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Heat Source Component Development Program

QUARTERLY REPORT FOR JULY-SEPTEMBER, 1976

Report Date: October, 1976

Battelle

Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

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HEAT SOURCE COMPONENT DEVELOPMENT PROGRAM

Quarterly Report for July-September, 1976

Compiled by
William M. Pardue

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: October, 1976

Battelle
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

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FOREWORD

This is the first 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. 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.

The activities covered in this new series of reports are all conducted under Contract No. W-7405-eng-92, Task 94. The specific components development efforts which are described herein are: a selective vent for helium release from the fuel containment, an improved reentry member and an improved impact member; the latter two items will hopefully be combined into a single "bifunctional" member material concept which is designated "RIM" (an acronym for Reentry Impact Member). Finally, supportive studies of a thermochemical nature are reported herein.

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HEAT SOURCE COMPONENT DEVELOPMENT PROGRAM

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 constraining particulate release and egress or ingress of other gases. 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

Finalization of fabrication parameters was accomplished for preparing spherically shaped nonselective vents.

Work This Period

BCL labyrinth nonselective vents have been on accelerated test with fuel simulants and have passed helium at rates many times greater than the generation rate in all test cases as reported in previous monthly reports. The simulant fuel was prepared from equal weights of MgO and molybdenum powders. With this simulant, the gaseous species of MoO_3 and MgO gives a combined vapor pressure of about 2×10^{-6} atm at 1800 K and 1×10^{-4} atm at 1900 K. During the past period considerable attention has been directed toward processing of vents and to nondestructive evaluation of vent performance.

One major change has occurred in processing; that is, the 1900 C sintering of the cermet particulate filter has been removed from the processing schedule and has been replaced with two 1400 C pre-sinter firings. Final densification of the filter then occurs during the hot-pressing stage. It has been established that hot pressing could be carried out as low as 1600 C in a matter of a fraction of an hour. This relatively low-temperature processing has eliminated the problems of iridium grain growth which had been encountered in the original processing schedule.

A test facility has been devised so that throughput in torr liters per second can be determined as a function of vent back pressure, and with different gases if desired. This is a nondestructive test and is incorporated in the vent processing just prior to the final clean-up in hydrogen. The device consists of a chamber which can be filled with a gas to any desired pressure. The vent is located in the end of the chamber and immediately on the side of the vent away from the chamber is a vacuum gauge. There is a length of tubing leading from the vent, forming a known conductance and then a mating vacuum gauge. From the pressure drop in the test chamber and the readings across the known conductance, it is possible to compute the conductance of the vent itself.

During this period, primary emphasis was placed on the development of spherically curved nonselective iridium vents, such as are required for incorporation into the MHW-PICS. Although the basic design of the BCL labyrinth vent remained unchanged, new fabrication techniques were required to produce a vent compatible with current welding techniques used at Mound Laboratory. The chambered and 8-hole sides of the vents were cut from 10-mil and 5-mil iridium sheet, respectively. The labyrinth pattern was then electric discharge machined onto the 10-mil piece and the outer edge was ground to a uniform thickness of 5 mils. Eight 5-mil tapered holes were machined into the vent backing plate. All dimensions are shown in Figures 1 and 2.

After machining, the vent pieces were annealed for 3 hours at 1200 C in vacuum. Pieces were then cold formed on steel dies to the appropriate spherical curvature. Each vent was filled by slurry impregnation with a 1:1 ratio by weight of ThO_2 and Ir powders, fired in vacuum for 15 minutes at 1400 C, and then refilled and refired at the same temperature. The vent backing plate was then aligned on the vent and the assembly transferred to a graphite die for bonding.

A graphite die of appropriate curvature was prepared to accommodate the assembled vent and is shown in Figure 3. Vents were hot pressed in argon at 1600 C and 2000 psi for 30 minutes. To evaluate the Ir-Ir bond between vent pieces after pressing, a first vent was sectioned, mounted and polished for metallographic examination. A microstructure of this specimen (Figure 4) shows the quality of the metal bond. Results of the examination indicated that pressing conditions employed had been adequate. As a second test of bond

From 0.010 in. Stock

From 0.005 in. Stock

FIGURE 1. BCL VENT

FIGURE 2. BCL VENT

FIGURE 3. GRAPHITE DIE FOR HOT PRESSING SPHERICALLY CURVED VENTS

50X

8H937

FIGURE 4. IRIDIUM-IRIDIUM INTERFACE IN NONSELECTIVE VENT
AFTER HOT PRESSING AT 1600 C, 3000 PSI, FOR
30 MINUTES (ALSO SHOWN IS CROSS SECTION OF
PLENUM CHAMBER)

integrity, a vent was prepared with the 5-mil holes of the backing plate aligned over the solid surface of the chambered piece, that is, shifted so as not to allow gas passage through the hole and into the chamber area. This vent was then hot pressed and tested for He throughput at room temperature. The result was a flow of less than 10^{-9} std cc/sec. Eight new holes were then machined over the labyrinth chambers; the vent was again tested and found to pass helium.

After hot pressing the vents were fired in 0.5 atm of hydrogen at 1200 C for 3 hours to remove carbon residue from the graphite dies.

To assure that heat treating and firing the vents in a tungsten element furnace was not depositing impurities on the vent, especially in the areas to be welded, a small section of a vent edge from a finished vent was analyzed by optical emission spectroscopy and compared with a piece of as-received iridium sheet. The analysis showed the amount of tungsten to be unchanged and thorium to be less than 300 ppm in both samples.

By mid-August, three vents were completed and ready for shipment to Mound Laboratory. Each vent was tested for helium flow at room temperature and the throughput was found to be $(5 \pm 1) \times 10^{-3}$ torr liters/sec. These vents were delivered to Mound Laboratory on September 15, 1976.

IMPROVED IMPACT MEMBER
(W. H. Duckworth - Task Leader)

Introduction

The objective of this program is to obtain a basic understanding of the mechanisms of high strain rate failure in materials that might be used for impact protection of RTG heat sources. From this information, a preferred materials system for significantly improved impact members will be selected, or developed and demonstrated. Not only is the behavior of the inert impact member itself of significance, but also the mechanical interactions between the fuel, containment shell, and impact member are complex and critical to overall performance.

Summary

During the period covered by this progress report, the experimental screening of carbon-base materials for impact-member use was essentially completed, and a technical report on this work was prepared. Also, the special experimental impact shells of the MHW type ("golf-ball" wound) were received from HITCO, and their testing was initiated as a next stage in understanding the impact behavior of carbon-carbon materials so that they can be tailored to provide improved materials. Work on a new phase was initiated, that of determining effects of temperature on energy-absorption characteristics of carbon-base impact materials. A test apparatus for obtaining load-deflection data to 2500 F was designed and is essentially constructed. Also, the design of an apparatus for determining effects of loading rates to ≥ 300 ft/sec on energy-absorption characteristics was finalized, and the apparatus is now under construction. Finally, continued progress was made in developing and refining the HONDO analyses so that it can be applied with assurance in predicting the dynamic mechanical response of carbon-carbon composite impact materials.

In the experimental screening of carbon-base materials for impact-member service, 14 carbon-carbon composites representing 6 generic types and three reentry-grade bulk graphites have been evaluated. Their failure modes and energy-absorption capacities were determined using the quasi-static bend test and the multiple-fracture test. The ranking of the materials on the basis

of energy-absorption capacity from the two tests was essentially the same, and none of these materials performed appreciably better than the MHW impact-shell carbon-carbon composite material.

The extent to which the fuel characteristics govern the overall impact response continues to be considered a very important factor in the screening and selection of preferred carbon-carbon materials*. Information relating to these characteristics must be available prior to final selection of the impact-member material.

Work This Period

Experimental Screening of Carbon-Base Impact Materials (G. K. Bansal and W. H. Duckworth)

The following additional carbon-base materials were evaluated during the past quarter for energy-absorption characteristics using both the quasi-static bend test and the multiple-fracture test:

- (1) Two carbon-carbon (C/C) composites obtained from General Electric Company, a 7D weave and a coarse polar weave (12, 4, 4)
- (2) A reentry-grade bulk graphite, Graphnol N-3, obtained from Oak Ridge National Laboratory.

This brings to 17 the total number of materials tested to date as shown in Table 1. The coarse polar-weave composite was not evaluated in the multiple-fracture test because it was not supplied in the flat-panel form required for the test.

Specimen cutting plans for the 7D weave and the coarse polar weave are shown in Figures 5 and 6, respectively. Specimens identified by numbers, 1 to 3, were for the multiple-fracture test and those identified by letters, A to F, were for the quasi-static bend test. Tests were conducted as described in the April, 1975, letter report**.

* Simonen, F. A., Bansal, G. H., and Duckworth, W. H., "RTG Impact Member Materials: A Preliminary Technical Review", BCL Report to ERDA, dated June 30, 1975.

** Informal Battelle letter report for April, 1975, on Tasks 67 and 68, Contract No. W-7405-eng-92, May 12, 1975.

TABLE 1. ENERGY-ABSORPTION TESTING RESULTS

Material	Fracture Mode	Work-of Fracture Test					Multiple-Fracture Test			Note
		Maximum Load, lb	Energy Absorbed, in.-lb		Work of Fracture, γ_f , in.-lb/in. ²	Fracture Mode	Maximum Load, lb	Total Energy Absorbed, in.-lb		
			Total, U_f	Initiation, U_i						
<u>Bulk Graphites</u>										
Union Carbide ATJ-S	Precipitant	7.5	0.031	0.015	2.07	0.50	Precipitant	2000	<100	
POCO AXF-5Q	Precipitant	12.0	0.033	0.027	1.22	0.53	Precipitant	3200	<100	
ORNL Graphnol N-3	Precipitant	9.0	0.045	0.040	1.13	0.72	Precipitant	5800	370	
<u>Cloth Lay-Ups</u>										
Super Temp Pyco Bond 418-80	Precipitant	18.5	0.91	0.22	4.24	14.60	--	--	--	2-D knit
HITCO Pyro Carb 508	Progressive	13.6	1.15	0.19	6.05	18.40	Progressive	4275	1150	
HITCO Pyro Carb 903	Progressive	14.0	0.80	0.13	6.15	12.80	Progressive	6250	1725	
Carborundum Carbitex 700	Progressive	25.0	2.06	0.35	5.89	33.00	Progressive	9500	2000	
<u>Felts</u>										
Super Temp Pyco Bond 407-80	Precipitant	13.1	0.13	0.05	2.37	2.05	Precipitant	5300	325	3-D
Super Temp Pyco Bond 807-82	Precipitant	11.5	0.08	0.03	2.56	2.31	Precipitant	5100	142	2-D
<u>Multidirectional Weaves</u>										
FMI 3-D Weave PN 491B	Precipitant	44.5	0.75	0.28	2.68	15.00	--	--	--	2-2-3 weave
FMI 3-D Weave PN 557B	Precipitant	27.5	0.53	0.26	2.04	10.50	--	--	--	2-2-4 weave
AVCO Pierced Fabric MOD 3A	Slightly progressive	23.0	0.63	0.23	2.74	10.08	Precipitant	6000	450	
AVCO Pierced Fabric MOD 3B	Progressive	9.0	1.23	0.26	4.73	19.68	Progressive	3200	1180	
GE 3-D Weave	Slightly progressive	62.5	0.98	0.52	1.88	15.68	Slightly progressive	6850	665	2-2-3 weave
GE Polar Weave	Precipitant	33.2	0.55	0.45	1.22	8.80	--	--	--	12-4-4 weave
GE 7-D Weave	Progressive	32.5	1.05	0.25	4.20	16.80	Precipitant	9500	1040	
<u>MHW Impact Member - Wound Sphere</u>										
GE/HITCO Pyro Carb 814	Progressive	11.0	~2.00	~0.05	~40.0	30.00	Progressive	3350	1520

FIGURE 5. SPECIMEN CUTTING PLAN FOR GE 7D WEAVE MATERIAL

FIGURE 6. SPECIMEN CUTTING PLAN FOR GE COARSE POLAR WEAVE MATERIAL

Load-deflection curves for the GE 7D weave material are shown in Figures 7 and 8, and those for the coarse-polar weave material in Figure 9. Load-deflection curves for the Graphnol were similar to those for ATJ-S or POCO-AXF-5Q graphites.

The critical stress-intensity factor, K_{IC} , required to initiate a crack and the work of fracture (γ_f) were determined from the quasi-static bend test. Calculated values of K_{IC} and γ_f are listed in Table 2. The total energy-absorption capacities of the GE 7D weave material and the Graphnol were determined using the multiple-fracture test from the areas under the load-deflection curves, and found to be 1040 and 370 in./lb, respectively.

To provide confidence in data from the quasi-static bend test, replicate specimens of a single material were tested under identical conditions. Load-deflection curves for seven specimens of Carbitex 700 material are shown in Figure 10. (Notches in the four specimens of Figure 10(a) were cut perpendicular to those in the three specimens of Figure 10(b).) Maximum loads for specimens in Figure 10(a) were 25 ± 2 pounds and areas under the curves were 2.06 ± 0.27 in./lb, with work of fracture (γ_f) values of $\sim 33 \pm 4$ in.-lb/in.². Similarly, for specimens in Figure 10(b), maximum loads, areas under the curves, and work-of-fracture values were 30.8 ± 0.8 pounds, 1.45 ± 0.05 in./lb, and 23 ± 0.8 in.-lb/in.², respectively. The above data reflect a high degree of reproducibility for the quasi-static bend test.

As stated in the March monthly report, comparable data for use in accounting for sphericity effects in the GIS plug specimen are needed on carbon-base materials other than the GIS. Three multiple-fracture tests, one each on three different specimen configurations, were performed on Carbitex 700 material. Load-deflection curves are shown in Figure 11, and the total energy absorbed along with the maximum load in each is given in Table 3.

Figure 11 and Table 3 show that the energy-absorption capacity of Carbitex 700 in panel form is ~ 60 percent higher than that in the form of a spherical plug with threads. If the GIS material is assumed to exhibit a fracture mode* similar to that of Carbitex 700, the GIS is also expected to absorb ~ 60 percent higher energy in the panel form than in the form of a

* Post-mortem examinations of specimens from Carbitex 700 and the GIS plug indicate that both materials exhibit crushing mode in multiple-fracture test.

FIGURE 7. LOAD-DEFLECTION CURVES IN QUASI-BEND TEST FOR GE 7D WEAVE MATERIAL

FIGURE 8. LOAD-DEFLECTION CURVES IN MULTIPLE-FRACTURE TEST OF GE 7D WEAVE MATERIAL

FIGURE 9. LOAD-DEFLECTION CURVES IN QUASI-STATIC BEND TEST OF GE COARSE POLAR WEAVE MATERIAL

TABLE 2. ENERGY ABSORPTION DATA FROM QUASI-STATIC BEND TEST

Material	Specimen Identification	Energy Absorbed, in.-lb		$\frac{V_p + V_i}{V_i}$	Fracture Energy, ² (γ_f), in.-lb/in.	Maximum Load, lb	K_{IC} , lb-in. ^{-3/2}
		$V_p + V_i$	V_i				
GE 7D weave	A & B	1.05	0.25	4.20	16.80	32.5	4150
GE polar weave (coarse)	A	0.53	0.50	1.06	8.48	43.5	5550
Ditto	B	0.55	0.45	1.22	8.80	33.2	4240
"	C	0.43	0.40	1.08	6.88	36.5	4660
"	D	0.50	0.25	2.00	8.00	25.6	3265
"	E	0.06	0.03	2.00	0.96	10.9	1350
"	F	0.05	0.03	1.67	0.80	5.5	700
Graphnol	--	0.045	0.04	1.13	0.72	9.0	1145

FIGURE 10. LOAD-DEFLECTION CURVES IN QUASI-STATIC BEND TEST OF CARBITEX 700 MATERIAL

FIGURE 11. LOAD-DEFLECTION CURVES IN MULTIPLE-FRACTURE TEST OF CARBITEX 700 MATERIAL

TABLE 3. ENERGY ABSORPTION DATA FROM MULTIPLE-FRACTURE TEST
(DEPENDENCE ON SPECIMEN CONFIGURATION)

Material	Specimen Dimensions, in.		Total Energy Absorbed, in.-lb	Maximum Load, lb
	Configuration	Thickness		
Carbitex 700	2 x 2-in. flat panel	0.50	2000	9,300
Ditto	1.72-in.-diam disk	0.50	1800	8,500
"	Spherical plug (1.72-in. diam)	0.50	1250	6,500
GIS	Spherical plug (1.72-in. diam)	0.50	950	3,350
Ditto	2 x 2-in. flat panel	0.50	1520*	--
"	1.72-in.-diam disk	0.50	1055*	--

* Estimates based on above data for Carbitex 700.

spherical plug. Energies for flat 2D C/C GIS materials such as panels and disks calculated from this assumption and the measured absorption capacity of a GIS plug are given in Table 3.

With this work, the experimental screening of various available carbon-base materials for impact-member service is considered to be essentially completed and, as a result, a draft technical report, "Experimental Screening Study of Carbon-Base Materials for Impact Members in Isotopic Heat Sources", was prepared during the quarter. Table 1 summarizes the results of the quasi-static bend test (work-of-fracture test) and multiple-fracture test on the 17 carbon-base materials studied. As shown in Figure 12, an approximate proportionality existed between energy-absorption values from the two tests; i.e., each test ranked the various materials in about the same order on the basis of energy-absorption capability.

None of the materials performed appreciably better than GE's MHW impact-member (GIS) material in the tests. Two cloth lay-up composites, HITCO's Pyro Carb 903 and Carborundum's Carbitex 700, were about equivalent in performance to the MHW material, while the bulk graphites and felt-base composites ranked least effective as energy absorbers according to the test results.

Of the materials evaluated, Pyro Carb 903 is currently considered the best prospect for a bifunctional heat shield and impact member. Its high density (1.80 g/cm^3) indicates potentially good ablation resistance to accompany its indicated good performance as an energy absorber.

Development of Preferred Impact-Member Materials (W. H. Duckworth and G. K. Bansal)

The mechanical response and energy absorption by the MHW impact shell (GIS) have been the subject of past experimental studies in the program. A technical report covering these studies is being prepared, and a draft copy will be submitted to NRA shortly. The studies have provided a basis for concepts of improved C/C composite impact materials of the GIS type (i.e., "golf-ball-wound" composites).

In the studies, failure modes and energy absorptions of GIS specimens were determined under stress-state conditions approximating those encountered in service and using either yielding or nonyielding fuel simulants. In the case

FIGURE 12. RELATION OF ENERGIES ABSORBED IN WORK-OF-FRACTURE AND MULTIPLE-FRACTURE TESTS

of a yielding fuel, the fuel exhibits significant plastic flow under radial compressive loads that are insufficient to initiate crushing in the GIS material.

A principal finding from the studies was that energy absorption by the GIS can be quite small with a yielding fuel. In this case, the GIS fractures from hoop tension with little or no crushing. With a nonyielding fuel, significant energy is consumed in crushing, and microscopic examination of the crushed material suggests that the formation of new surfaces by fracturing of fiber into very short lengths is the important energy-consuming process.

It becomes apparent, therefore, that the preferred C/C composite will be one that exhibits a crushing mode of failure, and whether crushing will occur in service depends on the resistance to deformation of the fuel as well as on strength properties of the composite.

The concepts that have been developed for preferred impact-member materials consider the cases of both yielding and nonyielding fuel, and address the question of what might be done in tailoring a composite that would increase energy absorption in each case or which would tend to assure the crushing mode of failure with a given fuel. The use of a different fiber than Thornel 50 offers a practical method for tailoring an improved composite, and the concepts focus on possible effects on failure mode and energy absorption from changes in fiber characteristics. Fiber strength is indicated to be the major factor controlling composite performance, with fiber size, fiber spacing, and fiber modulus being less important factors.

To determine whether the concepts are fundamentally sound, experimental GIS's featuring principally controlled variations in fiber strength were obtained from HITCO, the GIS manufacturer*. The special 2D fiber wound materials that were obtained are being designated RIM's** and have different properties. Three RIM's were ordered with fibers having different properties than the reference Thornel 50WYG130 fiber, as shown below (50WYG130 shown for comparison).

* Informal Battelle letter report for March, 1976, on Tasks 67 and 68, Contract No. W-7405-eng-92, April 29, 1976.

** RIM is the acronym for reentry impact member.

<u>Fiber</u>	<u>$10^6 E,$ psi</u>	<u>$10^3 \sigma_f,$ psi</u>	<u>Fiber Diam, μ</u>	<u>Fiber/ Tow</u>	<u>Tow/ Yarn</u>	<u>Diam, in.</u>	<u>Pre- cursor</u>	<u>Type</u>
50WYG130	57	315	5-9	720	2	0.015	Rayon	Graphite
75WYL160	78	380	6	720	2	0.015	Rayon	Graphite
WYB85	6	90	8.9	720	2	0.03	Rayon	Graphite
300WYB90	33	385	3.7	1000	1	--	PAN	Graphite

Two additional RIM's were obtained with standard T-50 fiber, but one was wound looser and one tighter than standard MHW-GIS. Another variable was to obtain a RIM that was of thicker wall than the reference GIS, and the final variation was to obtain a duplex RIM with inner wall of high strength 75WYL160 fiber and an outer wall of low strength WYB85 fiber.

The special materials were received from HITCO in mid-August and load-deformation tests of them, as outlined in our information progress report to NRA, dated April 25, 1976, are now underway. The data obtained will be reported in our next quarterly report.

Of interest to the problem of impact resistance is the information recently reported by LASL on the high strain rate testing of copper balls contained in various carbon materials, i.e., Carbocell, GIS, and bulk graphite. The results indicate that there is less deformation of a copper ball protected by bulk graphite than by C/C composites. Analyses of the results show that the mode of deformation of the copper is as would be expected from the theory of elasticity referred to as Hertzian contact stress*. Fractures resulting from impact of two hard, unyielding materials without the presence of a cushioning medium are found to have a characteristic conical fracture surface initiated at the contact point, in this case within the graphite. This conical surface propagates and effectively focuses the stress on a small area of the contained copper ball, resulting in the localized deformation configuration observed. Because the deformation is so localized, it would be expected that the PIC surrounding the fuel would be ruptured severely with such an impact member. In addition, work hardening of the copper in the area affected may well have prevented a total deformation representative of the situation encountered with the brittle PuO_2 material.

* Simonen, F. A., and Duckworth, W. H., "Analysis of Ceramic Materials for Impact Members in Isotopic Heat Sources", BMI-X-670, May 14, 1976.

Effect of Elevated Temperature on Energy Absorption
by Impact-Member Materials
(J. H. Peterson)

Energy absorption of impact-member materials has been measured only at room temperature in the testing done so far in the Battelle program. Although it is anticipated that elevated temperatures will not have an appreciable effect on energy absorption characteristics of these carbon-base materials, this must be verified. Accordingly, a test apparatus has been designed and is being constructed which will permit testing at temperatures up to 2500 F.

The basic test configuration will be the same as that used for the room-temperature tests of MHW impact shells (GIS's). The test utilizes an internal mandrel with a spherical radius matching that of the impact member. A flat platen applies the load to the external surface at a uniform cross-head movement of 0.05 in./min. In the case of the room-temperature tests, hardened steel tooling has been used. For the elevated-temperature tests, KT^(R)-grade silicon carbide tooling has been procured from the Carborundum Company.

Although most of the forces on the test set-up are axial, some lateral loads can exist owing to nonuniform failure of the impact member. At room temperature, the test arrangement is very short and compact with most of the fixturing being considerably larger in diameter than the impact member. This configuration remains quite stable should any lateral loading exist. However, in an elevated-temperature test, the relationship between diameter and length must be considered to keep the heater length to a minimum. The diameter should be as large as possible to minimize any lateral movement during the test, but must be limited because much of the compression load train is within the furnace. To obtain maximum heat input into a small volume, induction heating has been selected in preference to a resistance heated furnace. This will be accomplished by induction heating of a graphite susceptor surrounding the impact member and the associated tooling. An inert gas environment surrounds the test set-up. Also, the graphite susceptor, which will be at temperatures in excess of that of the test specimen, will minimize the possibility of any oxidation of the test specimen. A remote LVDT is being incorporated into the load train to measure the actual deformation of the impact member during test rather than relying on the cross-head movement. Initial tests will be at

0.05 in./min cross-head speed, but the apparatus and testing machine are capable of higher deformation rates. A schematic of the test apparatus is shown in Figure 13.

All components for the test apparatus are at hand, and assembly and check-out of the system is in progress. It is estimated that actual testing of impact-member materials will be initiated by mid-October, 1976.

Dynamic Analysis of Impact Materials
(L. E. Hulbert)

The work in this quarter has been directed toward the comparison of HONDO analyses of a copper ball impact with the results obtained at LASL using their modified version of NONSAP. Both codes are based on the finite-element method, but they differ in certain ways.

- (1) HONDO uses an explicit time integration approach, while NONSAP uses the implicit approach.
- (2) HONDO uses a constant strain quadrilateral element, while NONSAP uses a quadratic strain assumption with mid-side node elements.
- (3) Because of the differences in time integration, different approaches are used to enforce the boundary conditions on the impact face as the ball deforms.

Analyses have been performed using HONDO and NONSAP of a copper ball impacting a rigid steel plate at 94 meters/sec. Although the exterior shapes predicted by the two different programs were very close to one another, the predicted surface strains across the impact surface of the copper ball were considerably different. As described in previous progress reports, LASL predicted maximum strains of the order of 20 percent with NONSAP, while Battelle predicted strains of the order of 60 percent with HONDO.

It has been repeatedly shown in the past that there is little difference in the predictions of dynamic events between codes using implicit and explicit time integration procedures. The principal difference is in speed of the calculation and in the trade-off between being able to capture transient details and evaluating longer time events. Thus, we believe that the differences between the HONDO and NONSAP analyses can be attributed either to the boundary condition at the interface between the ball and the steel wall or to the order of the finite elements used.

FIGURE 13. ELEVATED-TEMPERATURE TEST APPARATUS FOR IMPACT-MEMBER MATERIALS

A study is being carried out to determine the effect of the impact plane boundary condition used in HONDO. Basically, this condition uses a very stiff spring to push back any node that "penetrates" the impact surface. Since the spring force is activated only when a node penetrates the wall, there is a tendency for the nodes to oscillate around the interface. It is possible that when the spring pushes the nodes back out of the interface it induces a higher plastic deformation in the element than is realistic. This plastic strain is not reversed when the node leaves the surface since the interface force is set to zero at that time. A check of the calculations performed to date shows that, in fact, there is oscillation in the nodes contacting the impact face. The oscillation period is about 15 to 30 time cycles in the calculation.

The determination of the effect of this boundary condition on the strains will be investigated by incorporating a more realistic boundary condition developed by John Hallquist at Lawrence Livermore Laboratory. Dr. Hallquist has indicated that he will send Battelle his developments as soon as he is permitted to do so.

The second difference noted above is the order of finite elements used in the programs. The effect of these different orders is that HONDO requires more elements to achieve the same order of accuracy as NONSAP. This does not mean that NONSAP requires fewer nodes than HONDO since the higher order element in NONSAP requires mid-side nodes, which doubles the number of nodes and the size of the stiffness matrix than when the constant strain elements are used. We will investigate the effect of element size by reducing the element size at the impact face in the HONDO model. A second test may be made with the ADINA code which was recently acquired by Battelle. This program is an improved version of the NONSAP code and provides dynamic analysis with 8-node quadrilateral elements, giving quadratic strains. Depending on the results of the variation in element size using HONDO, ADINA will be used to check the effects of using quadratic strain elements.

Once the uncertainties in the dynamic HONDO analyses have been resolved, HONDO will be used to perform the analyses of the spherical heat sources with carbon-carbon composite impact members. To permit these analyses, BCL engaged Dr. Fred Simonen, now at Battelle's Northwest Laboratories, to develop a deformation model that will permit representing the crushing effects in the carbon wraps. A further refinement of HONDO includes slide lines capability

that has been developed by Dr. Hallquist. This capability will be inserted in Battelle's HONDO code when we receive Dr. Hallquist's results. With these new refinements, HONDO should have the capability to model all of the needed effects in the carbon-wrapped fuel with the iridium shell.

It is planned to complete the verification of HONDO and the enhancements in the last quarter of 1976.

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 which were described previously in this report.

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.

Work on this new test was initiated during the period covered by this report. The test design was established and 95 percent of the parts were fabricated. During the next quarter, the equipment will be set up, calibrated, and tests conducted.

In the following sections, various aspects of this new test are described, including the selection of the impact geometry and the designs of the projectile accelerator and motion and force transducers to meet the exacting requirements of this test.

Impact Geometry. It was desired to simulate the impact geometry, forces, and kinetic energies encountered in impacting a flat, unyielding surface. Historically, the impact surface has been granite because this material was the hardest naturally occurring material which might be impacted by a reentering heat source. Many tests have shown that granite is generally not broken or plastically deformed by such an impact. Thus, the property of interest on initial impact in the granite is its elastic impedance, or the product ρC where ρ is its density and C is its sonic velocity. This property, together with the impedance of the impact member and the impact velocity, determines the initial impact pressure. However, before deformation of the impact member has proceeded very far (probably <0.04 in.), the initial impact pressure will have been modified by shock reflections to approach the pressure produced by the deceleration forces. Hence, the use of any undeforming material which has a high elastic impedance relative to the impact material will produce similar impact conditions.

During a real impact, the parameters which influence the rate processes and magnitudes of forces developed (aside from the impact-member properties) are the impact velocity, the initial kinetic energy, and the initial momentum of the impacting body.

Two principal approaches to the conduct of an instrumented impact test are possible which will meet the criteria of adequate simulation of the important impact parameters. These are

- (1) Impact of a complete reentering fuel assembly
 - (simulated), with impact protection, against a fast force transducer.
- (2) Impact of a hard-faced projectile against an impact material mounted on a high impedance force transducer.

The first approach has the advantage of use of a complete fuel assembly such as the FSA of the MHW under real impact conditions. However, two disadvantages of this approach appear. The principal one is that the collection of reliable deformation-time data is very difficult on a near-continuous time base. A secondary disadvantage is that launching the complete FSA requires a large carrier (sabot) and the attendant large launcher and sabot stripper-arrester.

The second approach can yield a good impact simulation by making the mass and velocity of the impacting projectile equal to that of the fuel assembly, thus duplicating the important impact parameters. Through use of a high-impedance, undeforming force transducer, and projectile impact with the member while the projectile is still guided by the launching barrel, several schemes become feasible for measurement of the deformation-time curve. Projectile launch is simplified in this approach. Thus, the second approach is chosen for implementation in this test.

Figure 14 shows a section view of the impact geometry being utilized in this new test, shown at the instant of projectile impact, as set up for testing in the MHW impact shell configuration. It should be noted that the tup in Figure 14 (Key No. 8) is replaceable to allow testing of other configurations. In the following sections, the approach to the design of the launcher, projectile, motion transducer, and force transducer will be described.

Launcher Design. The projectile launcher design was established to be a small compressed gas gun after evaluation of an available rotary arm machine. It was found that the compressed gas gun offered several advantages including cost, accuracy of impact control, and deformation measurement.

The required projectile weight was established to be 1.0 lb based on the weight of an MHW-FSA with a GIS in place. The required impact velocity in the vicinity of 300 ft/sec is quite low for a gun device. Some installations using a long-barreled gun capable of reaching much higher velocities have experienced considerable dispersion in muzzle velocity when low velocities were desired. This arises because the acceleration forces on the projectile become comparable in magnitude to the friction forces between the projectile and barrel. To minimize this potential difficulty in the present design, the barrel length was intentionally made short in order to keep the acceleration forces high. To keep friction low and for other reasons, the projectile material contacting the barrel was chosen to be aluminum, while the barrel was fabricated from stainless steel. This combination will allow running the barrel completely clean and dry which will contribute to reproducible friction effects.

For the desired impact geometry shown in Figure 14, it was desired to allow the projectile to come to constant velocity in the last few inches of its travel so that the velocity determined by the velocity pins in the

FIGURE 14. SECTION VIEW OF IMPACT GEOMETRY

KEY:

(1) Gun Barrel
 (2) Grounding connector
 (3) Velocity pins
 (4) Dielectric sleeve

(5) Projectile skirt (Al)
 (6) Projectile face (steel)
 (7) Test specimen
 (8) Replaceable tup

(9) Force transducer bar
 (10) Strain gage location
 (11) Epoxy bar support
 (12) Transducer support

barrel would be the same as the impact velocity. To achieve this, vents could be milled in the sides of the gun barrel, to allow the accelerating gases to escape prior to the last inches of projectile movement. However, during the travel of the projectile past the vented area, the projectile is still accelerating until venting is "complete", i.e., the pressure behind the projectile has dropped to a low enough value that the projectile velocity will be essentially constant. In order to determine the required vent size, it was found necessary to write and exercise a finite-time-step computer program because the nature of the governing equations for adiabatic expansion and gas venting precluded direct integrations. It is planned that this computer-based analysis will be published in detail in a later report.

The design approach was to choose the maximum feasible vent area in the sides of the barrel and still maintain integrity of the barrel, and then via parametric analysis, to determine the trade-offs between required barrel length, initial high pressure gas accumulator volume, initial accumulator pressure, and required venting length to reduce the projectile acceleration to the point that the projectile velocity would change less than 1 percent in the final inches of travel. It was also desired to construct a facility with as much flexibility and capability for impact testing as possible within the above guidelines. Based on the parametric analysis and the above design guidelines, a gun design was selected which appeared to be a reasonable trade-off of capabilities.

The parameters of the selected launcher design are as follows:

Accumulator volume	= 60 in. ³
Maximum accumulator pressure	= ≥1000 psi
Total barrel length	= 36.37 in.
Unvented acceleration length	= 24.00 in.
Total length of vented area	= 8.50 in.
Total vent area	= 44.88 in. ²

The predicted performance characteristics of the launcher design are shown in Figure 15. Figure 15(a) shows the projectile mass versus impact velocity capability of the launcher for an accumulator pressure near the maximum expected to be used. Figure 15(b) shows the predicted variations in projectile impact velocity with initial accumulator pressure. It will be noted that the predicted impact velocity changes about one ft/sec per 1.8 psi change in accumulator pressure.

(a)

(b)

FIGURE 15. PREDICTED LAUNCHER CHARACTERISTICS

Projectile Design. It was desired to have a projectile with a strong undeforming front impact surface with a total weight of 1.0 lb to match the weight of a MHW FSA for the initial impact experiments. To meet this objective, hardened steel was chosen for the front surface of the projectile. To allow this front surface to be the maximum thickness in keeping with practical limitations, a two-piece projectile design was selected as shown in Figure 14. The thickness of the aluminum projectile skirt was chosen to be the minimum which would keep the elastic expansion of the skirt to <0.001 in. in diameter when launched by 300-psi gas pressure. Allowance for this expansion was made in selection of the projectile diameter and tolerance with respect to the launch tube diameter.

Motion Transducer. With the choice of impact geometry as shown in Figure 14, several methods become available for measurement of the deformation, all based on measurement of the motion of the projectile within the barrel. From among the various photo-optical and electrical systems considered, a capacitance change technique was selected for this application.

The main features of this transducer in the launcher are shown in Figure 14. The launcher barrel (Key 1) is electrically isolated from ground and charged to a voltage E_0 by the transducer circuit. The projectile moves down the barrel into the dielectric sleeve (Key 4) before contacting the grounding connector (Key 2). As the projectile begins to exit the muzzle of the launcher, the capacitance between the barrel and projectile begins to change. This change in capacitance will be linear with the projectile motion after an initial transient associated with fringing effects. The transient will be ended before impact with the test specimen.

Briefly, the theory of operation of the transducer is as follows. From the basic expression for capacitance,

$$Q = CE \quad , \quad (1)$$

differentiation leads to

$$I = \frac{dQ}{dt} = E \frac{dC}{dt} + C \frac{dE}{dt} \quad , \quad (2)$$

where

I = current

Q = charge

C = capacitance

E = voltage.

In the transducer circuitry, the voltage, E , is maintained constant at E_0 , which suppresses the last term in Equation (2). The current, I , required to maintain E_0 during the event, is measured and converted to an output voltage, E_1 . Because the rate of capacitance change is linearly related to the velocity, the measured current is linearly related to the velocity, V , as

$$V = K_1 E_1 \quad , \quad (3)$$

where K_1 is the calibration constant of the apparatus which depends on both electrical and mechanical parameters. Thus, the first output from the transducer is directly proportional to the velocity. This output is then electronically integrated as a function of time to produce a second output voltage, E_2 , which is linearly related to the projectile position x :

$$x = K_2 E_2 \quad . \quad (4)$$

Calibration of the transducer will be conducted in two stages to determine the two constants K_1 and K_2 . Calibration of the velocity output will be performed with the aid of the velocity trigger pins (Key 3, Figure 14) which will make two independent measurements of the projectile velocity just before the projectile leaves the barrels during initial firings with no target in place. These firings will be part of making the initial calibration of the projectile velocity versus accumulator pressure which is necessary in any event as a part of qualifying the new apparatus.

The second part of the calibration is done using an auxiliary signal generator to obtain the integration constant of the electronic integrator.

This apparatus will be described in more detail in the next quarterly report when some operating experience will be available.

Force Transducer. The design study of the force transducer for this application resulted in a special purpose transducer designed to provide measurement of the fast-rising stress pulse without the complicating effects of reflected stress waves which plague many fast-force transducers.

The essentials of the design are shown in Figure 14. The test specimen (Key 7) is supported entirely by the force transducer. The force transducer is composed of several parts as follows. The replaceable tup (Key 8) is formed of hardened steel and machined to a good fit with the initial test specimens in the MHW configuration as shown. The tup is made replaceable in the event it becomes

damaged during impact testing. The tup is mated to the force transducer bar (Key 9) through a specially machined threaded joint designed to minimize stress wave reflections.

In operation, suddenly applied forces at the tup generate stress waves which propagate down the bar at sonic velocity. As the stress waves pass the strain gage station (Key 10), they are measured by a full strain gage bridge bonded to the transducer bar. This method of monitoring transient stress pulses has been frequently applied in the so-called split Hopkinson bar high strain rate test apparatus. Stress wave reflections are avoided during the impact time of interest by making the transducer bar long enough that reflections from its free end will not return to the gage station until after the impact event is complete. In addition, the bar is epoxy-potted (Key 11, Figure 14) into a long sleeve (Key 12) which should have the effect of gradually damping the stress waves as they pass along, although the transducer design does not depend on this damping quality.

The rise time of the force transducer will be limited by the time necessary to produce a so-called "bar wave" in which the lateral Poisson relaxation has taken place. This time will probably be in the order of $15 \mu\text{sec}$ (10^{-6} sec) for a smoothly rising input pulse. In this time period, the projectile at an impact velocity of 300 ft/sec will have crushed a maximum of only 0.054 in. of the RIM material during which the stress magnitude is expected to be smoothly rising. All other characteristic times associated with the measurement will be shorter, the longest being the time required for the stress wave to pass the strain gage length, about $1.25 \mu\text{sec}$.

The length of the transducer bar was selected at 60 in. With allowance for the position of the strain gage, this length will provide approximately $570 \mu\text{sec}$ of reflection-free recording time. Kinematic analysis of the time required to arrest a projectile traveling initially at a velocity, V_0 , in a distance, x_0 , shows, for the uniform deceleration case, that

$$t_k = 2 x_0 / V_0 \quad , \quad (5)$$

where t_k is the time to arrest motion with a constant deceleration force. For the present case with $x_0 = 0.5$ in. and $V_0 = 300$ ft/sec, $t_k = 277.8 \mu\text{sec}$, well inside the measuring time capability. In a separate analysis not reproduced here, it was found that the time t_0 to arrest the motion of projectile traveling initially at a velocity V_0 in distance x_0 for a decelerating force which varies linearly with travel by a factor, n , is given by

$$t_o = \frac{t_k}{2} \sqrt{\frac{n+1}{n-1}} \log (n + \sqrt{n^2 - 1}) , \quad (6)$$

where n is the ratio of the maximum to minimum decelerating force.

Equation (6) shows that the arrest time is a slowly varying function of variation in the deceleration force. Some representative values are given below:

<u>n</u>	<u>t_o, μsec</u>
1	277.8
2	316.8
4	370.0
7	414.8
20	538.5 .

The above values show that a large variation in the deceleration force with travel is unlikely to produce a measuring time requirement in excess of that available so long as t_k [Equation (5)] does not increase greatly, due to changes in material thickness or impact velocity.

Two other factors to consider are the maximum force which the transducer can measure and the resolution or minimum force. In the present case, the bar was made from AISI-4340 aircraft-quality bar heat treated to produce a yield strength in excess of 150,000 psi. If we choose 100,000 psi as the maximum allowable operating stress, the transducer can measure loads up to ~200,000 lb, well in excess of our requirements.

The gages selected and installed on the bar, together with the power supply and readout system, should provide measurements of a minimum force down to perhaps 100 to 200 psi with a similar precision as yet to be determined, depending on the reduction of the background noise that can be achieved.

During the next quarter, the assembly of the test apparatus will be completed, all systems checked out and calibrated, and tests conducted with materials now on hand at Battelle.

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 RTG applications 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.

Summary

The efforts in this task are directed toward several related studies. Analytical models have been developed to analyze the thermal stress response of C/C composites for RTG applications. Constitutive equations have been developed for C/C composites in a form suitable for finite-element stress calculations. Initial formulations were based on a knowledge of fiber and matrix configurations. Stress calculations have been performed on two-dimensional shapes and temperature profiles. An appropriate thermal and ablation response model was completed and used to analyze the effect of various shapes on required heat source weights and containment temperatures. A feasibility study was performed on an experimental method of determining the oxidation mechanism of carbon materials in the reentry transition regime between diffusion- and rate-controlled behavior. Detailed experiments of this type are being prepared.

Work This Period

Thermal and Ablation Response Models
for Shape Sensitivity Studies
(G. R. Whitacre)

During this quarter the thermal and ablation response model was used to analyze the effect of various shapes on required heat source weight and containment temperatures using the 150-watt single heat source model. The actual results of this study are reported in the section on "Results of Shape Sensitivity" of this report. In this study, the impact-member thickness was adjusted

to match the allowable impact velocity using simplified crushing models which will be described in this section. This additional variation and criterion had not been used in the previous screening and sensitivity studies which used a constant impact-member thickness of 0.46 inches. The arbitrary constraint that the ablator thickness is adjusted to be twice the maximum calculated recession has been used in all the analyses made to date. Various modes of heat source reentry were assumed and compared in the shape study and a number of two-dimensional cases were run.

In order to make valid comparisons for the different reentry modes and heat source configurations, the baseline spherical 150-watt configuration was rerun using two-dimensional heat-transfer fall-off from a stable stagnation point. The impact-member thickness was adjusted to match the allowable impact velocity for a sphere. The subsonic drag coefficient was also changed to the laminar value of 0.47 rather than the previously used turbulent value of 0.12. This was done because of the delay in boundary layer transition caused by a hot surface as is discussed in the section "Review of Subsonic Drag Data and Needs" of this report.

Figure 16 shows the allowable impact velocity as a function of impact-member thickness expressed nondimensionally as radius ratios. The curves are based on geometric crushing models with a measured energy absorption level for the impact carbon-carbon material of 9470 in.-lb/in.³ crushed. The models assume that the kinetic energy of the fuel plus its containment member is absorbed by the crushing of the appropriate volume of impact member. At small radius ratios, the energy is absorbed by the crushing of the impact member outside the plane tangent to the fuel containment member as shown in the sketch on the left of Figure 16. At large radius ratios, it is assumed that the energy is absorbed by the crushing of the impact material which would be swept out as the contained fuel moves through the impact member as shown on the right. At intermediate radius ratio values, the velocity from both models is calculated and the smaller impact velocity is used. The switching point between models occurs at a radius ratio of 2.0 for the cylindrical case and at 1.7 for the spherical case. In calculating the allowable impact velocity, the actual weight and size of the fuel plus containment members for the 150-watt spherical and cylindrical models were used. However, so long as the effective density of the fuel plus containment would remain at about 0.34 lb/in.³, these curves would apply to other sizes if the energy absorption level of the carbon-carbon material remained at 9470 in.-lb/in.³.

FIGURE 16. ALLOWABLE IMPACT VELOCITY AS A FUNCTION OF RADIUS RATIO

For the case of end-on impact of a cylinder or disk, i.e., crushing of a flat surface against another flat surface, the required thickness of an impact member for a specified impact velocity is given directly by the formula

$$t = \frac{W_F V^2}{2g \pi R_F^2 E_C} ,$$

where

- t = thickness of impact member
- W_F = weight of fuel plus containment member
- V = impact velocity
- g = acceleration of gravity
- R_F = radius of the contained fuel
- E_C = energy absorption level.

This equation or the curves in Figure 16 were used to adjust the impact-member thickness until the allowable impact velocity matched the calculated impact velocity for the computed trajectory.

Figure 17 shows the various geometric options available in the thermal and ablation response model. The two-dimensional spherical cases were analyzed using the spherical option with both r and ψ variation. (All previous analyses considered only the r variation.) Cylindrical heat source designs were analyzed by one of two options. The cylindrical option can be two-dimensional in r and θ . Some analyses were conducted assuming only r variation with the stagnation heat flux applied at the surface. However, additional analyses were performed which considered the θ variation with the two-dimensional heat flux fall-off from a stable stagnation point using Klett's^{*} report. (This same information was used for the fall-off in the two-dimensional spherical runs as suggested by Klett.) When end effects become significant, the axisymmetric model option must be used. Because variations with θ are no longer considered, the two-dimensional side-on stable reentry mode cannot be analyzed with this option. However, side-on spinning or stagnation line heating with end wall heating can be treated in addition to end-on reentry.

* Klett, R. D., "Drag Coefficients and Heating Ratios for Right Circular Cylinders in Free Molecular and Continuum Flow From Mach 10 to 30", Report No. SC-RR-64-2141, Sandia Corporation, December, 1964.

FIGURE 17. THERMAL AND ABLATION RESPONSE MODEL OPTIONS

Figure 18 shows the baseline 150-watt cylindrical design for a L/D ratio of 1.0. The fuel diameter and length are 1.493 in. The radiation gaps and iridium containment shell thickness of 0.020 in. are the same as was used for the 150-watt spherical baseline case. The thickness of the T-50 wound reentry impact member (RIM) for side-on reentry is adjusted so that the allowable impact velocity obtained from Figure 16 matched the calculated impact velocity for the computed trajectory. The thickness of the POCO AXF-5Q ablator was adjusted to be twice the calculated recession. Although the predicted recession on the flat face is much less for end-on than for side-on reentry, the thickness used for the cylindrical side was also used on the end to be conservative. The impact-member thickness is also taken to be the same on the end as that determined for the side-on impact.

The two-dimensional nodal network used is shown in the upper half of Figure 18. The iridium containment shell and the adjacent radiation gaps were combined into a single nodal layer with equivalent thermal properties to simplify the model. For side-on reentry only one-half the heat source is analyzed since the results are symmetrical about a plane through the center of the heat source. If the heat source is assumed to be spinning in side-on reentry, an integrated heat flux which is 0.269 times the stagnation line flux is applied to the cylindrical surface. The heat flux on the end face of the heat source is 0.147 times the stagnation line flux. These ratios were obtained from calculations by Klett. Analyses were also performed with the stagnation line heat flux applied over the entire cylindrical surface. This represents the more conservative assumption and is approximately equivalent to the approach used in the preliminary screening and sensitivity analyses where the stagnation point flux over the entire spherical heat source was used. These results also demonstrate corner effects near the stagnation line for the side-on stable reentry mode.

For end-on or disk reentry analysis with short L/D ratios, the axisymmetric (r,z) option was used. The disk can also be considered as a reasonable two-dimensional representation of a parallelepiped entering face on. The nodal network shown in Figure 18 was expanded to include both ends of the heat source since the center plane is no longer a plane of symmetry for end-on reentry. The heat flux and pressure distribution used in the disk analyses were taken from the curves for the SNAP 19/Pioneer heat source*. These curves are

* "SNAP 19/Pioneer F Safety Analysis Report--Volume II, Accident Model Document", Teledyne Isotopes Report No. INSD-2873-42-2, June, 1971.

FIGURE 18. BASELINE CYLINDRICAL HEAT SOURCE DESIGN FOR $L/D = 1$

nondimensionalized by dividing by stagnation values and are based on NASA-Langley test data. Although the curves are for a hexagonal shape entering end-on, the nondimensional distribution should be essentially the same for the disk in end-on reentry.

Drag coefficients must be supplied to the response model in order to determine the reentry trajectories. For all side-on cylinder analyses, the HSA drag coefficients reported by General Electric* were used. For end-on reentry of cylinders or disks with flat ends, the subsonic drag coefficients were obtained from Figure 21 of Hoerner** as a function of L/D. For supersonic and hypersonic drag, the following approximate formula from Klett*** was used:

$$C_D = 0.090 (2-K) ,$$

where

K = density ratio across the normal shock.

The various options of the thermal and ablation response model are capable of handling all the shapes and reentry modes studied in the shape sensitivity studies. It also appears that we will be able to analyze all preferred material systems as they are postulated. However, it is planned to incorporate some improvements such as a better model of the CO/CO₂ surface chemistry when it becomes available. In this way, confidence in the model will be improved and conservative safety factors such as allowing twice the recession thickness can be reduced.

Review of Subsonic Drag Data and Needs (G. R. Whitacre)

The impact velocity of an RTG heat source configuration is determined by the subsonic drag coefficient of the impacting body. In fact, since RTG heat source assemblies are essentially in free-fall at impact, the impact velocity can be expressed by

* "Multi-Hundred-Watt Radioisotope Thermoelectric Generator Program Annual Report, Part II", July 1, 1972-Dec. 31, 1972, Document No. GESP-7097, GEMS-415, Table 4, 10-4.

** Hoerner, Sigward F., Fluid-Dynamic Drag, published by the author, 1965.

*** Klett, R. D., *ibid.*

$$V_{\text{impact}} = \sqrt{\frac{2gW}{\rho C_D A}},$$

where

g = the acceleration of gravity

W = the weight of the body

ρ = the atmospheric density

C_D = the drag coefficient

A = the cross-sectional area on which the drag coefficient is based.

If the ballistic coefficient β is used, the expression becomes

$$V_{\text{impact}} = 29.0 \sqrt{\beta},$$

where

$$\beta = \frac{W}{C_D A} \text{ in lb/ft}^2 \text{ and } V_{\text{impact}} \text{ is in ft/sec.}$$

The drag coefficient is determined not only by the shape of the body but also by the type of flow around the body and the roughness of the body. In subsonic flow around most shapes, the turbulent subsonic drag coefficient is much less than laminar drag because the flow remains attached further around and the form or base drag is reduced. This lower turbulent drag occurs in spite of the fact that the friction drag at the surface is higher for turbulent flow than for laminar flow and increases with the roughness of the surface. For example, for smooth spheres, the accepted literature values* for the subsonic drag drop from 0.47 for laminar flow to 0.12 for turbulent flow. For W/A's characteristic of RTG heat source configurations as used in our screening and sensitivity studies, the impact velocity is thereby increased from 237 to 469 ft/sec.

Because of the importance of the subsonic drag coefficient in determining impact velocity and the possible variations in the value of the drag coefficient, a review was conducted of subsonic drag data and current additional needs in the area. This was especially timely since ERDA-sponsored work at

* Hoerner, Sigward F., "Fluid-Dynamic Drag", published by the author, 1965, pp 3-8 to 3-10.

Virginia Polytechnic Institute^{*} had just become available and a proposal for additional work in the area had been made by VPI^{**}. The results of this review are as follows:

- (1) The completed work on cylinders (GE drop tests and the VPI wind tunnel work) appears adequate for the determination of the MHW/HSA subsonic drag and resultant impact velocity. The effect of surface temperature is very important because the flow remains laminar for HSA surface temperatures as low as 400-600 F for a smooth surface and 1100 F for a rough surface.
- (2) The completed study on spheres at VPI appears adequate to determine the FSA impact velocity in the conceivable but unlikely case that it reenters alone. The results can also be used for other spherically shaped bodies. The turbulent drag coefficients found varied from 0.25 for smooth to 0.3 for rough surfaces and therefore are much higher than the literature value of 0.12. In addition, the flow remains laminar for spheres having surface temperatures as low as 200-400 F.
- (3) The proposed additional work on cylinders and spheres would improve the understanding of flow around these bodies but this alone would not justify the cost of the proposed program at this time.
- (4) The main benefit of the complete proposed program would be for theoretical prediction of the drag of heated bodies of other shapes. However, for generalized studies, the laminar and turbulent drag coefficients for other heat source shapes likely to be used can be obtained from the literature. The effect of elevated surface temperatures can be estimated as a delay in transition based on the previous studies.

* (a) Carr, S. R., and Marchman, J. F., III, "Drag of Heated Circular Cylinders", VPI-AERO-042.

(b) Marchman, J. F., III, and Sanford, D. C., "Drag of Heated Spheres", VPI-AERO-043, ERDA Grant No. E(49-15)-3070, March, 1976.

** Marchman, J. F., III, "An Investigation of Boundary Layer Transition and Separation on Heated Cylinders and Spheres", proposal to ERDA-NRA dated April 22, 1976.

- (5) For possible advanced shapes, the prediction of stability and mode of reentry (especially approaching impact) becomes very important and the proposed study does not appear to address this problem.
- (6) For a specific advanced design of unique shape, drag tests on a heated body should probably be conducted even if the proposed work had already been successfully completed. In addition, stability testing should also be done to determine the most probable mode of reentry and the stability characteristics of the reentry body.

Results of Shape Sensitivity Studies

(A. A. Boiarski)

A major goal of this design study was to identify preferred materials for RTG heat source reentry members. Spherical heat source models with various material combinations were examined and optimum weight material systems were identified. The thrust of the design study then shifted to shapes other than spheres. The main objective of these shape studies was to identify any overall system advantages and/or materials problems that could exist for nonspherical geometries.

In general, other shapes--such as disks and parallelepipeds--should have lower impact velocities since the sphere design provides maximum weight-per-unit cross-sectional area for a given thermal inventory. This expected lower impact velocity for nonspherical-shaped heat source configurations would be a system advantage and could result in a lower total weight for the heat source package.

Of course, there are obvious disadvantages to nonspherical geometries. Abnormal local heating and stress concentrations may become critical in the unsymmetric geometries and the impact analysis is complicated by the presence of end-on and corner-type collision scenarios.

To examine the thermal and ablation response of these nonspherical geometries, the one-dimensional RETAC computer code was altered to include two-dimensional configurations*. Both cylinder- and disk-shaped heat source

* See Figure 17 for response model options.

configurations were examined. However, only the baseline materials configuration* was examined in this study. It is expected that certain trends established in this baseline design analysis would be applicable for other material combinations as well.

A cylindrically shaped $150 W_{(t)}$ fuel form was used in the cylindrical heat source model (see Figure 19). An iridium containment member, T-50 wound RIM, and POCO graphite ablator thicknesses were initially selected by multiplying the spherical values by the ratio of cylinder-to-sphere fuel form diameter.

For the cylindrically shaped heat source configuration, the weight and ballistic coefficient (i.e., $W/C_D A$) is determined assuming no end caps on the cylinder. Hence, only large L/D (cylinder length/diameter) cases are expected to give meaningful results using this particular model, even though some runs with an L/D near unity were also made. In the final weight designation, an estimate of the end-cap weight and volume was included based upon ablator and impact thickness determined for the cylindrical portion of the heat source. Using the disk option of the thermal and ablation model, the "end-cap" weight is included directly in the analysis.

Various combinations of length to diameter (L/D) and thickness to diameter (t/D) were examined for the cylinder and disk geometries, respectively. Also, special $L/D = 1$ shapes were included. Several reentry mode and reentry heating scenarios were also examined. All the various cylindrical and disk sources were flown on a minimum γ (i.e., maximum recession) trajectory where

$$\gamma = 5.3^\circ$$

$$V_I = 36,000 \text{ ft/sec} .$$

Ablator and impact thicknesses were selected according to the iterative procedure discussed earlier in this report.

Results of the impact and ablation calculations for these various heat source geometries are summarized in Table 4.

The specific input shape, entry mode, and assumed heating mode are specified in the table along with the types of gaps (i.e., helium filled or vacuum) assumed to exist between the various material members. Several code outputs listed in Table 4 are the critical containment temperatures, heat shield

* See Figure 18 for baseline configuration.

FIGURE 19. BASELINE CYLINDRICAL HEAT SOURCE DESIGN

TABLE 4. SUMMARY OF CONFIGURATION ANALYSES FOR SPHERICAL, CYLINDRICAL AND DISK HEAT SOURCE SHAPES

Shape	Entry Mode	Heating	Gaps	Containment Member			V _{Impact} ft/sec	Heat Shield Recession, in.	Heat Source			View
				T _{Initial} C	T _{Max} C	T _{Impact} C			Wt., lb	Vol., in. ³	Density, lb/in. ³	
Sphere	Stable	Stagnation	Vacuum	1256	2320	1459	236	0.169	2.074	21.2	0.0978
Sphere	Stable	Stagnation	He filled	1060	2290	1082	236	0.169	2.074	21.2	0.0978	
Sphere	Stable	2-D	Vacuum	1252	1868	1336	220	0.151	2.113	22.31	0.0947	
Sphere	Stable	2-D	He filled	1053	1920	988	221	0.153	2.080	21.71	0.0958	
L/D = 1 cylinder	Spinning	Spinning + end wall	He filled	1025	1712	737	222	0.061	1.864	19.394	0.0961
L/D = 1 cylinder	Side-on- stable	Stagnation + end wall	He filled	1022	2423	704	223	0.138	2.290	26.028	0.0880	
L/D = 1 cylinder	"	Stagnation + end wall	Vacuum	1208	2378	1297	223	0.137	2.290	26.028	0.1034
L/D = 1.7 cylinder	"	Stagnation	Vacuum	1228	2328	1331	215	0.160	2.116	20.46	0.1034	
L/D = 3.2 cylinder	"	Stagnation	Vacuum	1175	2415	1212	194	0.156	2.099	19.05	0.1102	
L/D = 6.1 cylinder	"	Stagnation	Vacuum	1132	2496	1080	176	0.146	2.151	18.298	0.1176	
L/D = 6.1 cylinder	"	2-D	Vacuum	1132	1973	1058	176	0.136	2.000	16.65	0.1201	
L/D = 6.1 cylinder	"	2-D	He filled	983	2262	415	176	0.139	2.003	16.66	0.1202	
L/D = 3.2 cylinder	"	2-D	He filled	998	2200	589	194	0.147	2.100	18.47	0.1137	
L/D = 1 disk	End-on- stable	2-D	He filled	1012	2266	690	199	0.145	2.125	22.50	0.0944
L/D = 0.68 disk	"	2-D	He filled	1007	2338	522	166	0.125	1.644	13.17	0.1248	

T_{ALL} (upper) = 2255 C - (200 C below melting).T_{ALL} (lower) = 800-1175 C - (embrittlement).

recession values, and impact velocities. Further outputs include the overall heat source weight, volume, and corresponding effective density (i.e., total weight divided by total heat source volume).

A few terms given in Table 4 should be further clarified. A "stable" entry mode is one where the vehicle is not spinning such that it has a single constant-flow, stagnation point or line. "Side-on" as opposed to "end-on" stable implies that either the curved side of a cylinder (see Figure 19) or the flat end of a disk is aligned with the vehicle trajectory (i.e., incoming flow). "Stagnation heating" means that the high heating value characteristic of the stagnation region is assumed to exist around the whole surface of a sphere, or around the circumference of the cylinder. "2-D" heating implies that only the stagnation point encounters stagnation heating and the heat flux falls off with distance from the stagnation point. "End-wall" heating is the heating value assumed to apply to the configuration areas that are not lined up in the flow direction (i.e., the ends of a cylinder heat source entering "side on").

The most realistic, yet conservative, results of those listed in Table 4 are for the stable entry, 2-D heating conditions for the sphere (actually axisymmetric heating) and the disk heat sources. For the cylindrical design, side-on stable and 2-D heating for $L/D > 1$ and spinning + end wall for the $L/D = 1$ are most applicable.

As can be seen in Table 4, the impact velocity decreases for increasingly thin cylinders and flatter disks (i.e., $L/D > 1$). The lowest velocity calculated to date was that of a $L/D = 0.68$ disk. The impact velocity of 166 ft/sec is 25 percent less than the nominal 220 ft/sec value for the 2-D heated, stable sphere entry. The reduced impact velocity implies that less impact protection is required which results in a lower vehicle weight with increasing cylinder L/D and decreasing disk L/D ratios. Again, for the $L/D = 0.68$ disk, the weight was 22 percent less than for the baseline sphere configuration. These weight savings result in a more efficient heat source design.

However, these alternate shape RTG's are not without problems. Examination of the resulting iridium containment shell maximum and impact temperature values reveals that the containment material temperature limits are exceeded for some of these modified shapes. A clearer picture of the iridium temperature difficulty is shown in Figure 20, where the containment member's peak heating and impact temperature values are plotted for all the configurations listed in Table 4. The temperature limit shown in this figure is defined in a straight-

forward manner since it was selected to be 200 C below the melting point of iridium. However, the lower temperature limit is more difficult to define accurately. This low-temperature limit exists due to the fact that iridium will remain ductile above certain temperatures and will be brittle at lower temperatures. The specific temperature where the ductile-to-brittle transition takes place is a function of many variables and is a subject of study in itself*. Assuming that a value of impact elongation less than 10 percent will result in brittle failure, the data given by Schaffhauser for a 280 ft/sec impact were used to establish the embrittlement limits for various iridium-tungsten alloys listed in Figure 20.

Note that for more realistic entry modes (i.e., 2-D heating and helium-filled gaps) the cylinder and disk containment shell peak temperatures are at or near the limits of material integrity (i.e., softening near melting). However, spherical heat source designs are well below the high temperature limit. Note also that the minimum impact temperature for the realistic cylinder and disk entry modes is below the ductile-to-brittle transition temperature for even the most ductile material limit. On the other hand, the spherical containment shells are well above the limit for the DOP-14 and DOP-4 materials even though the temperatures are marginal for the undoped iridium containment shell.

In summary, the significant decrease in vehicle impact velocity for nonspherical shapes provides a weight savings but introduces an iridium embrittlement problem. The low impact velocity implies that the vehicle will take longer to impact after peak heating, hence, more time is provided to cool the containment member to below the embrittlement temperature. A possible solution to this problem would be to introduce further radiation gaps or insulation materials to prevent the containment shell from cooling excessively before impact. This would, of course, add weight to these designs which would negate some of the advantages of using nonspherical geometries in the first place.

Based upon these studies, it appears to be worthwhile to consider nonspherical shapes for advanced heat source systems configurations assuming fuel, metal containment, and RIM fabrication can be satisfactorily achieved.

* Schaffhauser, A. C., "Isotopic Power Materials Development Quarterly Progress Report for Period Ending March 31, 1976", ORNL-TM-5482, June, 1976.

Thermal Model Coupling to Thermal Stress Predictions
(G. R. Whitacre)

In the previous work on thermal stress predictions as part of the screening and sensitivity studies, only one-dimensional analyses were performed. For these cases, the temperature distributions in the ablator material predicted by the thermal and ablation response model were examined at various times in the steep-angle trajectory case. Plots were then made of selected critical temperature profiles and these temperature profiles became the input to the one-dimensional thermal stress code. Because the thermal model uses a finite difference network and the stress code uses a finite-element network, direct nodal temperature transfer was not practical.

As preferred material systems are being developed and studied, two-dimensional thermal analyses are being conducted with the thermal and ablation response model. In addition, in the study of alternate shapes, two-dimensional effects are significant and are also being considered. It, therefore, becomes imperative that the thermal stress code consider these two-dimensional effects also. The thermal stress code is capable of handling these 2-D cases although some additional input and running time are required. The missing link, thus, was a means to rapidly visualize and select the potential critical two-dimensional temperature distributions and then convert the thermal response model output to temperature input at the selected finite-element nodal locations. Thus, in this quarter work was begun on the automation of the coupling of the thermal model to the stress model.

The process is being done in three stages using existing subroutines and graphics where possible. First, the two-dimensional temperature profiles at various times in the trajectory are being transferred to a file at the time of the thermal and ablation response model run. Then, secondly, after searching the results for potentially critical temperature profiles at various times, selected profiles will be examined graphically. Two possibilities are being investigated for this graphical display--first, an isotherm plot in two dimensions, and, second, a temperature plot in which temperature becomes the third dimension on a three-dimensional graph plot. After a final selection of critical times is made, the third stage of the coupling involves the interpolation of the finite difference temperature data for the selected times into finite-element nodal temperatures. This will be automated using an existing bivariate linear

interpolation routine. Initially, these three stages will be done as batch computer operations, but later consideration will be given to using interactive graphics, particularly in selecting the critical temperature profiles.

The entire thermal model coupling to thermal stress prediction should be completed during the next quarter.

Thermal Stress Response of RTG Reentry
Thermal Protection Materials
(L. E. Hulbert)

Development of materials for RTG heat source reentry thermal protection has, until recently, been directed toward the bulk graphites. Although these have proven generally satisfactory, it is known that for some severe abort conditions these graphites offered very small margins of safety. Because of the apparently greater capabilities of carbon/carbon composites to resist thermal stress fracture, studies were begun to evaluate their use as reentry heat shields for RTG heat sources.

Because of the strict reliability requirements imposed on the materials used for the RTG heat source reentry thermal protection member, it is clearly necessary to be able to quantitatively characterize the thermal stress response of carbon/carbon composite heat shields to all possible reentry conditions. At the present time, it is not possible to predict the thermal stress failure response of carbon/carbon composites with sufficient confidence that carbon/carbon heat shields can be qualified for RTG heat source members. The principal reason for this is that the thermal stress fracture response of carbon/carbon composites is not well understood.

Because of the work that has been carried out on the use of carbon/carbon composites in severe reentry conditions by DoD, a request was sent from Mr. Glen Newby on June 21, 1976, to a number of DoD offices in the Air Force and Navy for information on their efforts to develop thermal stress fracture data of carbon/carbon composites. A number of responses were received that outlined the various programs that are being directed toward understanding the behavior of carbon/carbon composites.

These responses have been carefully reviewed at Battelle with the following conclusions:

- (1) Essentially all of the described programs were well-known to the technical community, including Battelle.
- (2) Most of these programs have been directed toward the gross response of the carbon/carbon composites in particular configurations and were not fundamental studies of the material behavior.
- (3) Only one DoD program was identified that is directed toward developing fracture criteria necessary to predict the safe thermal stress conditions for RTG heat source reentry protection designs.

In a further attempt to identify and evaluate the current research programs in carbon/carbon composite materials, L. E. Hulbert attended the "1976 Army Symposium on Composite Materials: Influence of the Mechanics of Failure on Design", held at Bass River, Massachusetts, September 14-16, 1976. This conference included both formal presentations and short presentations of "work in progress". The conference was devoted to the study of all types of fiber and matrix systems. However, a number of papers were devoted to behavior of carbon/carbon composites. Although a number of important microstructural behavior phenomena in carbon/carbon composites were reported, no general fracture prediction methodology for these materials was discussed.

After reviewing all of the various research programs outlined above, Battelle believes that the development of adequate fracture prediction methods for carbon/carbon composites may be possible based on the research being conducted in two programs. Both programs are based on a micromechanics modeling approach to the actual behavior of a crack propagating in a composite.

The first program is being conducted by Walt Rosen of Materials Sciences Corporation for the Naval Surface Weapons Center. The second program is being carried out by Battelle-Columbus^{*} for NASA-Ames Research Laboratory. Attempts have been made to acquire the latest reports of the research being conducted by Rosen for NSWC with no success to date. Thus, a detailed evaluation of this work cannot be made as yet. However, we have reviewed many of Rosen's earlier reports. From these reports, Rosen's approach does not seem to be quite as

* Kanninen, M. F., Rubicki, E. F., Griffith, W. I., and Broek, D., "Fundamental Analysis of the Failure of Polymer-Based Fiber-Reinforced Composites", NASA-CR-2689, April, 1976.

fundamentally oriented as the Battelle approach. The apparent result is that the Battelle approach may lead to an analysis method that is slower but more realistic than that being developed by MSC. Efforts will continue to obtain the reports of MSC's work so that a fair evaluation of their approach can be made.

At the request of ERDA, Battelle is formulating an outline for a research program directed toward the establishment of a thermal stress failure prediction methodology for carbon/carbon composites. Possibilities of joint funding of this research by DoD and ERDA are being explored, as it is outside the scope of the current contract, although considered to be an important aspect of an understanding of the problem.

Graphite Chemistry Study to Define Ablation Behavior
During the Low Velocity-Low Altitude Portion of
Reentry Trajectories
(A. A. Boiarski)

The behavior of graphite in reentry environments has been extensively studied, both theoretically and experimentally, for many years. Correlations have been obtained describing the mass loss rate and the heat-transfer rate including mass loss and combustion effects for a variety of trajectories. However, there are several regimes that are still not well understood. These questionable regions include the rate-controlled regime and the transition between the diffusion-controlled and rate-controlled regimes of mass loss as a function of wall temperature. The trajectory scenario where the RTG heat source encounters these regions is a combination of low velocity and low altitude conditions where the surface temperature is also low. In this case, the mass loss is high and is dependent upon the surface temperature which controls the reaction rates. Also, the exothermic combustion of carbon in the boundary layer increases heating to the surface above the usual aerodynamic heating or reduces the aerodynamic cooling effects. The amount of heating is not well known at this time because the amount of CO and CO₂ in the boundary layer is not well known and the amount of exothermic heat release is highly dependent upon knowing the surface layer chemistry.

The low velocity-low altitude portion of the trajectory is of special concern in the design of heat shields for RTG's since the heat source spends considerable time in the diffusion, transition, and rate-controlled mass loss

regimes associated with these trajectory conditions. Also, considerable attention must be directed to the heat source capsule impact condition and the ability to contain the enclosed fuel. The capsule material strength is highly dependent upon the impact temperature and also the amount of material that remains after ablation since some designs may utilize a portion of the heat shield for impact protection. Finally, the impact velocity, which governs the ultimate fate of the heat source upon impact, is dependent upon the final weight which is, in turn, influenced by the mass lost during reentry.

The uncertainty in surface temperature at which the transition regime occurs and the associated uncertainty in surface oxidation chemistry have led to relatively conservative and somewhat inconsistent assumptions in the design of RTG heat source heat shields. These conservative assumptions have led to obvious weight penalties. An attempt was made by Knight, et al.^{*}, to infer the CO to CO₂ mass fraction ratio, λ , at a graphite wall by measuring mass loss and wall temperature, then computing λ using various theoretical models. They were not successful because the data scatter was too large to infer any realistic value.

A new technique to directly determine the wall chemistry has been formulated which utilizes the Laser Raman Scattering (LRS) principle to identify chemical species and their concentrations.

Feasibility Experiments. Since the LRS technique has already been utilized to make measurements of the gases of interest in other nonrelated applications, a feasibility experiment was formulated to address two specific questions; namely, could the LRS method make composition profile measurements within the boundary layer (i.e., very close to the surface) of a hot carbon sample, and would the gray body radiation coming from the hot graphite interfere with these measurements?

Details of the LRS technique will not be presented here since they are available in many references^{** ***}. It suffices to say that the technique can

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

** Hertzberg, G., Spectra of Diatomic Molecules, D. VanNostrand Co., Inc., New York, 1950.

*** Boiarski, A. A., "Shock Tube Diagnostics Utilizing Laser Raman Spectroscopy", NSWC/WOL/TR 75-53, April, 1975.

be used to measure the amount of CO , CO_2 , N_2 and O_2 present in a gas, and, also the gas temperature. Briefly, the technique utilizes a focused laser beam to generate a small region of high photon flux within a gas. The gas molecules scatter this incident radiation with a resultant shift in the wavelength of the scattered light over the incident light as a result of the photon/molecule interaction. This wavelength shift is different for each kind of scattering molecule. Hence, the scattered radiation due to the presence of CO_2 in the laser focal region is a known color that is different from that scattered by N_2 or O_2 . Also, since the amount of scattered radiation at a particular wavelength (i.e., color) is directly proportional to the number of scatterers, the Raman scattering intensity is directly proportional to the number density of the various gases present within the small region of the laser focal point.

A portion of this scattered radiation is collected with appropriate optics and an image of a prescribed length of the focal region is itself focused onto the slit of a spectrometer for spectral analysis. In this manner, the intersection of the laser beam with the optical axis defines a small region or scattering volume from which the measurements are obtained. Hence, the density of various gas species can be measured at a point in an unknown gas sample. This point can be moved around within a given region by optical means to produce a profile of species density within the boundary layer or gas region. The explanation of temperature measurements is more involved but can be conceptually understood if each vibrational energy state of a particular molecule is treated as a separate species. Then it is clear that each of these species will itself have a characteristically different Raman frequency and will correspondingly scatter incident radiation depending upon the population of this species (i.e., energy state). Since the energy state population is directly related to temperature, the temperature can be obtained by measuring the details of the Raman spectrum of a particular molecule.

With this brief theoretical background in mind, a block diagram for the present feasibility experiment (shown in Figure 21) can be discussed. For reference, photographs of the overall experimental setup and details of the measurement region are also shown in Figures 22 and 23. A pulsed Nd:YAG laser was utilized in the present experiment. The primary laser wavelength at $1.06 \mu\text{m}$ was frequency doubled to produce an output wavelength of $5320 \text{ \AA}$. The pulse width was 100 nanoseconds and the repetition rate was 60 pulses per second,

FIGURE 21. BLOCK DIAGRAM OF FEASIBILITY EXPERIMENT SETUP TO DETERMINE GRAPHITE SURFACE CHEMISTRY

FIGURE 22. OVERALL VIEW OF GRAPHITE SURFACE CHEMISTRY EXPERIMENT

FIGURE 23. DETAILED VIEW OF MEASUREMENT REGION FOR GRAPHITE SURFACE CHEMISTRY EXPERIMENT

providing an average laser power of 20 milli-watts. This laser beam was focused into a spot size of approximately 80 μm diameter at a point just above a heated graphite rod. Raman scattered radiation was then collected at right angles to the incident beam and focused onto the slit of a SPEX 3/4 meter scanning spectrometer. The spectrally resolved radiation was converted to an electrical signal by an RCA C31034-A photomultiplier tube (PMT in Figures 21 and 22).

The PMT photoelectric pulses due to Raman scattering events are counted using the gated electronics and digital storage system. A gated signal from the laser power supply is suitably delayed in order to "turn on" the electronics at a time when the Raman photoelectric pulse just reaches the electronics input connector. Gate times of only 200 nanoseconds were employed in this experiment so any radiation occurring outside this narrow time region was automatically rejected (i.e., shorted to ground). Only background radiation from the graphite rod that happened to coincide in time with the laser pulses was recorded. Hence, a 1.67×10^5 reduction in measured background count rate was obtained with the gated electronics package.

The graphite sample used in this experiment was a parallelepiped with a square cross section machined from a block of POCO-AXF graphite. The ends of the sample were clamped to electrodes of a high current power supply. Controlled currents were passed through the sample to produce wall temperatures in the range of interest. A pyrometer was used to set and keep the surface temperature at a known prescribed value. The heated graphite rod was clamped with two pieces of asbestos and surrounded on the upper surface by an aluminum enclosure covered with a glass top. Holes were provided in the enclosure to allow the laser beam to enter and exit.

The view shown in Figure 23 is similar to that observed by the collection lens. Hence, the scattering volume is flanked by the hot carbon rod so this gray body radiation is directly observed by the optical system. As shown in Figures 22 and 23, a copper tube gas line is located over the graphite sample to provide for air or nitrogen blowing over the hot graphite heat shield simulant.

In the feasibility experiments, nitrogen was blown over the heated graphite sample to determine how close to the sample nitrogen concentration and temperature could be measured without encountering laser rejection problems. Also, the background radiation problem could be observed under more controlled conditions (i.e., no oxidation mass loss due to the presence of oxygen near the graphite surface) using a nitrogen environment. The results of these measurements

are shown in Figure 24 for room-temperature conditions (i.e., unheated sample) and various elevated sample wall temperatures. The measurements in Figure 24 were made at a point 0.006 in. above the graphite surface with a resolution of ± 0.0015 in. The results shown in Figure 24 indicate that no laser reflection problems are encountered in making measurements close to the graphite surface. However, as the wall temperature, T_w , increases, the background intensity, $I_{\text{background}}$, increases exponentially. Hence, the ratio of signal to background noise at 1020 C is 1:1. Above this temperature, the Raman signal will continue to shrink in proportion to the background intensity causing difficulties in data interpretation. It is obvious that for the range of temperatures of interest (i.e., $T_w = 1500$ to 2000 C) the background intensity would be too high such that the present system would not give useful results. Air was not introduced into the hot graphite boundary layer because, at the maximum temperatures attainable in the present experiment, negligible graphite oxidation would have occurred.

Two improvements that would significantly reduce this background emission problem can be realized by using a violet laser instead of the green laser which was used in the feasibility experiments. For example, the $3371 \text{ \AA}$ nitrogen pulsed laser or the doubled ruby laser available at Battelle would be excellent candidates. The Raman intensity would be an order of magnitude greater than with the green laser and the laser pulse width is only 10 nanoseconds wide (one order of magnitude less than for the green laser), so that a corresponding order-of-magnitude greater background reduction can be realized. Secondly, the gray body output from the graphite surface in the blue region of the spectrum will be considerably less than that for the green. According to Planck's Radiation Law, the radiant flux ratio at two different spectral regions at two different temperatures can be written as

$$\frac{W_1(\lambda_1 T_1)}{W_2(\lambda_2 T_2)} = \left(\frac{\lambda_2}{\lambda_1} \right)^5 \frac{\left(e^{c/\lambda_2 T_2} - 1 \right)}{\left(e^{c/\lambda_1 T_1} - 1 \right)}, \quad (1)$$

where

W = the radiation flux, watts/m² · mμ

λ = the wavelength, meters

T = the temperature, K

$c = 1.44 \times 10^{-2}$.

FIGURE 24. N_2 RAMAN DATA FROM A POINT 0.006 INCH ABOVE A GRAPHITE SURFACE FOR VARIOUS WALL TEMPERATURES COMPARED TO THEORY FOR INDICATED GAS TEMPERATURES AND NUMBER DENSITIES

Assuming that $\lambda_1 = 6070 \text{ \AA}$ and $\lambda_2 = 3650 \text{ \AA}$ (i.e., N_2 Raman wavelengths for Nd:YAG and N_2 -pulsed lasers, respectively), then for a graphite sample at $T_1 = 1293 \text{ K}$ (1020 C as in the feasibility experiment) and $T_2 = 1973 \text{ K}$ (1700 C), Equation (1) gives $W_1/W_2 = 0.24$. This implies that, for gray body radiation at realistic wall temperatures approaching the diffusion limit and utilizing the narrow pulsed violet laser, the measured background count rate will be approximately one-half of the value obtained during the feasibility experiments for a wall temperature of 1020 C. It would thus appear to be feasible to directly determine graphite surface chemistry under realistic conditions using this technique.

In summary, the experiment to determine the feasibility of measuring the surface chemistry on a hot graphite sample proved quite encouraging. Laser reflections were not a problem and measurements as close as 0.006 in. from the body surface were obtained with adequate resolution. This distance is within the thermal and species concentration boundary layers on hot graphite samples for the velocities of interest. This feasibility experiment also showed that a narrow pulse (i.e., 10 nanosecond half width) violet laser source was required in order to utilize Raman scattering in the presence of hot graphite gray body emission. Estimates obtained from experimental measurements at known conditions show that for a nitrogen-pulsed laser source the signal-to-noise ratio should be adequate to make measurements of N_2 , O_2 , CO_2 , and concentrations at the hot graphite wall for a known wall temperature. These measurements would prove to be invaluable in the assessment of the graphite surface chemistry which is of importance to the heat shield design for RTG applications and could be used in formulating realistic safety specifications for the required heat shield thickness.

Progress on Surface Chemistry Experiments With Revised Laser System.

Following analysis of the results obtained in the feasibility experiments, work was undertaken to set up the low velocity flow tests using the revised laser system. A high-volume flow blower was tested using a hot-wire anemometer to determine the blower exit flow velocity. Velocities in the range from 100-300 ft/sec were obtained. Further flow contraction could extend the velocity to values approaching 500 ft/sec if warranted.

A circular plug model like that shown in Figure 25 was designed and fabricated from ATJ-S graphite to investigate methods of uniformly heating the sample to elevated temperatures. A 5-kw induction heater was used and optical pyrometer measurements of surface temperature indicated that a temperature of 1400 C was easily achieved on the sample centerline by wrapping the induction coil on the large diameter of the plug. Some nonuniformity was evident in the temperature distribution, however, since the sample outside edges near the coil were about 100 C hotter than the center of the sample. Attempts will be made to eliminate this nonuniformity by wrapping another coil around the smaller diameter plug. By properly balancing the power input to the two coils, the nonuniform heating problem should be eliminated. An AGA Thermovision camera was used during these preliminary tests to make a thermal image photograph of the graphite surface in order to assess surface temperature uniformity. This instrument can be set to indicate small variations in temperature across the model surface.

Work in the near future will include the design of an aerodynamic model to incorporate inductive heating of the replaceable heat shield sample. This will facilitate the conduct of tests using alternate heat shield materials. The pulsed laser and required spectroscopic instrumentation will also be assembled.

FIGURE 25. HEAT SHIELD SPECIMEN GEOMETRY FOR SURFACE CHEMISTRY EXPERIMENTS

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 the actual or potential RTG heat source designs by basic studies of mechanistic nature.

Summary

An evaluation was conducted of the possible volatile species which could emanate from $^{238}\text{PuO}_2$ fuel and lead to interaction with other heat source components to plugging of vents, or other deleterious behavior.

Work This Period

As part of a continuing effort to establish a thermodynamic basis for prediction of reaction potentials involving plutonium fuel in contact with candidate cladding materials, some experiments have been performed on a matrix of impurity ingredients.

Although plutonium is a quite stable oxide and is available in a relatively pure form by ceramic standards, still consideration must be given to impurity migration and volatilization as possible contributors leading to vent closing or iridium degradation reactions. Because of the very large mass of plutonium in a heat source, even relatively small percentages of impurities can be significant. Table 5 lists the typical major impurities of the production $^{238}\text{PuO}_2$ fuel as compiled by NRA*.

An experiment using a physical mixture of CaO , Fe_2O_3 , NiO , Cr_2O_3 , in the approximate ratios of the above with NaAlSiO_4 , W , Ir and U_3O_8 was carried out in the Nuclide 12-90-HT mass spectrometer. The purpose of the U_3O_8 was to serve as an oxygen source for the experiment. The mixture was placed in a BeO Knudsen cell and heated in vacuum. At 1097°C , where the partial pressure of O_2 was 3×10^{-7} atm, sodium was observed at a pressure of 4×10^{-8} atm. At 1410°C , the O_2 pressure was 7×10^{-7} atm and the sodium pressure was 1×10^{-7} atm.

* Personal communication - Dr. Del Plymale.

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

A gaseous complex molecule was detected and by mass assignment a tentative identification as Na_2IrO_4 has been made. Using a conservative cross section, it is calculated that the partial pressure of Na_2IrO_4 at 1410 C is 5×10^{-9} atm when the oxygen pressure is 7×10^{-7} atm, and the sodium pressure is 1×10^{-7} atm. For this reaction then

$$\Delta G_{1680}^{\circ} = -139 \text{ K cal .}$$

One can estimate that the heat of formation may be as great as

$$\Delta H_{\text{f} 1680}^{\circ} = -310 \text{ K cal .}$$

This would indicate that this is a molecule of great stability. It is significant to note that it was identified and that under the same temperature-oxygen potential conditions there was no evidence of either IrO_2 or IrO_3 .

Although the oxygen pressures used in this investigation are higher than those likely present within a fuel sphere, oxygen is not the full story since the transport depends as much on the sodium vapor pressure as it does on oxygen. Although we did not detect it, possibly a CaIrO_4 molecule may also exist under somewhat less oxidizing conditions.

At 1490 C, the O_2 pressure was still 3×10^{-7} atm but the sodium had dropped to 8×10^{-8} , indicating that the sodium is being depleted from the sample. Ni was observed at 1×10^{-6} atm and Fe was observed at 1×10^{-7} atm. Table 6 summarizes the data obtained from these experiments.

Figure 26 shows the vent test assembly which is on test at the BCL Plutonium Laboratory. Two grams of $^{238}\text{PuO}_2$ was placed in the molybdenum body, then a vent fabricated at BCL by M. P. Rausch* was sealed into place with the molybdenum cap. The molybdenum support tube provides an evacuation channel for the test assembly. This tube will also act as a condensor in the event that any plutonium escapes through the vent. The support tube has a temperature gradient from the 1400 C test temperature to less than 100 C on the end which is connected to water-cooled copper. The test is running at 1400 C in vacuum, and is being used to obtain the thermodynamics of actual impurity migration as well as vent performance.

During the quarter, a report describing the Pu-Ir phase relationships as a function of temperature and composition was prepared and reviewed by Dr. Leo Brewer. His comments have been received and incorporated into a draft which will be submitted for NRA approval prior to publication. Finally, a new Knudsen cell was designed and constructed for the mass spectrometric study of plutonium materials.

* See section on "Helium Vent Development".

TABLE 6. OBSERVED SPECIES OVER MIXTURE
OF FUEL IMPURITY SIMULANTS

Temperature, C	Species	Pressure, atm
1097	O ₂	3 x 10 ⁻⁷
	Na	4 x 10 ⁻⁸
1410	O ₂	7 x 10 ⁻⁷
	Na	1 x 10 ⁻⁷
	UO ₂	3 x 10 ⁻⁷
	UO ₃	1 x 10 ⁻⁷
	Na ₂ IrO ₄	6 x 10 ⁻⁹
	O ₂	3 x 10 ⁻⁷
1490	Na	8 x 10 ⁻⁸
	UO ₂	1 x 10 ⁻⁶
	UO ₃	8 x 10 ⁻⁷
	Ni	1 x 10 ⁻⁶
	Fe	1 x 10 ⁻⁷
	O ₂	8 x 10 ⁻⁹
1572	Na	1 x 10 ⁻⁹
	UO ₂	6 x 10 ⁻⁷
	UO ₃	4 x 10 ⁻⁷
	Ni	5 x 10 ⁻⁶
	Fe	1 x 10 ⁻⁶

FIGURE 26. VENT TEST ASSEMBLY

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