GULF GENERAL ATOMIC

AEC RESEARCH AND
DEVELOPMENT REPORT

GA-8998, Suppl. B
C-92a, Systems for Nuclear
Auxiliary Power (SNAP) -
Isotopic SNAP Program
(M-3679, 65th Ed.)

LIGHTWEIGHT, LOW-BALLISTIC COEFFICIENT, RADIOISOTOPE FUELED

CAPSULE PROTOTYPE DEVELOPMENT PROGRAM

FINAL REPORT, SUPPLEMENT B

TEST DATA AND ANALYSIS

By

F. D. Postula
N. B. Elsner
M. R. Emken

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May 1971

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1.0 Introduction

This Supplement presents details of tests which were conducted to evaluate the capsule reentry coating performance. A series of simulated reentry heating (hyperthermal) tests were completed in the Sandia high enthalpy arc tunnel using 4-inch diameter tantalum alloy spherical models. Two types of coatings were investigated: pyrolytic carbon and hafnia. These were applied in various thicknesses using various application techniques.

After exposure to the simulated reentry environment, materials test specimens were cut from each model to evaluate the performance of the individual coating and application. These evaluations were done for two reentry conditions: launch abort and orbital reentry. The structural integrity of each model was maintained throughout each test.

Hyperthermal tests were conducted on three 8-inch diameter capsules. Two instrumented capsules were tested to obtain data on the distribution of temperatures on the fuel container. One capsule had fuel container supports with radiation interchange ports while the other had supports without the ports. These results were compared to those predicted with the aerothermal computer model described in the Final Report and substantiated the thermal modeling techniques. A third 8-inch capsule was a prototype capsule which had been vibration tested and was impacted after reentry heating.

Metallographic specimens were cut from the stagnation region of the 4-inch models. These were examined to evaluate the behavior of the W-CVD undercoat which protected the tantalum substrate.

Physical data was compiled from available data on the ultimate tensile strength, yield strength and elongation characteristics of tantalum, T-111 and T-222 alloys.

2.0 Four-Inch Hyperthermal Testing

A total of 15 four-inch diameter models were exposed to simulated reentry testing in the Sandia HEAT facility. Of these 12 were coated with either pyrocarbon or hafnia reentry coating.

Six of the models were instrumented at five locations with W-W26R_e thermocouples. This arrangement is shown in Figure 1. An approximately 7/8-inch hole was machined through the back of the model to allow pass through of the leads. A stem mount was designed as reusable and to provide positive mechanical attachment to the specimen while allowing for variation of the reentry coating thickness. The thermocouple leads terminated at the base of the mount in a hermetic connector. This feature allowed for shipping of the assembled specimen/mount and a simple plug-in at the test facility to the sting. An argon purge line was attached to the adapter near the hermetic connector. A small flow rate was introduced during the test in an attempt to avoid contamination of the specimen interior by residual air.

The other uninstrumented models were mounted on graphite cones which adapted them to the facility sting. A typical arrangement is shown in Figure 2 (the 8-inch capsule is shown). When this mount was used the models were cemented into a cup shaped cavity with C-9 carbon cement.

Tests on the 4-inch models were conducted in two phases. Phase I tests simulated the heating which occurs on the capsule during launch abort. Phase II tests were programmed to simulate the long heating profile of orbital reentry.

2.1 Phase I Tests

The objective of this phase was to expose the reentry coatings to the most severe thermal and mechanical reentry environment: that of the fourth stage abort reentry. To do this it was necessary to determine what conditions to operate the HEAT facility so that the transient temperatures and maximum temperatures would be matched. This was done by simulating the 4-inch model on the computer using the basic thermal model of the aeroshell described in Section 3.1 of the Final Report. It was found that both the transient

NOTES

2. REMOVE ALL BURRS AND SHARP EDGES
3. ITT CANNON ELECTRIC LOS ANGELES CALIF
4. GA THERMIONIC STOCK
5. LEE SPRING CO 30 MAIN ST BROOKLYN NEW YORK.
6. WALDES KOHINOOR INC LONG ISLAND CITY, N.Y.
7. LENGTH OF ITEMS 15 & 24 MUST BE HAND FITTED TO PRODUCE A PRE-LOAD OF APPROX. 10 LBS ON ITEM 17 SPRING. LENGTH WILL VARY WITH THICKNESS OF REENTRY COATING ON TEST SPHERES.
8. ITEM 17 MUST BE HAND SELECTED FOR A MIN. I.D. OF .743 (WITHIN MANUFACTURERS STD. TOLERANCE LIMITS)
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Fig. 2 - Capsule mounted on graphite core in
HEAT facility

and spatial temperatures could be well matched by using a constant heat flux of $228 \text{ B/ft}^2\text{sec}$. This match is shown in Figure 3.

Furthermore, the maximum predicted temperature of the structure at the stagnation point could be achieved at about 30 seconds test time. This allowed simulation of the most significant portion of the orbital reentry temperature profile during Phase I. Figure 4 compares these temperatures. It can be seen that the abort (Phase I) case introduces extreme temperature gradients.

Fifteen runs were made using 12 models as shown in Table I. All nine models which were reentry coated were successful; e.g. the tantalum alloy structure remained intact. Photographs of each model taken immediately after testing are shown in Table II. It was planned to find a location and tunnel setting which would allow pre-heating of the models to about 500°C prior to the reentry test. Calculations showed that the heating rate would have to be less than $5 \text{ B/ft}^2\text{sec}$. The lowest value obtained in the tunnel was $7.5 \text{ B/ft}^2\text{sec}$ and was at the plume edge. This rate caused a higher temperature at the stagnation point (noted during Run 1) so two pre-heat cycles were tried (Run 2).

Run 3 was made with a trial model having a 0.040 thick tantalum structure. This model was used to check-out the operating procedure of the facility. Run 4 was planned for the full 30 second duration, however, was aborted when the data collecting computer shut down. The same model was run a second time in Run 5 and survived the test. Some of the HfO_2 coating was lost from the stagnation region on both the trial and 4TO9 models. This was typical for the hafnia coated models. Review of the movie film taken during the testing revealed that most of the coating loss occurred at the end of the test. This may be caused by differential contraction and thermal stresses between the coating and the tantalum alloy substructure. In any event, Run 5 demonstrated that the sphere remains intact during abort reentry even with a loss of coating in the reentry region.

A different warm-up cycle was used for Runs 6, 7 and 8 in an attempt to make the initial temperatures more uniform. Both pyrocarbon coatings on models 4011 and 4018 cracked during the preheat cycle, however, the spheres survived the reentry test intact.

Fig. 3 - Matched transient thermal gradients - Phase I hyperthermal tests

Fig. 4--Stagnation point temperature during orbital reentry and Phase I hyperthermal tests

Table I - Phase I Hyperthermal Test Summary

Run	Model	Coating	Undercoat	Structure	Mount	Exposure		Reentry	Comments
						Ht.Flux	Pre-Heat		
1	4T09	.020 HfO ₂	W-spray	T-111	stem	7.5B/ft ² s	60 sec*	---	Pre-heat at plume edge.
2	4T09	" "	" "	" "	" "	7.5*	2(60s)*	---	" " " " " " " "
3	Trial	.015 HfO ₂	W-spray	Ta	stem	228*	2(60s)*	30 sec*	Sphere intact. Coating gone 30 deg. around stag. point.
4	4T09	.020 HfO ₂	W-spray	T-111	stem	228*	2(60s)*	15 s*	Run abort. Sphere intact. Coat spalled in 2 areas near stag. point.
5	4T09	" "	" "	" "	" "	228*	2(60s)*	30 s*	Sphere intact. Coating like run 3.

6	4T12	.020 HfO ₂	W-CVD	T-111	stem	218	4(30s)	31 s	Sphere intact. Coating raised free about 15 deg. around stag. point.
7	4011	.015 Pyro-C	W-CVD	Ta	stem	231	4(30s)	31 s	Sphere intact. Coating cracked on side during pre-heat. Cracked at weld during run. Small hole in coat near stag. pt.
8	4018	.014 Py-C	W-spray	Ta	stem	233	4(30s)	31 s	Sphere intact. Coating cracked during pre-heat; fourth of front gone.

9	4019	.010 HfO ₂	W-CVD	Ta	cone	223	none	31 s	Sphere intact. Coating intact with small blisters near stag. pt. Cone broke on removal; sphere dented in fall.
10	4010	none	none	Ta	cone	239	none	30 s	Top slumped-in and was removed. Sphere fell from mount.
11	4020	.033 Py-C	W-CVD	Ta	cone	255	none	30 s	Sphere and coating intact. Little erosion

12	4T11	.030 HfO ₂	W-CVD	T-111	stem	217	none	30 s	Sphere intact. Small coating spot gone near stag. point.
13	4T13	.045 Py-C	W-CVD	T-111	cone	228	none	30 s	Coating damaged prior to test. Had cracks from stag. pt. No coating removed during test. Cone broke on removal; sphere dented.

14	4021	none	.002 W-CVD	Ta	cone	226	none	15 s	2-inch hole in 10 sec. Rotated 45 deg.
15	4023	none	.003 W-CVD	Ta	cone	228	none	17 s*	Similar to 4021. Ablation obvious.

* Data not recorded.

TABLE IIa
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: Trial

Test: Phase I

Reentry coating: 0.015 HfO_2

Structure: 0.040 Ta

Undercoat: W-spray

Mount: stem

Exposure: Pre-heat: 2-60 sec cycles at $7.5 \text{ B/ft}^2 \text{ sec}$

Reentry: 30 sec run at $228 \text{ B/ft}^2 \text{ sec}$

Bend Tests: (Coupons were not cut from this specimen)

TABLE IIb
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4T09

Test: Phase I

Reentry coating: 0.019-0.017 HfO₂ Structure: 0.020 T-111

Undercoat: W-spray

Mount: stem

Exposure: Pre-heat: 2 trial runs at 7.5 B/ft² sec of 3-60 sec cycles
2 test runs of 4-60 sec cycles

Reentry: 2 runs, first 15 sec, second 30 sec at 228 B/ft²sec

Bend Tests

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.184 in.	0.616 in.	85.4 lb/in.
2	0.198	0.620	86.4
3	0.170	0.730	102.0 broke
4	0.182	0.681	77.5
5	0.179	0.708	86.5
6	0.166	0.705	92.8
7	0.142	0.695	83.0
8	0.177	0.730	45.8 broke
9	0.154	0.707	87.5
10	0.183	0.701	77.0

TABLE IIc
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4T12

Test: Phase I

Reentry coating: 0.020-0.018 HfO_2 Structure: 0.020 T-111
Undercoat: W-spray/W-CVD Mount: stem
Exposure: Pre-heat: 4-30 sec cycles at $7.5 \text{ B/ft}^2\text{sec}$
Reentry: 31 sec run at $218 \text{ B/ft}^2\text{sec}$

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.258 in.	0.653 in.	68.3 lb/in.
2	0.233	0.654	67.4
3	0.248	0.743	69.8
4	0.236	0.732	67.5
5	0.231	0.674	67.5
6	0.227	0.699	76.3 cracked
7	0.165	0.781	57.0
8	0.199	0.722	52.8
9	0.172	0.750	44.1
10	0.182	0.692	55.0

TABLE IIId
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4011

Test: Phase I

Reentry Coating: 0.020-0.018 Py-C Structure: 0.020 Ta

Undercoat: W-CVD

Mount: Stem

Exposure: Pre-heat: 4-30 sec cycles at $7.5 \text{ B/ft}^2 \text{ sec}$

Reentry: 31 sec run at $231 \text{ B/ft}^2 \text{ sec}$

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.184 in.	0.660 in.	68.0 lb/in
2	0.198	0.660	64.7
3	0.158	0.693	62.6
4	0.166	0.688	59.6
5	0.169	0.716	49.7
6	0.160	0.715	60.0
7	0.155	0.726	38.7
8	0.146	0.706	50.7
9	0.137	0.689	43.8
10	0.157	0.662	40.8

TABLE IIe

FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4018

Test: Phase I

Reentry coating: 0.023-0.018 Py-C Structure: 0.020 Ta

Undercoat: W-spray

Mount: stem

Exposure: Pre-heat: 4-30 sec cycles at $7.5 \text{ B/ft}^2\text{sec}$ Reentry: 31 sec run at $233 \text{ B/ft}^2\text{sec}$ Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>	
1	0.172 in.	0.648 in.	40.1 lb/in.	broke
2	0.171	0.670	38.0	broke
3	0.177	0.660	40.1	broke
4	0.158	0.705	53.2	broke
5	0.189	0.671	65.6	broke
6	0.173	0.699	46.8	broke
7	0.173	0.618	74.5	broke
8	0.195	0.681	76.5	
9	0.170	0.809	47.0	broke
10	0.199	0.727	54.8	broke

TABLE II f
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4019

Test: Phase I

Reentry coating: 0.014-0.013 HfO_2 Structure: 0.020 Ta

Undercoat: W-spray/W-CVD Mount: cone

Exposure: Pre-heat: none

Reentry: 31 sec run at $223 \text{ B/ft}^2 \text{ sec}$

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.216 in.	0.719 in.	35.2 lb/in.
2	0.228	0.736	35.5
3	0.222	0.760	39.6 with coating
4	0.235	0.790	32.8
5	0.199	0.682	44.7
6	0.240	0.722	37.9
7	0.204	0.721	47.5
8	0.236	0.713	44.0
9	0.189	0.727	48.1 with coating
10	0.223	0.752	40.8 with coating

TABLE IIg
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4010

Test: Phase I

Reentry coating: none

Structure: 0.020 Ta

Undercoat: none

Mount: cone

Exposure: Pre-heat: none

Reentry: 30 sec run at $239 \text{ B/ft}^2 \text{ sec}$

Bend Tests: (No coupons cut from this specimen.)

TABLE IIh
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4020

Test: Phase I

Reentry coating: 0.039-0.037 Py-C

Structure: 0.020 Ta

Undercoat: W-CVD

Mount: cone

Exposure: Pre-heat: None

Reentry: 30 sec run at $255 \text{ B/ft}^2 \text{ sec}$

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.160 in.	0.660 in.	39.4 lb/in.
2	0.170	0.661	38.2
3	0.153	0.670	41.1
4	0.164	0.690	37.2
5	0.158	0.694	38.6
6	0.146	0.673	44.5
7	0.143	0.704	41.3
8	0.144	0.690	38.2
9	0.142	0.659	38.0
10	0.139	0.661	41.0

TABLE IIi
FOUR-INCH HYPERThermal MODEL TEST RESULTS

Model: 4T11

Test: Phase I

Reentry coating: 0.031-0.029 HfO_2

Structure: 0.020 T-111

Undercoat: W-spray/W-CVD

Mount: stem

Exposure: Pre-heat: none

Reentry: 30 sec run at $217 \text{ B/ft}^2 \text{ sec}$

Bend Tests: (This specimen was kept for Phase II testing.)

TABLE IIj
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4T13

Test: Phase I

Reentry coating: 0.045 Py-C

Structure: 0.020 T-111

Undercoat: W-CVD

Mount: cone

Exposure: Pre-heat: none

Reentry: 30 sec run at $228 \text{ B/ft}^2 \text{ sec}$

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.189 in.	0.660 in.	49.7 lb/in.
2	0.222	0.654	46.0
3	0.176	0.725	48.9
4	0.173	0.771	46.9
5	0.183	0.705	54.6
6	0.167	0.642	48.5
7	0.162	0.709	52.5
8	0.185	0.720	46.5
9	0.174	0.703	54.0
10	0.184	0.705	53.9

TABLE IIk
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4021

Test: Phase I

Reentry coating: none Structure: 0.020 Ta

Undercoat: 0.002 W-CVD Mount: cone

Exposure: Pre-heat: none
 Reentry: 15 sec run at $226 \text{ B/ft}^2 \text{ sec}$

Bend Tests: (No coupons cut from this specimen.)

TABLE III
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4023

Test: Phase I

Reentry coating: none

Structure: 0.020 Ta

Undercoat: 0.003 W-CVD

Mount: cone

Exposure: Pre-heat: none

Reentry: 17 sec. run at $228 \text{ B/ft}^2 \text{ sec}$

Bend Tests: (No coupons were cut from this specimen.)

No pre-heat cycle was used on Runs 9 through 15 since it appeared that unrealistic thermal stresses were being introduced by the procedure. Of these tests the most promising in appearance were the HfO_2 coating on model 4019 and the pyrocarbon coating on model 4020. When the facility sting arm was being swung from the plasma stream the graphite cone stem of model 4019 broke allowing the model to drop to the test chamber floor. Although the sphere was dented the coating remained intact.

The pyrocarbon coating on model 4T13 had been inadvertently damaged prior to the test by a bump against the test chamber door frame. As a result the coating had cracks from the stagnation point but was still on the model. It remained on the model during the test, however, the cone mount broke as the model was being removed from the stream. The coating came off in the fall against the chamber floor. Local heating of the T-111 structure beneath the cracks can be seen in the photographs.

Models 4010, 4021, and 4023 were run to see whether an uncoated sphere or one having only the W-CVD undercoat would survive abort reentry. Although they did not survive it, they exhibited an interesting aerodynamic behavior. Once the stagnation point was breached, hot gasses could enter the sphere interior. The interior heating caused the bond between the sphere and graphite cone to give away. In each case the sphere rotated so that the hole turned away from the stagnation point. This aerodynamic effect would be accentuated during actual reentry due to the weight imbalance caused by the mass loss.

Of the six instrumented capsules temperatures were not recorded for either the trial model or model 4T09. Temperatures successfully recorded are shown in Figures 5, 6, 7 and 8. Figure 9 gives the predicted temperatures at the same locations as calculated for a 0.100 thick pyrocarbon coating. The temperatures measured at 30 seconds are compared to the predicted values in Table III. The 120° data for model 4018 appears erroneous as does that for model 4T11.

Stagnation point temperature difference between models 4018 and 4011 indicate that 4011 did not have an adherent coating. This was verified when the bend test coupons were cut from these models.

Fig. 5 - Measured temperatures on 4-inch hyperthermal model 4T12

Fig. 6 - Measured temperatures on 4-inch hyperthermal model 4011

Fig. 7 - Measured temperatures on 4-inch hyperthermal model 4018

Fig. 8 - Measured temperatures on 4-inch hyperthermal model 4T11

Fig. 9 - Predicted temperatures for 4-inch hyperthermal models

TABLE III COMPARISON OF 4-INCH HYPERTHERMAL MODEL TEMPERATURES

Model	Coating	Stag. Pt	60°	120°
Predicted values	0.100 Py-C	2080°C	1720°C	960°C
4018	0.020 Py-C	2060	--	350(?)
4011	0.020 Py-C	1600	1300/1310	770/860
4T12	0.019 HfO ₂	1850	1250/1310	860/1350
4T11	0.030 HfO ₂	1660	--	510(?)

The temperature difference measured at the stagnation point between models 4T12 and 4T11 suggested a variation of substrate temperature with the HfO_2 coating thickness. This is plotted in Figure 10. A linear extrapolation to zero thickness yields a temperature closely corresponding to the calculated surface temperature during abort (refer to Section 3.1 of the Final Report).

Fig.10- Temperature drop due to Hafnia coating

2.2 Phase II Tests

Phase II of the hyperthermal testing simulated temperatures resulting from orbital reentry using 4-inch models. In order to establish the test conditions the heating parameters of the capsule during orbital reentry were obtained from computer simulations. These are the stagnation cold wall heat flux, stagnation enthalpy and stagnation pressure shown in Figures 11, 12, and 13, respectively.

Once having these values, temperatures of the 4-inch model were calculated for the heating profile under tunnel conditions. It was found that a slightly greater heat flux was needed on the 4-inch model to ensure a 2100°C structure temperature than results on the capsule during reentry. This is attributed to the heat conduction away from the stagnation point on the smaller sphere with the same wall thickness. The matched heat flux conditions are shown in Figure 14.

Since the HEAT facility operating range is 5:1, the lowest heat flux setting was $40 \text{ B/ft}^2 \text{ sec}$ for a peak of about $200 \text{ B/ft}^2 \text{ sec}$. The resulting test heat flux profile is shown in Figure 15. The initial heating time at $40 \text{ B/ft}^2 \text{ sec}$ was adjusted to yield the equivalent integrated heating up to 600 seconds of reentry time. Enthalpy was selected as the second control parameter because the hot wall heat flux depends on a close simulation of the proper flow enthalpy.

Test conditions used for the Phase II reentry simulation are shown in Figure 16. A close simulation was achieved on the cold wall heat flux profile. The stagnation pressure rose somewhat too rapidly and was under the required value at peak heating. The enthalpy profile was a poor match initially yet was more than adequate to generate the required hot wall heat fluxes.

Models run in the Phase II hyperthermal tests are listed in Table IV. Test results are shown in Table V. None of the models were instrumented. Each was cemented to a graphite cone mount. These mounts were reinforced with a 3/8-inch molybdenum rod passing through the center. The rod was pin fastened to a clip welded to the aft hemisphere of the model. This arrangement insured against breakage of the mount during the test sequence.

Fig. 11 - Stagnation cold wall heat flux during orbital reentry -
8-inch sphere capsule

Fig. 12 - Stagnation enthalpy during orbital reentry

Fig. 13 - Stagnation pressure during orbital reentry

Fig. 14 - Cold wall heat flux to simulate orbital reentry on 4-inch hyperthermal model

Fig. 15 - Test enthalpy and heat flux to simulate orbital reentry on a 4-inch hyperthermal model

Fig. 16 - Phase II hyperthermal test conditions

TABLE IV

PHASE II HYPERTHERMAL TEST SUMMARY

Run	Model	Coating	Undercoat	Structure	Mount	Surface Temp.	Comments
1.	4008	0.010HfO ₂	0.005 W-spray over W-CVD	Ta	Cone	1980°C	Coating intact until heating stopped. Then stagnation area coating removed.
2.	4026	0.005HfO ₂	0.005 W-spray over W-CVD	Ta	Cone	1920°C	Coating behavior as model 4008. During run model vibrated 30 to 60 cps with 1/8-inch amplitude.
3.	4027	0.003HfO ₂ over 0.006 zone of 70% HfO ₂ 30%W	0.003 W-spray over W-CVD	Ta	Cone	1940°C	Sphere intact. Small blisters in coating during run, otherwise as above models.
4.	4T11	0.030HfO ₂	0.005 W-spray over W-CVD	T-111	Stem	2060°C	Model previously exposed and coating missing at stag. point. Burnout occurred after about 40Csec

TABLE Va
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4008

Test: Phase II

Reentry coating: 0.009-0.007 HfO_2 Structure: 0.020 Ta

Undercoat: W-spray/W-CVD Mount: cone

Exposure: Pre-heat: none
Reentry: Phase II heating profile

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.194 in.	0.691 in.	39.2 lb./in.
2	0.208	0.708	38.0
3	0.183	0.687	38.8
4	0.157	0.685	42.0
5	0.196	0.704	40.8
6	0.170	0.725	41.8
7	not cut		
8	not cut		
9	not cut		
10	not cut		

TABLE Vb
FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4026

Test: Phase II

Reentry coating: 0.011-0.009 HfO_2 Structure: 0.020 Ta
Undercoat: W-spray/W-CVD Mount: cone
Exposure: Pre-heat: none
Reentry: Phase II heating profile

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.208 in.	0.702 in.	52.9 lb/in.
2	0.219	0.685	45.2
3	0.173	0.709	42.1
4	0.179	0.725	59.1 cracked
5	0.181	0.678	89.0 broke
6	0.160	0.711	44.4
7	0.171	0.657	53.2
8	0.189	0.682	46.5
9	0.165	0.773	44.9
10	0.175	0.686	46.3

TABLE Vc

FOUR-INCH HYPERThermal MODEL TEST RESULTS

Model: 4027

Test: Phase II

Reentry coating: 0.017-0.015 total
HfO₂ over zone of
70% HfO₂ 50% W

Structure: 0.020 Ta

Undercoat: W-spray/W-CVD

Mount: cone

Exposure: Pre-heat: none

Reentry: Phase II heating profile

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1	0.186 in.	0.706 in.	68.8 lb/in. broke
2	0.208	0.721	63.0 broke
3	0.182	0.684	81.9
4	0.166	0.665	67.5 broke
5	0.173	0.760	65.4
6	0.167	0.708	74.3
7	0.141	0.649	67.4
8	0.183	0.680	62.8
9	not cut		
10	not cut		

TABLE Vd

FOUR-INCH HYPERTHERMAL MODEL TEST RESULTS

Model: 4T11

Test: Phase II

Reentry coating: 0.031-0.029 HfO_2 Structure: 0.020 T-111
Undercoat: W-spray/W-CVD Mount: stem

Exposure: Pre-heat: none

Reentry: (Previously exposed to Phase I test.)
Phase II heating profile

Bend Tests:

<u>Coupon</u>	<u>Width</u>	<u>Length</u>	<u>Test Results</u>
1			
2			
3	Contained in forward part that was ablated away		
4			
5			
6			
7	broke during cutting		
8	0.171	0.655	25.7 lb/in. broke
9	broke during cutting		
10	broke during cutting		

Little difference could be detected between the outward behavior of the coating on specimens 1, 2, and 3. All gave adequate protection of the underlying structure. It is noted, however, that several small blisters developed on model 4027. Subsequent testing of the bend coupons revealed that the substructure had been embrittled.

In each case the coating remained intact until the tunnel was shut down at which time the coating raised up in the stagnation region. This was attributed to differential expansion between the coating and the tantalum. Exposure of models 4008 and 4026 to the cooldown air stream caused some of the raised coating to be blown away. Model 4027 was removed from the stream and not exposed to cooldown flow. Surface temperatures were estimated by a pyrometer measurement at a location 40° away from the stagnation point.

The fourth model, model 4T11, had been previously tested in Phase I. This model had the coating cracked and partially removed in a 2-inch circle about the stagnation point as a result of the original exposure. It was included in Phase II to assess the extent of protection afforded by a damaged coating.

3.0 Eight-Inch Hyperthermal Testing

A total of three eight-inch prototype capsules were exposed to simulated orbital reentry heating in the Sandia HEAT facility. The characteristics of these capsules are given in Table VI. All three had the same reentry coating: 0.005 HfO_2 over a 0.005 blended zone of 50% HfO_2 and 50% W. (These test capsules did not have the W-CVD undercoat.) This coating was selected based on the apparently adequate performance of a similar coating run on 4-inch model 4027 in Phase II testing. Subsequent post-test evaluation of model 4027 revealed that the coating which had been applied was actually about 80% thicker than intended. Also subsequent bend tests of coupons cut from model 4027 showed that some embrittlement had occurred. These data were not available, however, at the time when the 8-inch capsule coating was selected.

Other common factors between the three 8-inch capsules are the aeroshell, Pt shell and the fuel simulant. For all three capsules the fuel container was seal welded in helium and the Pt shell girth welded in helium. The Hyper I and Hyper II capsules were instrumented while Comp I which was to be subsequently impacted was uninstrumented. Comp I was designed for impact, hence the buffer shells and the 0.070 thick fuel container. Hyper I and Hyper II differed only in the fuel container supports. Those of Hyper I were solid while those of Hyper II had nine radiation interchange ports machined in them.

Hyper I and Hyper II capsules were each instrumented with five thermocouples as shown in Figure 17 to record the transient temperature response of the capsule. Since it was most important to evaluate the heating of the platinum secondary shell, four thermocouples were attached to this shell with a redundant set immediately behind the stagnation point of the aeroshell. Mounting of these capsules was accomplished by using a cut-off stem mount previously used on the 4-inch models. The inner tantalum stem was EB welded to a tantalum thermocouple pass-through and the assembly EB welded to the aft aeroshell hemisphere. After final EB welding of the aeroshell and reentry coating, the capsule interior was pumped down and the thermocouple leads potted with epoxy.

Temperature response of these thermocouples was predicted with the computer thermal model described in Section 3.1 of the Final Report. These calculations used the planned test heating profile which was the same as

TABLE VI

EIGHT-INCH HYPERTHERMAL TEST CAPSULE CHARACTERISTICS

Capsule	Reentry Coating	Aeroshell	Support	Buffer	Pt Shell	Fuel Container	Simulant
Hyper I	0.005 HfO ₂ over 0.005 50% HfO ₂ 50%W	0.020 T-111	0.010 Ta solid	None	0.010	0.020 T-111	ZrO ₂
Hyper II	↓	↓	0.010 Ta ports	None	↓	0.020 T-111	↓
Comp I	↓	↓	0.010 Ta solid	2-0.020 Ta	↓	0.070 T-111	↓

Fig.17 -Hyperthermal wind tunnel eight-inch capsule

that used for the Phase II 4-inch models, Figure 15, except that the peak cold wall heat flux was reduced to $190 \text{ B/ft}^2\text{sec}$ which corresponds to actual reentry conditions. The predicted values are given in Figure 18.

A summary of the eight-inch hyperthermal tests is given in Table VII. All capsules were preheated in a furnace installed within the HEAT facility test section prior to reentry simulation. The heating period was limited to 30 minutes with the tunnel at vacuum based on the capability of the steam-ejection pumping system. After the preheat time the plasma jet was started and the capsule lowered out of the furnace and swung into the stream. The resulting temperatures were from 300°C to 400°C at insertion in the plasma jet flow.

Detailed test results are presented in the following sections.

3.1 Hyper I Test

A heavy walled 8-inch calorimeter model was designed to measure the stagnation heat flux in the hyperthermal wind tunnel, Figure 19. An asymptotic calorimeter having a heat flux range of $0\text{-}300 \text{ B/ft}^2\text{sec}$ was mounted at the stagnation point. Water cooling lines and the electrical lead passed through the standard hyperthermal model mount. Splash water cooling was provided to prevent overheating of the uncoated tantalum sphere.

Heating conditions for the Hyper I capsule were established by using the 1-inch diameter facility calorimeter to obtain the transient profile which was adjusted for the measurements made by the 8-inch diameter calorimeter model supplied by GGA. For example, at the same tunnel setting the facility calorimeter yielded equivalent heating at the stagnation point of an 8-inch sphere of $40 \text{ B/ft}^2\text{sec}$ while the 8-inch calorimeter gave $33 \text{ B/ft}^2\text{sec}$. Therefore, the tunnel flow conditions as set by the facility calorimeter were programmed for a heating rate about 21% higher than that recorded on the 8-inch calorimeter. This profile (shown in Figure 20) was used for the Hyper I capsule since it was believed that the difference in heating may be a consequence of geometric scaling between the 1-inch and 8-inch calorimeters.

Excellent temperature data was measured by all five thermocouples

Fig. 18 - Predicted 8-inch hyperthermal test temperatures

TABLE VII

EIGHT-INCH CAPSULE HYPERTHERMAL TEST SUMMARY

Run	Capsule	Coating	Undercoat	Structure	Mount	Surface Temp.	Comments
1	Hyper I	0.005 HfO ₂ over 0.005 zone of 50% HfO ₂ 50% W	0.003 W-spray	0.020 T-111	Stem	2300°C	Heat flux 20% too high. Aeroshell burnout at 447 sec. Run continued to completion. Capsule interior gutted.
2	Comp I				Cone	~ 1700°C	Heat flux 20% too high. Run terminated at 368 sec. when coating loss noted.
3	Hyper II				Stem	above 2200°C	Coating blistered over forward hemisphere at 1400° to 1500°C. Run aborted at 432 sec. due to severe coating removal.

Fig. 19 Heat flux calibration sphere for hyperthermal wind tunnel

Fig. 20 -Hyper I and Comp I hyperthermal test conditions

located on the Hyper I capsule throughout the test. These are shown in Figure 21; a redundant measurement was made on the platinum shell top (located directly beneath the aeroshell stagnation point) and gave essentially duplicate results. An additional measurement was made with the facility pyrometer, Figure 22, and is in agreement with the thermocouple data.

During the test run the tunnel flow conditions are continuously changed to yield the required heat flux by changing both the plasma jet torch current and gas flow. Since the capsule aeroshell is thin its temperature responds rapidly to variations in heating rate. Therefore, the variations in the temperature measured on the aeroshell stagnation point reflect a variation in the tunnel heating conditions. Similar variations in the measured platinum top temperature obviate the possibility of anomalous thermocouple behavior. From Figure 21 it is seen that aeroshell temperature rose sharply to 2300°C at about 440 seconds and then dropped to about 2060°C .

Monitoring of the on-line temperature measurements indicated burn-out of the aeroshell at 447 seconds, however, the decision was made to continue the test through peak heating (from 450 to 490 sec) to the end of the heating profile at 560 seconds. During this time considerable burning of the capsule occurred as can be seen from the photographs of Figure 23. The forward hemisphere, fuel container, platinum shell and fuel container supports were consumed. The interior of the capsule was coated with a thick white glassy material, tantalum oxide.

3.2 Comp I Test

An ATJ graphite ice cream cone type mount was designed to support the composite test capsule during hyperthermal testing, Figure 24. The center of the graphite mount contained a 3/8-inch molybdenum shaft which was pin fastened to a clip welded to the aft aeroshell hemisphere of the capsule. Graphite cement was not used in this mount since the capsule had to be removed for subsequent impact testing.

The Comp I capsule was tested under the same heating profile conditions as Hyper I capsule, Figure 20. This was done so that it could be exposed

Fig. 21 - Measured Hyper I capsule hyperthermal test temperatures

Fig. 22 Pyrometer temperatures measured on Hyper I capsule

Side view

Front view

Capsule interior heated by plasma jet stagnation condition.
White glassy material is tantalum oxide.

Fig.23 - Post-test condition of Hyper I hyperthermal capsule

Fig. 24 Composite test capsule mounted for hyperthermal test

to reentry conditions prior to the rocket sled impact test which was scheduled for the same day. During the run the stagnation region was carefully observed on the TV monitor and the test was aborted at 368 sec when some coating loss was noted. This capsule was uninstrumented and, thus, the only measure of temperature was from the optical pyrometer aimed at a location 3 inches away from the stagnation point. Recorded temperatures of this location were about 1700°C at the time the test was terminated, Figure 25.

The capsule was then taken to the impact test site where it was photographed, Figure 26. These pictures show some blistering of the coating in the stagnation region and asymmetric removal which may indicate asymmetric heating.

3.3 Hyper II Test

Before testing the Hyper II capsule the 8-inch calorimeter model was carefully inspected. This revealed that the tantalum sphere had been damaged when it was inadvertently driven against the ceiling of the tunnel test section. Therefore, it was also possible that the asymptotic calorimeter at the stagnation point was also damaged. It was decided to reprogram the heating rate profile using the facility calorimeter as the standard; it had been used successfully for the 4-inch model tests. The new test conditions established for Hyper II are shown in Figure 27.

Again, excellent temperature data were measured during the test run as shown in Figure 28. The pyrometer temperatures, Figure 29, are in agreement, however, rise faster during the heat pulse. This indicates a much higher surface temperature compared to the underlying T-111 aeroshell. Such a high surface temperature could result if the hafnia coating were raised from the aeroshell as would occur by blistering.

Observation of the capsule during the test showed severe blistering of the hafnia coating over the top third when the stagnation temperature read between 1400° and 1500°C. As the high heat flux portion of the profile was reached with the corresponding increased stagnation pressure the blistered coating began to be blown away. The run was aborted at 432 seconds

Fig. 25 Pyrometer temperatures measured on Comp I capsule

Side view

Front view

Capsule at impact test site with exterior thermocouples
secured by stainless steel strap.

Fig. 26 Comp I capsule after hyperthermal testing

Fig. 27 Hyper II hyperthermal test conditions

Fig. 28 Measured Hyper II capsule hyperthermal test temperatures

Fig. 29 Pyrometer temperatures measured on Hyper II capsule

when burn-out occurred in the aeroshell. Figure 30 shows the Hyper II capsule after testing. It seems odd that the stagnation point was intact while adjacent regions were burned-out. The hot test gas flow entered the capsule interior and directly heated the fuel container. Removal of the fuel container revealed that it was still intact, Figure 31, however, a portion of the Pt shell was vaporized by the direct heating.

3.4 Eight-Inch Test Evaluation

The eight-inch capsule tests were successful in establishing the internal heat transfer characteristics of the design. Table VIII gives a comparison between internal temperature drops measured for Hyper I and Hyper II capsules and calculated. The calculations were based on having no radiation interference from the fuel container supports. This approach resulted in the lowest temperature drop between the aeroshell stagnation point and the Pt shell.

Hyper II capsule had fuel container supports with radiation interchange ports. These helped heat transfer to the side and bottom of the Pt shell. The Pt top temperature on the Hyper I capsule was somewhat closer to the aeroshell stagnation point than Hyper II. This was probably a result of the higher temperatures on Hyper I which caused increased heat transfer.

Differences between the calculated values and Hyper II results can be a result of contact between the Pt shell and the fuel container surface. A good thermal contact is assumed for the thermal model which would cause more uniform temperatures.

Blistering of the coating may have been caused by tungsten oxide vapor formation in the 0.005 zone of 50% HfO_2 50% W beneath the 0.005 HfO_2 . The diffusion controlled oxidation rate varies as the square root of the pressure. Stagnation pressure at peak heating was considerably higher for these tests than actually occurs during reentry, Table IX, and was correspondingly lower for the 4-inch model tests. This would be an explanation of the better coating performance noted on model 4027 in addition to the larger thickness on the 4-inch model.

Side view

Front view

HfO₂ coating blistered on forward surface. 0.020 T-111 aeroshell breached in stagnation region. Fuel container intact. Pt secondary shell melted at small local areas due to exposure to plasma jet flow.

Fig. 30 - Post-test condition of Hyper II hyperthermal capsule

4-in. dia. fuel container. Part of
0.010 Pt shell vaporized. 0.020
T-111 container intact.

Fig. 31 - Hyper II fuel container after reentry test

TABLE VIII

TEMPERATURE DROP FROM AEROSHELL STAG. POINT TO Pt SHELL

Test Time	Pt Top			Pt Side			Pt Bottom		
	Calc.	Hyper I	Hyper II	Calc.	Hyper I	Hyper II	Calc.	Hyper I	Hyper II
200 sec	480°C	235°C	265°C	610°C	585°C	510°C	700°C	735°C	570°C
300	480	300	360	630	680	635	720	820	700
370	470	290	425	630	720	705	710	860	775
429	480	400	470	640	1080	980	730	1200	1075

TABLE IX
STAGNATION PRESSURE COMPARISON - TEST VS REENTRY

Condition	Stag. Pressure, P_{t_2}	P_{t_2}	P_{t_2}
		$\frac{(P_{t_2})}{(P_{t_2})_{\text{reentry}}}$	$\sqrt{\frac{(P_{t_2})}{(P_{t_2})_{\text{reentry}}}}$
Reentry	0.075 atm.	1.00	1.00
4-inch test	0.0435 atm.	0.58	0.76
Hyper I test	0.19 atm.	2.54	1.59
Hyper II test	0.128 atm.	1.71	1.31

4.0 Material Evaluation

Tests and analyses were conducted to establish processing techniques for applying the reentry protection coatings. Results of the effort is reported in Section 3.2 and 5.1 of the Final Report.

The following sections present some of the detailed data used in the material evaluation.

4.1 W-CVD Coating Performance

A principal reason for conducting the 4-inch hyperthermal wind tunnel model tests was to evaluate the performance of the W-CVD undercoat in providing a contamination barrier for the tantalum alloy during reentry. Bend test coupons were cut from these models after reentry exposure and tested to determine whether contamination had occurred. The bend test results were shown in Tables II and V.

Several conclusions of the coating performance can be made based on the results of these bend tests. These are listed in Table X. The results clearly indicate that the W-CVD undercoat was beneficial in protecting the underlying structure.

Metallographic specimens were cut from the stagnation point of six 4-inch models: three from Phase I tests and three from Phase II tests. These are shown in Figure 32 enlarged 75X and 250X. All had the W-CVD undercoat which can be seen most clearly in the 250X enlargements as the bright continuous layer adjacent to the understructure.

The porous layer which appears above the W-CVD layer on Figure 32a, b, d, e, and f is the plasma sprayed tungsten layer. This does not appear on the Figure 32c specimen which was pyrocarbon reentry coated. The plasma sprayed HfO_2 layer is visible in the center photo of Figure 32b.

Microhardness measurements were made on two of the specimens: 4T12 (Figure 32a) and 4T13 (Figure 32c). This survey is shown in Table XI and shows that 4T12 is harder. These results are in agreement with the bend test results of the coupons given in Table II.

TABLE X

COATING PERFORMANCE ANALYSIS BASED ON BEND TEST RESULTS

Test Analysis	Compared Models
1. W-CVD undercoat better than W-spray - under HfO_2 under Py-C	4T12 : 4T09 4011 : 4018
2. HfO_2 and Py-C protection similar	4019 : 4020
3. HfO_2 coating should be ≥ 0.010 thick	4008 : 4026 : 4027
4. HfO_2 performance same for 30 sec or 8 min. exposure	4019 : 4008
5. Ta and T-111 results similar	4020 : 4T13
6. Stem mount detrimental compared to cone mount	
with HfO_2 coating	4T12 : 4019
with Py-C coating	4011 : 4020

75X

250X

Model: 4T12

Test: Phase I

Reentry coating: 0.019 HfO_2

Structure: 0.020 T-111

Undercoat: W-spray/W-CVD

Mount: stem

Fig. 32a Metallographic specimens from stagnation point of
4-inch hyperthermal model after test

75X

75X with reentry coating

250X

Model: 4019

Test: Phase I

Reentry Coating: 0.013 HfO_2 Structure: 0.020 Ta

Undercoat: W-spray/W-CVD Mount: cone

Fig. 32b Metallographic specimens from stagnation point of
4 in. hyperthermal model after test

75X

250X

Model: 4T13

Test: Phase I

Reentry coating: 0.045 Py-C

Structure: 0.020 T-111

Undercoat: W-CVD

Mount: cone

Fig. 32c Metallographic specimens from stagnation point of
4-inch hyperthermal model after test

75X

250X

Model: 4008

Test: Phase II

Reentry coating: 0.008 HfO_2

Structure: 0.020 Ta

Undercoat: W-spray/W-CVD

Mount: cone

Fig. 32d Metallographic specimens from stagnation point of
4-inch hyperthermal model after test

75X

250X

Model: 4026

Test: Phase II

Reentry coating: 0.005 HfO_2

Structure: 0.020 Ta

Undercoat: W-spray/W-CVD

Mount: cone

Fig. 32e Metallographic specimens from stagnation point of 4-inch hyperthermal model after test

75X

250X

Model: 4027

Test: Phase II

Reentry coating: 0.016 total of
 HfO_2 over 50%
 HfO_2 /50% W

Structure: 0.020 Ta

Undercoat: W-spray/W-CVD

Mount: cone

Fig. 32f Metallographic specimens from stagnation point of
 4-inch hyperthermal model after test

TABLE XI MEASURED MICROHARDNESS SURVEY ON T-111 STRUCTURE

Specimen	Location in 0.003 Steps		DPH Values for 25g Load
4T12	outboard	(1)	429
		(2)	413
		(3)	302
		(4)	258
		(5)	258
	inboard	(6)	274
4T13	outboard	(1)	357
		(2)	351
		(3)	302
		(4)	283
		(5)	274
	inboard	(6)	283

4.2 Collected Tantalum Alloy Data

A literature survey was conducted to establish material strength values to be used in the helium venting analysis of Section 3.3 in the Final Report.

The collected data are summarized in Table XII and shown graphically in Figures 33 through 38.

TABLE XII TANTALUM ALLOY DATA

Test Temp	Source	Pure Ta			T-111			T-222		
		UTS	YS	% Elong.	UTS	YS	% Elong.	UTS	YS	% Elong.
Room Temp	NRC	48-45	35-35	20-40	85-92	75-81	20-24			
	Westinghouse	25	10	35				125	120	20
	Fansteel			30				110	100	28
	Battelle	29.4	26.3	36	90.4	90.4	29			
1093°C =2000°F	Westinghouse					29			49	
	NRC (SR)				*60.7	*55.1				
1200°C	Battelle	11	9		61.7	34.9	18.0			
1315°C =2400°F	NRC	15			40	33.1	41.7			
	Westinghouse					26			35	
	Battelle				37.3	23.5	36.0			
1370°C =2500°F	NRC				37	32	13			
	Westinghouse				37.5	23.5		50	45	20
1480°C	Battelle	5.3	4							
1650°C =3000°F	NRC	5			16-20	17.9	29			
	Fansteel							22	21	25
	Battelle	4.0	3.1		14.8	11.9	48			
1760°C =3200°F	Fansteel							20	18	28
1926°C =3500°F	NRC	2			11.2	11.2	31			
	Fansteel				13	12.6	34	16	14	30
	Battelle	*3.9	*2.8		13	12.6	34			
2000°C =3632°F	NRC	.75								
2052°C	Battelle	*2.78	*1.26	*43						
2204°C =4000°F	NRC				4.2	3.97	33			
2398°C	Battelle	*2.38	*.6	*47						

*Stress Relieved Condition

Fig. 33 - Pure Ta (recrystallized) tensile strength vs temperature

Fig. 34 - Pure Ta (recrystallized) yield strength vs temperature

Fig. 35 - Pure Ta percent elongation vs temperature

Fig. 36 - T-111 tensile strength vs temperature

Fig. 37 - T-111 yield strength vs temperature

Fig. 38 - T-111 percent elongation vs temperature

5.0 Conclusions and Recommendations

The two objectives of the hyperthermal and material evaluation tests have been successfully accomplished: First, it was demonstrated by processing developed under this program that protective coatings can be applied to spheres of both tantalum and T-111. These coatings will maintain the underlying structure in a ductile form during the short reentry of abort or the long-term orbital reentry. Second, the prototype capsule tests evaluated the transient heat transfer within the capsule interior and verified the thermal model used to predict reentry temperatures.

Details of the coating performance were given in Table X. The significant factors of these results are:

1. The W-CVD undercoating provides protection of the tantalum from contamination.
2. Pyrocarbon reentry coating should be sized based on ablation, and
3. Hafnia reentry coating should be a minimum of 0.010 thick to prevent oxidation of the underlying tungsten layer.

An additional result was the substantial insulating benefit obtained from the hafnia coating. A temperature drop of over 150°C per 0.010 inch occurs at the peak heating rate.

The recommended reentry coating could be either pyrocarbon or hafnia but should have the W-CVD undercoating to afford reentry protection. It is further recommended that techniques be devised to reinforce the reentry coatings. One possible method might be to texture the tantalum sphere to provide a mechanical interlock for the plasma sprayed hafnia or deposited pyrolytic carbon. This is especially important for the pyrocarbon which is subject to mechanical shock damage.

Additional hyperthermal tests are recommended using instrumented 8-inch capsules of the Comp I design, i.e. having the tantalum buffer shells and 0.070 T-111 fuel container. These models should have the W-CVD undercoat and a hafnia coating of about 0.015 in. thick. Tests could be conducted with graphite shells as a substitute for the pyrocarbon coating.