30, are shown in Figure 13. The steel mandrel, used in Shot 22, is undeformed and shown for comparison. The vertical marks near the bottom of the smooth portion of the copper mandrels were made by the pipewrench used to unscrew the mandrels after the test. As shown, the mandrel from Shot 30 required considerably more force to remove than that of Shot 29. The deformations of the two copper mandrels are shown quantitatively in Figure 14. Note that, at the first full-diameter section, the lateral deformation of the MHW-GIS - protected mandrel is appreciably less than that of the ATJ-protected mandrel. Further down the mandrel, however, the GIS-protected mandrel suffered larger deformations. This was probably because of the higher load on the mandrel produced by stopping the projectile short of metal-to-metal contact in the GIS impact, as compared to the ATJ impact in which final projectile arrest occurred as a result of its direct contact with, and deformation of, the mandrel.

Figures 15, 16, 17 present the measured load-time, deformation-time, and load-deformation curves, respectively, for Shot 21, an ATJ target impacted at 150.6 ft/sec on a hardened (R_c 42-46) steel mandrel. Reference to Figures 15, 16 shows that the projectile continued to move toward the steel mandrel after disappearance of the load. The projectile later impacted the now bare mandrel producing deformation of both steel parts. A peak load on the steel mandrel of $\sim 53,900$ lb was recorded ~ 455 μ sec after impact, and did not go positive until ~ 380 μ sec after impact. The projectile recoiled from impact with the mandrel as measured from the slope of the position time curve after impact at a velocity of -66 ft/sec. The momentum balance for the two successive impacts of Shot 21 is summarized in Table 2 along with similar information for Shot 22. According to the momentum balance as shown in Table 2, the fractured particles of ATJ graphite carried away approximately 21% of the initial projectile momentum. However, the accuracy of this value is in some question since the overall balance included a 14% error. This error is not considered excessive, however, considering the relatively short line segments available for slope (velocity) measurement, the noise superimposed on the load record, and the incomplete dynamic calibrations available for the position transducer.

Figures 18, 19, 20 present the measured load-time, deformation-time, and load-deformation curves respectively for Shot 22, a standard MHW GIS impacted at 149.6 ft/sec on a hardened steel mandrel. Figure 18 shows that the projectile was arrested and rebounded after impact with the GIS material. Hence Figure 20 has peculiar form due to the projectile rebound. Figure 21 presents prints of flash X-rays taken at nearly identical times after impact for Shots 21 and 22. Figures 15-21 show that the GIS load transfer was much greater than that of the ATJ, and that the GIS prevented metal-to-metal contact between projectile and mandrel. As shown in Table 2, the momentum transferred during GIS impact was $5.38/1.30=4.14$ times greater than during ATJ impact.

The remaining instrumented tests were performed with targets on annealed copper mandrels which deformed during impact testing. The deformation of the mandrel cannot be readily separated from the deformation of the impact protection member and hence complete analysis of the type conducted on Shots 21 and 22 is not possible.

In Shot 23, a small gap of perhaps one-sixteenth inch between the graphite and the mandrel was discovered in the first flash X-radiograph taken of the experiment. The graphite target was apparently prevented from seating fully when placed on the mandrel by a slight interference on the rear portion of the graphite. Since this was not anticipated *á priori*, no provision was made on this test to guarantee it would not occur. In all later tests micrometer measurements verified that the targets were completely seated. The records from this test showed a gradually rising transmitted load which did not start to rise above the baseline, until $\sim 80 \mu\text{sec}$ after impact, and then rose to a maximum of only 6,900 lb during the next 130 μsec . A smooth decrease to zero load followed this low peak, followed by the secondary impact of the projectile against the now essentially bare copper mandrel.

The data from Shots 29 and 30 at near 285 ft/sec against ATJ and an MHW GIS on annealed copper mandrels respectively were not fully reduced during this report period. The results these shots show, however are qualitatively similar to Shots 21 and 22 run at a lower impact velocity against hardened steel mandrels. The principal qualitative difference in the ATJ shot, which is at least partially due to the higher impact

FIGURE 18. DEFORMATION - TIME CURVE FOR SHOT 22
MHW GIS ON HARDENED STEEL MANDREL IMPACTED
AT 149.6 ft/sec.

FIGURE 19. LOAD - TIME CURVE FOR SHOT 22.

FIGURE 20. LOAD - DEFORMATION CURVE FOR SHOT 22
THIS CURVE IS DERIVED FROM THE DATA OF
FIGURES 18 AND 19.

a. Shot 21 ATJ on Hardened Steel 248 μ sec After Impact Load has Already Fallen to ~ 0 . Projectile Velocity ~ 77 ft/sec.

b. Shot 22 MHW GIS on Hardened Steel 238 μ sec After Impact. Load is ~ 5700 lb, Projectile Starting to Rebound.

FIGURE 21. FLASH RADIOGRAPHS SHOWING DIFFERENCE IN BEHAVIOR OF ATJ AND GIS TARGETS

velocity, is that the load does not drop to zero after the initial maximum produced during break up of the ATJ, as indicated in Table 1. Also, due to the higher impact velocity, the load on the GIS was much higher and in fact went off scale at $\sim 75,000$ lb. From the recorded rise and fall of the pulse the probable limits of maximum load are 78,000-88,000 lb as shown in Table 1. The flash-X-rays show that the copper deformation began well before the first peak in load was reached in the ATJ shot which would also contribute to better trapping of the broken graphite and prevention of the load falling to zero.

In summary, these tests clearly show that the tensile strength of the MHW GIS material is effective in restraining lateral deformation of the core which it surrounds and in retaining the crushed, impacted fibers between core and impact face to provide a cushion against cladding rupture by potentially rough jagged impact surfaces. In contrast, the bulk ATJ graphite shatters early in the impact event carrying away a small fraction of the impact momentum but allowing greater lateral deformation of the core and final direct contact (with accompanying core deformation) between the core and impact face. This latter situation would be likely to cause cladding rupture if impact occurred against a naturally rough surface.

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 were reviewed during this reporting period and a revised set of tasks and subtasks in support of the GPHS program plan was defined in detail.

Summary

Efforts during this quarter in this Improved Reentry Materials Task were primarily directed toward defining technical activities to be performed by BCL in support of LASL on the development of the GPHS. These activities were focused on Task 3, Conceptual Designs, and Task 4, Trial Designs, of the GPHS Design Schedule^{*}, and were formulated to ensure technology readiness of the GPHS module reentry member by March, 1978. A work plan (program schedule) and associated budget requirements were also prepared. The technical activities and work plan were discussed in detail with LASL personnel and were reviewed with DNRA/ERDA and JHU/APL personnel.^{**} Work on these activities has not been initiated pending approval by LASL and authorization by DNRA/ERDA.

Nominal efforts were conducted in support of the evaluation of the bifunctional component configurations. These efforts were limited to a brief review of the films of the LCHPG heat source drop test conducted at NASA Wallops Island on August 30 and 31, 1976, and a brief review of pertinent work conducted by Sandia-Albuquerque, and preparation of a summary of the available thermophysical property data for carbon-carbon composites for the reentry thermal and thermal stress analyses.

Further progress was made in the surface chemistry studies regarding the assembly and checkout of equipment needed for the experiments. Technical papers on graphite surface chemistry were reviewed to determine the relevancy of the results obtained and the experimental technique used to the current measurements.

* See General Purpose Heat Source Program Plan, Prepared for DNRA/ERDA by LASL, CMB-5-77-873; Rev. 2, 31 March 1977 for Task designations.

** These task activities have been prioritized by LASL in terms of their relation to the GPHS program.

Work This PeriodTechnical Planning in Support of GPHS Program Plan (I. M. Grinberg)

Detailed technical planning activities were conducted during this reporting period in support of the GPHS development program. The effort included preparation of a work plan, in which specific activities to be conducted by BCL were defined, and an associated activities schedule. These activities were reviewed with LASL GPHS project personnel and were subsequently modified by BCL, based on these technical discussions, to be more compatible with the GPHS Program Plan and Schedule. They were subsequently reviewed in detail with LASL personnel. Given below is a brief description of these subtasks. Figures 22 and 23 are schematics which show the phasing of the subtask activities within each task. Calendar dates have been omitted from the schedules pending authorization from DNRA/ERDA to proceed on specific subtask activities.

Subtask 3.1A^{*} Identify GPHS Conceptual Designs. The objective of this subtask is to identify and select GPHS module conceptual designs and determine how these designs influence the choice of the reentry member material and geometry. LASL has primary responsibility for this subtask with BCL providing input.

The results of this subtask will be candidate GPHS conceptual designs/materials concepts. These results will be used in the remaining subtasks to provide a basis for the ranking of materials for the conceptual and trial designs by BCL and the subsequent selection of trial designs by LASL in Task 4.

Subtask 3.2C Fabrication Analysis and Cost Evaluations. The objectives of this subtask are to (1) provide preliminary fabricability analysis of heat shield materials/member concepts and cost evaluations for input to Subtask 3.3A, and (2) provide in-depth fabricability analysis and cost evaluations of heat shield materials/member concepts to guide the ranking of materials for the trial designs, Subtask 3.7A. This subtask is the prime responsibility of BCL.

* Subtask designations formulated for the combined Improved Impact Member and Improved Reentry Materials activities.

FIGURE 22. TASK 3. CONCEPTUAL DESIGN - REENTRY MEMBER (PROPOSED)

FIGURE 23. TASK 4. TRIAL DESIGN - REENTRY MEMBER (PROPOSED)

The results of this subtask will be information on fabricability and evaluations of cost for the conceptual GPHS module designs formulated in Subtask 3.1A and for the ranking of materials in Subtask 3.7A based on the analysis of Subtask 3.6A.

Subtask 3.3A Select Models for Thermal/Ablation and Thermal Stress Analyses. The objective of this subtask is to select and formulate the appropriate approaches to be used for modeling the thermal/ablation and thermal stress response of candidate GPHS heat shield materials. Primary responsibility for this subtask lies with BCL.

The results of this subtask will be the selection and formulation of thermal/ablation and thermal stress response models for analyzing, screening, and ranking candidate GPHS heat shield materials.

Subtask 3.4A Input Properties and Parameters. The objectives of this subtask are to (1) determine and/or obtain the operating and initial conditions and materials property data required for the GPHS module screening analyses and (2) select the analysis matrices for the thermal/ablation and thermal stress screening response modeling. Primary responsibility for this subtask will be with BCL.

The results of this subtask will be (1) the material property data and the aerodynamic and aerochemical information required to conduct the thermal/ablation and thermal stress screening analyses and (2) formulation of thermal/ablation and thermal stress screening analysis matrices identifying the specific material properties and their variations (quality-control requirements) and other variables (environmental/operational) to be considered in the analysis.

Subtask 3.5A Surface Chemistry Study. The objective of this subtask is to determine the surface chemistry characteristics of GPHS heat shield materials under subsonic reentry conditions in order to provide an improved criterion for specifying the heat source module heat shield (ablator) thickness. Battelle will have prime responsibility for this subtask.

The results of this subtask will be (1) correlations of surface chemistry, i.e., species composition and heat generation, for candidate

GPHS heat shield materials and (2) incorporation of these correlations into the thermal/ablation modeling analysis.

Subtask 3.6A Analytical Screening of GPHS Modules/Materials.

The objectives of this subtask are to (1) determine the thermal ablation, and thermal stress performance of GPHS heat shield members for the conceptual designs identified in Subtask 3.1A and (2) refine the conceptual designs based on the reentry response predictions consistent with GPHS performance requirements. This subtask is the prime responsibility of BCL.

The results of this subtask will consist of (1) ablation performance (recession) and temperature histories of a variety of candidate graphitic heat shield materials, (2) detailed thermal stress and strain profiles within the heat shield member at critical reentry times and comparisons of these levels with either failure stresses or strain to failure, (3) quantitative effect of impact member and/or other insulation material on clad temperature, and (4) recommendations of GPHS module trial designs which satisfy system performance and safety requirements.

Subtask 3.7A Ranking of Materials for Trial Design. The objectives of this subtask are to (1) rank candidate graphitic materials according to thickness required for reentry and the temperature limits experienced by the metal containment member for the GPHS module and (2) rank candidate graphitic materials according to thermal stress resistance. Prime responsibility for this subtask will be with BCL.

The results of this subtask will be a ranking of graphitic materials for use as heat shield members for the GPHS module based on (1) material thickness required for ablation, (2) maintenance of satisfactory containment material temperatures, (3) resistance to thermal stress failure, (4) fabricability and (5) integration into a GPHS module design which meets the design and performance requirements and safety specifications. These results will provide the basis for the selection of trial designs for further evaluation.

Subtask 3.8A Reporting. Reports and presentations on the results of the subtask activities of Task 3.

Monthly management-type letter reports to ERDA-NRA and LASL GPHS program manager.

Quarterly progress report of technical results and progress of activities on subtasks.

Summary topical reports as appropriate to report significant results of technical activities.

Oral presentations as needed to relate current results of task activities to ERDA-NRA and LASL GPHS program management. Input for LASL's report on BCL's Task 3 activities up to September 30, 1977, will be prepared and submitted to LASL.

Subtask 4.1A Select Trial Designs. The objective of this subtask is to select GPHS module trial designs for further evaluation. LASL has the prime responsibility for this subtask with BCL providing input.

The results expected from this subtask will be GPHS module trial designs for further evaluation.

Subtask 4.2A Define Test Requirements. The objective of this subtask is to define the test requirements for evaluation of the GPHS module trial designs and heat shield materials. LASL will have the prime responsibility for this subtask.

The results of this subtask will be the definition of the tests and conditions for evaluating the thermal, ablative, and thermal stress response of GPHS module trial designs and materials.

Subtask 4.3A Fabrication of Test Articles. The objective of this subtask is to procure, fabricate, and assure the quality of specimens for the tests of Subtasks 4.4A and 4.4B. LASL will have the prime responsibility for this task.

The results of this subtask will be specimens fabricated from two to three candidate impact and heat shield materials for impact, ablation, surface chemistry, thermal stress, thermophysical and mechanical property tests. Approximately six specimens of each material will be fabricated.

Subtask 4.3B Design Data and Quality Control. The objectives of this subtask are to obtain design data and specifications, and to identify quality control and nondestructive evaluation procedures required for final design and acceptance of impact and heat shield materials.

The results of this task will be design data for use by the designers and material specifications for the graphite and carbon-carbon composite materials needed for the reentry and impact members of the GPHS. Information on material quality control in terms of properties and performance characteristics will be associated with results of selected nondestructive evaluations.

Subtask 4.3C Fabrication Analysis and Cost Evaluation. The objective of this subtask is to provide information on the fabricability and associated cost of the GPHS module trial designs and materials. Emphasis will be placed on the heat source module graphitic-based heat shield (including the ablator) and insulation (if any) members. This subtask is the prime responsibility of BCL.

The results expected from this subtask will be information on the fabricability and associated cost of trial design GPHS module concepts and materials.

Subtask 4.4A Tests. The objective of this subtask is to provide data on the response of candidate heat shield materials and GPHS module trial design configurations to environmental conditions that simulate those conditions expected during reentry of the GPHS module. Prime responsibility for this task will be with LASL.

The results expected from this subtask will be property and performance data for candidate heat shield materials and GPHS module trial design concepts. This information will be used to analyze the overall response of the GPHS module to postulated abort conditions and to select a prototype design.

Subtask 4.5A Model Refinement. The objective of this subtask is to refine the GPHS module reentry performance models, as required, based on the test results of Subtask 4.4A. BCL will have prime responsibility for this subtask.

The results of this subtask will be improved performance models to determine the thermal, ablation, and thermal stress response of the GPHS module trial designs to reentry conditions.

Subtask 4.6A Performance Analysis. The objective of this subtask is to determine by analysis the performance of the GPHS trial designs and materials during normal operation and reentry. The results will provide the basis for the selection of a prototype design in terms of the ablation, thermal and thermal stress response of the heat source module, which will satisfy the performance requirements and safety specifications. BCL will have prime responsibility for this subtask.

The results of this subtask will be information on the response of GPHS module trial designs and materials to normal operation and reentry conditions. This information plus materials fabricability and cost analysis input will serve as the basis to select the GPHS module prototype design.

Subtask 4.7A Reporting. Reports and presentations on the results of subtask activities of Task 4.

Monthly management-type letter reports to ERDA-NRA and LASL GPHS program manager.

Quarterly progress reports of technical results and progress of activities on subtasks.

Summary topical reports as appropriate to report significant results of technical activities.

Oral presentations as needed to relate current results of task activities to ERDA-NRA and LASL GPHS program management.

Input for LASL's report from BCL's Task 4 activities up to June 30, 1978, will be prepared and submitted to LASL.

Subtask 5.1A Select Prototype Design. The objective of this subtask is to select the GPHS module prototype design. Prime responsibility for this subtask will be with LASL, with BCL providing significant input.

The results expected from this subtask will be inputs to the selection of a prototype design for the GPHS module. These inputs will be related to selection of module geometries and graphitic-based heat shield and insulator materials.

Evaluate Bifunctional Component Configurations (R. E. Hess)

Available data relative to the aerodynamic stability and impact velocity of rectangular shaped configurations were briefly reviewed to provide baseline information for the reentry analysis of GPHS modules. On the basis of data generated by Sandia in 1967 and 1968^{*,**} on rectangular-shaped flat plates, the subsonic reentry characteristic can be described as autorotation, that is, rotation about an axis normal to the airstream, which arises independently of the initial forcing function. The transient behavior is rather complex with an initial oscillatory phase prior to achieving the autorotation or tumbling mode. During autorotation, there is a precession of the plate relative to the flight path. Observations of the motion picture films of the LCHPG heat source drop test^{***} confirms the subsonic aerodynamic instability of these rectangular heat source configurations and the general tumbling behavior obtained by Sandia in the earlier tests.

On the basis of available test information, it appears that for the reentry thermal and thermal stress analyses, GPHS module configurations similar to those tested by Sandia and TES can be treated as maintaining broadside stability at hypersonic and supersonic velocities and random tumbling subsonically. However, "worst case" assumptions in terms of required heat shield thickness, thermal stress level, and clad temperature at impact will be used in order to satisfy the safety requirements.

The accumulation of thermal properties has been conducted at a nominal effort. During this reporting period, thermophysical properties for six Pyrocarb formulations - 406, 408, 409, 601, 901, and 903 were documented. Among the thermophysical properties of these materials, the thermal conductivity exhibits the greatest variation from material to material and as a function of temperature for a given material. Also for these two-dimensional materials which do not exhibit isotropy, different values must be used along and perpendicular to the lamina to adequately describe the thermal conductivity. Specific

* Bustamante, A. C., Stone, G. W., Jr., Autorotational Characteristics of Flat Plates and Right Circular Cylinders at Subsonic Speeds, SC-RR-67-778, November, 1967, Sandia Laboratories, Albuquerque, New Mexico.

** Bustamante, A. C., Free-Fall Rotation and Aerodynamic Motion of Rectangular Plates, SC-RR-68-132, August, 1968, Aerospace Nuclear Safety Department, Sandia Laboratories, Albuquerque, New Mexico.

*** The motion picture films were provided by R. Anderson, Teledyne Energy Systems, Timonium, Maryland.

heat, density, and emissivity are much less sensitive to changes from material to material and show less variation with temperature. Sufficient thermophysical property data are available-with judicious extrapolation-for reentry thermal analysis of these materials over the expected temperature range. Properties of other prospective graphitic GPHS heat shield materials are being assembled and a tabulation will be provided in the next quarterly report.

Surface Chemistry Studies (A. A. Boiarski)

Introduction. The objective of this subtask is to determine the boundary layer surface chemistry on candidate heat shield members at simulated reentry conditions. These data, when combined with a suitable theoretical model, will result in an improved criterion for specifying the heat shield (ablator) member thickness by removing ambiguities in the heat source design due to uncertainties in the surface oxidation process.

Current Progress. During this reporting period, some earlier published work was reviewed to determine its suitability for defining heat source ablator member oxidation. Also, the major electro-optical components of the Raman scattering apparatus were tested as a system and the need for a Pockels cell Q-switch was identified.

In the experimental work by Wicke and Wurzbacher^{*}, a quartz micro-probe was used to extract samples of gas at different distances from a carbon surface burning in a stream of oxygen. Thus, concentration profiles of oxygen, carbon monoxide, and carbon dioxide were obtained at various wall temperatures from 1000-1230 C using dry oxygen. Also, these profiles were obtained for varying amounts of water vapor (.0062-1.01 percent) mixed in the oxygen for a constant wall temperature of 1180 C. In the paper by Hugo, et al,^{**} the experimental work was interpreted assuming homogeneous combustion in order to determine the relationship between the concentration profiles and the important thermochemical and mass transfer processes.

The microprobe technique was examined to determine if it could complement or possibly replace the Raman scattering technique to determine wall chemistry. One possible method of obtaining microprobe data is shown schematically in Figure 24. A 0.5 mm diameter quartz probe could be inserted from the rear of an inductively heated graphitic sample.

* "Concentration Profiles Near a Carbon Surface Burning in a Stream of Oxygen - I. Experimental Results", Wicke, E., and Wurzbacher, G., Int. J. Heat Mass Transfer, Vol. 5, pp 277-289, 1962, in German.

** "Concentration Profiles Near a Carbon Surface Burning in a Stream of Oxygen -II". Calculation of the Concentration Profiles", Hugo, P., Wicke, E., and Wurzbacher, G., Int. J. of Heat and Mass Transfer, Vol. 5, pp 929-939, 1962, in German.

FIGURE 24. SCHEMATIC DIAGRAM OF A POSSIBLE METHOD TO MEASURE WALL OXIDATION CHEMISTRY USING A QUARTZ MICROPROBE

A hole would be required in the sample that was slightly larger than the probe diameter. The resolution of such a probe is at least 0.2 mm,^{*} so approximately 10 locations could be sampled in an estimated boundary layer thickness of 2 mm, including a sample very near the wall surface. The probe would have to be located on a traversing mechanism to hold the probe tip position constant during the sampling period since the graphitic surface would be receding due to combustion. Providing that the probe did not disturb the boundary layer flow or alter the wall chemistry due to catalytic reactions on the probe surface^{**} CO₂, CO, N₂ chemistry data could be obtained using this technique. A decided disadvantage to the microprobe method is that boundary layer temperature measurements cannot be obtained. Raman scattering, on the other hand, will yield both chemical composition and temperature data simultaneously. The Raman technique is also applicable at higher temperatures than the microprobe method since quartz will begin to soften above about 1800 C. Another disadvantage of the microprobe technique for concentration profiles is the time required to extract a sample. Times of the order of 10-15 seconds may be required and a sample or probe feeding mechanism may be required to maintain a relatively constant distance between the tip of the probe and the receding graphitic surface during the time the sample is being taken.

It appears that the microprobe technique, by itself, is not suitable to determine the species concentration profiles in the boundary layer. Rather, a combination of microprobe analysis at the wall and Raman scattering analysis in the boundary layer may be the best approach. The two techniques would then complement each other since scattering data at the wall could not be obtained due to the fact that laser light would scatter from and/or fluoresce surface material if the laser were focused too close to the wall.

An interesting aspect of the Wicke and Wurzbacher results was the high degree of dependence of CO concentration at the wall on water vapor content of the incoming oxygen stream. Further study will be devoted to this observation in order to assess its importance on reentry member heat shield wall chemistry since water vapor will always be present in the lower atmosphere in sufficient quantities to affect the graphite oxidation process.

* Not expected for quartz probe.

** Ibid.

Major parts of the electro-optical apparatus required to perform Raman scattering measurements were tested as a system during this reporting period (refer to Figure 22, p 56, in the Quarterly Report of October-December, 1976, for schematic of setup)*. A photograph of the pulsed Ruby laser is shown in Figure 25. The various components are identified in the photograph and a beam path is indicated. The frequency doubling crystal, which is not shown in the figure, is positioned on the optical rail between the front mirror of the laser and the external quartz prism. This prism is required in order to separate the desired blue $3472 \text{ \AA}$ radiation from the undoubled red laser light (see Figure 25). A helium-neon laser is used to align the ruby laser beam path with the remaining optical components.

Additional components of the experimental setup are shown in Figure 26. The unwanted red beam from the laser is intercepted by a polished black glass plate which absorbs 90 percent of the incident radiation. The remaining 10 percent of the red laser light is nearly all specularly reflected from this plate into a beam-absorbing device (device #1) made of two pieces of black glass set at a specific angle to each other. This laser absorber is designed such that the incoming light undergoes multiple reflections within the device so that only a very small amount of red laser radiation escapes to interface with the weak Raman scattering signal. The desired blue light misses the black glass plate and, after passing through a focusing lens (L1), is rotated 90 degrees, by a quartz internal reflecting prism. This prism directs the beam up toward the graphitic sample. Lens L1 is required to focus the doubled beam to a point on the centerline and just off the surface of the inductively heated graphitic sample. The beam passes through the sample region and is collected by another beam absorbing device (#2) identical to the first. Again, only a very small amount of the incident beam escapes from this absorbing device. Note also in Figure 26 the orientation of the air blower exit with respect to the graphitic sample.

The remaining major components of the Raman scattering apparatus are shown in Figure 27 before they were installed near the graphite sample.

*"Heat Source Component Development Program," Quarterly Report for October-December, 1976, BMI-X-676, January, 1977.

FIGURE 25. COMPONENTS OF RUBY LASER AND ASSOCIATED BEAM SEPARATING AND ALIGNMENT SYSTEM

FIGURE 26. OPTICAL COMPONENTS TO DELIVER THE DESIRED BEAM TO THE MEASUREMENT REGION AND DISCARD UNWANTED LIGHT

FIGURE 27. LIGHT COLLECTION, SPECTRAL ANALYSIS, AND DATA RECORDING COMPONENTS OF THE RAMAN MEASUREMENT SYSTEM

Raman scattered light is collected by lens L2 and focused onto the slit of a 3/4 meter spectrograph. At the exit plane of this instrument, an Optical Multi-channel Analyzer (OMA) is placed. The OMA converts the spectrally analyzed Raman photon flux into an electronic signal which is then stored on digital tape for further analysis. A standard lamp whose intensity as a function of wavelength was calibrated by the National Bureau of Standards was used to determine the variation in output signal across the OMA sensing screen. It was found to be ± 5 percent across 80 percent of the screen central region. A hollow copper cathode lamp was also used to determine the dispersion of the spectrograph and calibrate the wavelength dial of the instrument against the known copper spectrum.

The timing circuitry was installed to fire the laser and simultaneously turn on the OMA for a gate period of 50 nanoseconds to record only the Raman scattered radiation. A trigger pulse from the OMA itself was used to initiate the firing sequence. During this checkout, a significant variation in the time between the initiation of the laser trigger pulse and the appearance of the dye cell Q-switched output pulse was discovered. This time variation implied that a much broader gate period of approximately 200 μ sec would be required to ensure that the laser-excited Raman signal fell within the OMA recording period. This increased gate time would negate the advantage of using a blue laser in order to avoid background radiation from the hot graphitic sample. Hence, only surface chemistry for samples at low wall temperatures (i.e., $T_w < 1100$ C) could be examined with this circuitry. This variation in laser firing time is associated with the dye cell Q-switch presently employed to obtain the giant pulse laser output. A solution to the problem would be to use a Pockels cell Q-switch. The latter device will produce 10-15 nano-second laser pulses with only a small time variation which is less than the minimum OMA gate width. This remedy will be adopted as soon as possible. Minimum project delay should occur since the lower temperature data can be obtained with the present Q-switch device.

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 a mechanistic nature.

Summary

Indications have been obtained that previously unobserved gaseous molecules exist in the iridium-²³⁸PuO₂ system that might lead to unexpected long-term deleterious effects. Individual component of the total system have been analyzed to determine their total contribution toward vapor species and vapor transport of the components in the iridium-²³⁸PuO₂ system.

Work This Period

Experiments this quarter have centered on determining the possible existence of carbonyl compound formation which might lead to iridium transport.

The gases which make up the atmosphere surrounding the total system contain carbon monoxide. This gas mixture is in contact with the fuel cladding and the GIS and therefore has the opportunity to react.

The first transpiration experiment was conducted by placing iridium metal on a graphite block in a fused quartz tube as shown in Figure 28. The tube was evacuated and then back filled with carbon monoxide. The iridium and carbon were heated to 1200 C for 240 hours (with very little temperature gradient across the sample). Analysis of the sample afterward revealed no change of weight or appearance. This experiment was repeated with an elongated sample where the temperature gradient was established (1200 C to 800 C). The analysis of this sample showed no detectable

FIGURE 28. IRIIDIUM CARBONYL EXPERIMENT FURNACE

weight change, but there was an etched effect at about the 900 C point of the gradient. Electron probe analysis of the fused quartz and of the graphite failed to detect any iridium that would indicate transport. These data indicate that if the iridium carbonyl does exist the partial pressure of $\text{Ir}(\text{CO})_3$ is less than 10^{-10} atm.

Additional experiments are being carried out in the Nuclide mass spectrometer where the iridium is placed in a graphite Knudsen cell. Magnesium oxide is also added to provide a source of carbon monoxide by the reaction of the carbon and magnesia. This experiment certainly formed carbon monoxide but the existence of iridium carbonyl was not detected, probably because the carbon monoxide was depleted from the cell too soon. This experiment indicates that the iridium carbonyl does not exist under these conditions. However additional experiments which include the total system (fuel, fuel impurities, cladding, cladding impurities, and the GIS) are being designed. Another experiment is planned to increase the total oxygen pressure of the system by adding a carbonate to the Knudsen cell. Magnesium carbonate will probably be the first candidate. These experiments should answer any questions about the existence of iridium carbonyl.

The Pt 3008 alloy will also be used, as a Knudsen cell, to determine the interactions that occur when it is present in the system. Previous experiments indicate that pure platinum does not form complex gaseous molecules like the iridium. The experiment will be conducted like the previous experiment using the iridium cell with a sample from the same mixture of the oxide powders that were used in the iridium cell and the platinum cell.

Since sodium tungstate was observed coming from the iridium cell where the tungsten was present in quantities less than 1 percent, one would also expect to find sodium tungstate from Pt 3008 as the sample is 8 percent tungsten.

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

Progress this period included the continued development of selective vents as well as fabrication of nonselective vents for incorporation into platinum capsule lid assemblies. Tests were conducted with 5- and 10-mil platinum metal to determine an optimum bonding procedure, hot pressing parameters were established for preparing selective platinum vents, and a fabrication procedure for adding a uniform amount of filter material was developed.

Work This Period

Tests were conducted to determine the optimum temperature and pressure required to obtain a platinum-platinum bond during hot pressing. A number of 5- and 10-mil platinum coupons were hot pressed at temperatures from 1000-1400 C for 15 minutes under a nitrogen atmosphere. Inspection of samples that were mounted, polished, and etched indicated that bonding could be obtained in 15 minutes by hot pressing at a temperature of 1200 C and a pressure of 2000 psi. However, at lower temperatures the time required to obtain an equivalent bond increased logarithmically as a function of $1/T$. Figure 29 shows the metal-to-metal bond achieved by hot pressing at 1200 C and 2000 psi for 15 minutes. An equivalent bond was obtained at 1100 C and 2000 psi for 2 hours.

FIGURE 29. PT BOND FROM HOT PRESSING AT 1200°C AND 2000 PSI FOR 15 MINS

Development of a selective platinum vent continued and two spherically curved platinum vents were prepared, each with an anorthite particulate filter. Each vent was filled twice by slurry impregnation and fired at 1300 C. The first vent was hot pressed at 1400 C and the second at 1200 C. After pressing, cracks were observed on both sides of the platinum metal at the filter area. Metallographic examination of the vents indicated that cracks originated at the central area of each platinum-anorthite face. High compressive strength of anorthite at elevated temperatures was considered a possible cause for the metal failure during hot pressing. Therefore, any excess anorthite, above the surface of the labyrinth vent, might contribute to excess loads on the platinum metal in areas of the filter during fabrication.

A procedure was developed for insuring a more uniform filling of the anorthite. The slurry was applied and tamped down to the appropriate height with a small piece of platinum of spherical radius 2.08 cm equal to that of the backing plate (which would be bonded in the hot pressing stage). A third platinum test vent was prepared in this manner. After two fillings and firings at 1300C for 15 minutes in vacuum, the anorthite appeared to be uniform in height and the backing plate was secured for hot pressing. The vent was pressed at 1100C for 2 hours at 2000 psi under a nitrogen atmosphere. No cracking of the platinum was observed under 400X microscopic evaluation. The vent is currently under test and has been shown to be leak-free at room temperature.

Material to prepare capsule lid assembly vents was received from LASL June 20, 1977. A spark discharge fabrication technique was developed to machine vents directly into the capsule lid. The labyrinth pattern was recessed 2 mils into the capsule lid to allow for easy placement of the 5 mil backing plate. After machining, the vent pieces were washed in hot and cold Alconox, rinsed in alcohol, soaked in a 7.7N nitric acid solution, and rinsed in deionized water. Each vent has been twice filled by slurry impregnation with a 1:1 ratio by weight of ThO₂ powder and platinum black. They have been twice fired in vacuum for 15 minutes at 1225C and the 8-hole backing plates have been secured.

Vents designated BCL P-P-N1 (material lot E1-8A LASL) and BCL P-P-N2 (material lot 34 LASL) have been hot pressed at 1200C for 1 hour under a N₂ atmosphere. The helium throughput was measured for each

vent at ambient temperature and is 2.0×10^{-3} and 2.3×10^{-3} torr-liter/sec for vents BCL P-P-N1 and BCL P-P-N2, respectively. Fabrication of the remaining vents is underway. Completed vents will be shipped to Los Alamos prior to July 30, 1977.

Future work includes the continued development and testing of platinum selective vents and the fabrication of five nonselective spherical vents of MHW configuration from Pt 3008 for subsequent shipment to MRC.