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High Temperature
Protective Heat Shield

(Title Unclassified)

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UNITED STATES
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**High Temperature
Protective Heat Shield**

(Title Unclassified)

by

R. E. Riley
C. Landahl

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HIGH TEMPERATURE PROTECTIVE

HEAT SHIELD

by

R. E. Riley

C. Landahl

ABSTRACT

Development of a high temperature heat shield for a critical load-bearing portion of the hot end of a nuclear rocket engine reactor was investigated. This heat shield would be used in a hydrogen atmosphere to protect the load-support button of the tie rod or tie tube.

Tungsten metal alloys having reliable metallurgical and physical characteristics are constantly in demand for advanced nuclear engine applications, and, therefore, were selected for this investigation. The only structural property required of the material was that it be self-supporting at temperatures near 2500°C. A detailed account of the fabrication and testing methods is given, and the high temperature results are discussed.

The results of an actual field test in the Phoebus-1B reactor confirmed the results of laboratory work with the tungsten metal alloy materials. Data from the reactor test are given.

INTRODUCTION

The hot-end-support system of the Phoebus series of nuclear rocket engine reactors included niobium carbide-coated ATJ graphite multihole support blocks through which hot hydrogen passes on its way to the nozzle.

The support blocks were of the type shown in Fig. 1A at the end of this report. They were recessed at the hot end to afford heat shielding of the tie-rod button which receives the load of the core and transmits it from the hot end back through the tie rod to the cold-end support plate.

Analysis of the support system performance in the Phoebus-1A and NRX-A5 reactors showed that cracks at the base of the recess in the ATJ support

block could be hazardous to the tie rod or tie tube if the crack extended into adjacent support-block gas passages. This would produce severe corrosion of the graphite and jeopardize the life of the support system.

Engineering reviews for advanced reactor designs (larger and more efficient engines) called for cyclic operation at higher operating temperatures and longer running times. It was obvious that something had to be done to the support system. Among the solutions offered was shortening of the graphite support block (Fig. 1B) to eliminate the recess and, hence, the stress point at the base of the shoulder. With the recess gone, a heat shield was needed to protect the tie-rod button. It was proposed that the tie-rod

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button be surrounded by a cup resting on the central portion of the short multihole ATJ graphite support block. The base of the heat shield (cup) would become a part of the load train. The support block would be protected from hydrogen attack by a coating of niobium carbide.

Preliminary laboratory experiments tested the feasibility of using refractory carbides (tantalum or niobium) or tungsten metal alloys for the cup. Under simulated reactor operating conditions, i.e., hot-gas tests, the tantalum carbide cups cracked radially at their bases. In similar tests, two tungsten-molybdenum-rhenium cups showed little or no cracking. The results on the metal alloy, therefore, provided the basis for continuing investigations.

One problem of particular concern was that niobium carbide coatings applied by the vapor deposition process did not adhere well to the pyrocarbon load-bearing washers because of the great difference in expansion coefficient as a function of carbon orientation. Cyclic heating popped the coating off the edges of the washers leaving them susceptible to hydrogen attack. Accordingly, encapsulation and other forms of cladding were investigated. Clad or coated washers were not used in the assembly of the Phoebus-1B or Phoebus-2A cores.

In the Phoebus-2A reactor, in which tie tubes circulated the cold H_2 cooling gas from the support structure back to the inlet side of the fuel elements, it was necessary to change the shape of the graphite support block to include a central pedestal on which the pyrographite washer and tie-tube button rested. The reactor engineers felt that a cap configuration together with the cooling afforded the base of the pyrocarbon washer by the tie-tube button was sufficient protection for the anticipated running time of 20 min at rated power of 5,000 MW. The outside diameter of the pedestal was threaded to receive the tungsten alloy heat shield cap. Utilizing tie tubes improves the specific impulse for a given core operating temperature because it eliminates the 13% cold hydrogen bypass flow required for the tie rods of the previous reactor designs. The results of high temperature hot-gas testing and its influence on the selection of materials for both the cup and cap heat shield designs are discussed in detail.

SPECIMEN FABRICATION

Raw Materials

The raw materials used in this investigation include tungsten-molybdenum-rhenium, W-30 wt % Mo alloy powder, and ThO_2 .

1. Tungsten Powder. The tungsten powder used was a nominal 1.33μ average particle size obtained from General Electric. The bulk and tap density were 2.29 and 3.91 g/cc, respectively. The major contaminants were: O_2 , 2000 ppm; Fe, 100 ppm; Cr, 10 ppm; and Mn, 10 ppm. All other elements were below test limits of detection of spectrographic analysis.

2. Molybdenum Powder. The molybdenum powder had a nominal 2.45μ average particle size, and was purchased from Sylvania. The bulk and tap densities were 1.33 and 2.04 g/cc, respectively. The major detectable contaminants were: O_2 , 1200 ppm; and Fe, 3 ppm.

3. Rhenium Powder. The rhenium powder was purchased from Hermann Starck Co., and had a nominal 0.98μ average particle size. The bulk and tap densities were not available. The only major detectable contaminant was 50 ppm of iron.

4. Tungsten 30 wt % Molybdenum Alloy Powder. The alloy powder was purchased from Metals Refining Company, and had a nominal 1.19μ average particle size. The bulk and tap densities were 2.63 and 4.03 g/cc, respectively. The major contaminants were 3300 ppm O_2 and traces (10 to 100 ppm) of silicon and iron.

5. Thoria Powder. The thoria powder was purchased as Lindsay Chemical thorium oxide, code 115. It had a nominal 15μ average particle size, and was ball milled for 117 h to a nominal 1.0μ average particle size. The bulk and tap densities after ball milling were 1.76 and 2.28 g/cc, respectively. The oxygen content was 11.3%. The major detectable contaminants were: N_2 , 1 ppm; Al, 1000 ppm; Si, 50 ppm; Ca, 100 ppm; Cr, 2 ppm; Mn, 5 ppm; and Fe, 20 ppm.

Blending

The elemental constituents were weighed to the nearest 0.1 g and placed in a glass jar with aluminum agitator wires. The jar was sealed, and the charge was blended for 4 h. The charge was then examined visually. If there were segregated areas, it was blended for an additional 4 h. The blend was removed from the jar and passed through a 50 mesh screen. At this point the blend was ready to load into the plastisol hydrostatic pressing sac. If

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more than 2 h elapsed before pressing, the blend was reblended for 30 min.

Pressing and Sintering

The first few pressings were made as solid right cylinders or as tubes by hydrostatic pressing in a plastisol sac with or without a mandrel. It was immediately apparent that a technique had to be developed to conserve the very expensive constituents and minimize the machining time.

This was accomplished by hydrostatically pressing to the cup or cap configuration on a preformed mandrel at 45,000 psi. This system used about half the material required to make a right cylinder. To cut machining cost, the as-pressed preform was given a 15-h pre-sinter treatment at 1000°C in H₂. This allowed H₂ cleanup of the low density (~ 50% of theoretical) pressings and strengthened them enough for subsequent handling and machining. Machining at this point was by conventional methods using carbide tools, and required approximately 1 h per piece. This was compared to the need for nonconventional techniques (electrical discharge machining) and approximately 100 h per piece if the machining was done after final high temperature sinter of a right cylinder.

The machined cap or cup was then sintered in H₂ for 15 h at 1700°C, after which it was transferred to a high temperature furnace for final sintering in H₂ for 1.5 h at 2700°C. The shrinkage of a pressed component was studied as a function of alloy composition and thermal pressing cycles. The selection of the alloy and the thermal cycle, resulting in a reproducible shrinkage factor, determined the machining dimension of the cup after the 1000°C sinter. The final sintered pieces were determined to be within ± 0.005 in. of all finished dimensions, resulting in significant savings in machining cost. Figure 2 shows the step-by-step production of both cap and cup. The cap design involves a threaded section as shown in Fig. 3. The threading is accomplished by electrical-discharge machining after the final sinter and requires approximately 2 h per cap.

TESTING METHODS

We decided to hot-gas test the cup and cap designs as realistically as possible. Therefore, graphite support blocks were made by the Shops Department and coated by the High Temperature Chemistry Group with NbC by chemical vapor deposition from NbCl₅. Mock-ups of the tie-tube and tie-rod

configurations, the two basic test assemblies, are shown in Fig. 4. The cup specimen was held in place on the block by an NbC-coated graphite bolt. The cap specimen was screwed directly onto the NbC-coated support block.

The assemblies were attached to the exit-gas end of a Los Alamos Scientific Laboratory-designed hot dynamic hydrogen environmental test furnace of the type shown in Fig. 5. A detailed cross section of this furnace with the heater and specimen arrangement is shown in Fig. 6.

The furnace can heat 250 lb of hydrogen per hour to above 2500°C while maintaining a vessel pressure of 750 psi. The tests were usually run for 30 min or more. In several cases a cap was recycled to accumulate one or more hours of exposure. Not consistent with actual reactor conditions is the fact that no H₂ gas coolant was used in the supports holding the assembly in place. Therefore, all test results reflect an overtest of actual reactor temperature conditions. The test results are presented in Table I.

TEST RESULTS

The initial development work was done on cups of two arbitrarily chosen W-Mo-Re alloys. These alloys were chosen because we felt that the Mo and Re additions would aid in retaining some ductility after the specimens had been subjected to proposed cyclic or retest conditions.

Test cup 1-1 (Fig. 7)* contained 59.4 wt % W, 20.54 wt % Mo, and 20.42 wt % Re, and was hydrostatically pressed to a right cylinder at 45,000 psi from a powder blend. It was presintered at 1050°C for 15 h in H₂, intermediate sintered at 1700°C for 15 h in H₂, and then sintered at 2700°C in H₂ for 1.5 h to a final density of about 96% of theoretical. The as-sintered structure is shown in Fig. 7-A. The cup was hot-gas tested at 2400°C for 30 min in flowing H₂. No weight change was evident, but NbC from the graphite block was seen to be sticking to the bottom of the cup. There was no visual or metallographic evidence of a reaction on the outside of the cylindrical portion where there were thin sections due to non-uniform centerless grinding. There was a profound reaction on the inside of the cup (Fig. 7-B, C, and

* The magnifications of the microstructures shown in this report are 10% less than is indicated on the figures.

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TABLE I. PROTECTIVE HEAT SHIELD TEST DATA

Specimen No.	PyC Washer Type	Composition	Hydrogen			Run Time, min	Wt. Change, grams	Max. Change, o.d., mils	Appearance and Comments
			Temp., °C	Flow, lb/h	Pressure, psig				
Protective Cups									
1-1	Plain	W-Mo-Re	2400	140	550	30	None	+7.1	Shape OK. Sticking to NbC. Mud-flat cracks on sidewalls.
3-1	NbC-coated	W-Mo-Re-ThO ₂	2400	140	550	30	+0.33	+5.8	Cracked perpendicular to indices on base at two places. Pyrographite NbC sticking to bottom of cup.
4-1	NbC-coated	W-Mo-Re-ThO ₂	2400	140	550	30	+0.12	+5.6	Shape OK. No cracking visible.
4-2	NbC-coated	W-Mo-Re-ThO ₂	2400	140	550	30	+0.16	+6.9	Shape OK. Slight sticking to block. Discolored base.
4-3	Plain	W-Mo-Re-ThO ₂	2400	140	550	30	+0.61	+29.0	Distorted at base. Severe NbC sticking.
13-B	None	W-Mo-ThO ₂	2400	140	550	30	-0.04	+4.8	Badly cracked, yet unthreaded from mating part.
16	None	W-Mo-Re-ThO ₂	2400	140	550	36	None	+19.0	Shape OK. Unthreaded OK.
7-2	None	W-Mo-Re	2400	140	550	30	None	-3.4	Shape OK. Unthreaded OK.
Protective End Caps									
632-25	Plain	W-Mo-ThO ₂	2230	205	625	16	-	+9.0	Cap stuck to block. Fine cracks in base of cap.
632-26	Plain	W-Mo-ThO ₂	2230	205	625	30	+0.36	+17.9	Pinned to block. Had to split cap to unthread from block.
632-1	Mo-coated	W-Mo-ThO ₂	2400	140	550	30	+0.19	+2.1	Shape OK. Reaction zone inside radius of cap.
632-3	Mo-coated	W-Mo-ThO ₂	2400	140	550	30	+0.4	+14.9	Bulging over threads and near rounded end. Crazing in threads.
632-4	None	W-Mo-ThO ₂	2400	140	550	44	+0.17	+12.7	Slight bulging over threads.
632-8	Re spray-coated	W-Mo-ThO ₂	2250	205	625	30	+0.07	+5.4	Vented cap, 1/8-in. hole in crown. Looks OK.
	Ta-sheathed	W-Mo-ThO ₂	2400	140	550	30	+0.19	+5.7	Vented cap, 1/8-in. hole in crown. Shape OK. Cup looks good.
	Ta-sheathed	W-Mo-ThO ₂	2250	140	550	10 (3)	+0.24	+7.5	Same as above.
	Plain	W-Mo-ThO ₂	2250	205	550	30 (2)	-	-	Same as above.
632-9	Re-coated	W-Mo-ThO ₂	2250	140	550	10 (3)	+0.85	+25.0	Looks OK after 96 min.
			2400	140	550	10 (6)	-	-	
			2500	140	550	6	-	-	
632-18	Plain	Vapor-dep. Ta in threads	2400	140	550	30	-	+4.8	Cap split and stuck to block at NbC threads of block.
632-22	Re-Mo coated	W-Mo-ThO ₂	2400	140	550	30	-	-	Test of Re-Mo washer. Cap looks OK.
632-24	Ta-sheathed	W-Mo-ThO ₂	2400	140	550	30	-0.04	-	Cap looks OK. Ta-sheathed washer looks intact.
632-30	Ta-sheathed	W-Mo-ThO ₂	2400	140	550	22	-	-	Cap looks OK.

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D). An NbC-coated pyrolytic carbon (PyC) washer was placed between the cup and the NbC-coated bolt which held it in contact with the support block. The washer was too wide to fit inside the cup and was ground down to fit, thereby exposing the carbon next to the inner surface of the cup where a reaction zone was evidenced, particularly near the cup radius. The depth of the reaction zone at the radius (Fig. 7-B) was nearly half the wall thickness, diminishing to practically nothing at the cylindrical part of the cup (Fig. 7-C and D) farthest from the washer. This cup contained no grain-growth suppressant such as ThO_2 , and as a result of sintering and testing, grain growth was excessive and intergranular cracking was noticed, particularly near the radius of the cup where the reaction was the greatest. Figure 7-E shows what seems to be a liquid-phase reaction in the area adjacent to the PyC washer. The liquid apparently evaporates progressively, leaving the voids in the reacted area.

Cup 4-3 was tested in the same manner as cup 1-1. It was of the same basic composition except that 1.5 wt % ThO_2 was added to the powder blend. This cup gained 0.61 g of NbC from the graphite block which stuck to it. Metallographic examination showed nearly the same results as for cup 1-1 except that there were no cracks and the fine-grained structure due to the ThO_2 dispersion was retained. Figure 8 shows the structure of test cup 4-3 as-sintered (A) and after hot-gas testing (B-D).

Because the NbC coating from the support block stuck to the cup, we decided to use a barrier between the cup and the block to prevent sticking. Cups 3-1, 4-1, and 4-2, of the same composition as cup 4-3 were used to investigate this sticking problem. The cup, block, and barrier arrangements of these specimens are shown in Fig. 9.

Test cup 3-1 had a ThO_2 washer between cup and block and gained 0.33 g during the test. This was NbC from the PyC washer sticking to the inside of the cup and/or oxygen pickup from the ThO_2 washer which disintegrated during the test. Although the ThO_2 washer disintegrated to powder, there was no evidence of the block sticking to the cup.

Test cup 4-1 had a ThO_2 washer next to the bottom of the cup, and a Ta foil washer between the ThO_2 washer and the support block. This cup looked excellent after the test. It gained 0.12 g, attributed

to reaction products from the ThO_2 washer, the Ta foil, and the NbC coating in the presence of hot H_2 . There was no evidence of sticking or cracks even in the NbC-coated PyC washer inside the cup.

Test cup 4-2 had TaC powder plasma arc-sprayed to the inside and outside of its bottom before testing. The cup stuck very slightly to the block and to the NbC-coated graphite bolt used to support the cup. The cup gained 0.16 g, associated with NbC pickup from the PyC washer inside it. The information gained from these three cups suggested that the thorium washer acted as an insulator and prevented sticking. However, the designer preferred a tight fit at this interface to prevent serious erosion from gas leakage. Nevertheless, use of the metal alloy looked most promising for the cup application.

At this point we decided to switch to the threaded cap design. Since some cups were then being fabricated, we decided to thread them to investigate the machining techniques and check the thread design. These cups had been pressed on the cup preform mandrel.

Three different alloys were tested under identical conditions of temperature, time, and H_2 gas flow. Test cup 13-B was 70 wt % W and 30 wt % Mo with 1.5 wt % ThO_2 added. Test cup 16 was 59.72 wt % W, 31.17 wt % Mo, and 9.11 wt % Re with 1.5 wt % ThO_2 added. Cup 7-2 was 59.04 wt % W, 20.54 wt % Mo, and 20.42 wt % Re with no ThO_2 added. The reaction at the threads of these three cups threaded onto the NbC-coated support block is shown in Fig. 10. These metallographic results indicated that the higher the Re content, the less corrosion or attack on the threads. These results have been confirmed by actual reactor test results to be discussed later.

The first two protective caps using W-30 wt % Mo alloy powder plus 1.5 wt % ThO_2 (caps 632-25 and 26), were hot-gas tested at 2230°C exit gas temperature for 16 and 30 min, respectively. Uncoated graphite spacer tubes and pyrocarbon washers used in the test rig resulted in severe attack at the alloy threads. The loss of carbon from the uncoated graphite pieces was about equivalent to the weight gain of the test caps. Figure 11 shows the location and depth of corrosive attack. It was apparent from these tests that all graphite components must have a good NbC coating. The PyC washer, however, poses a coating problem due to differences in thermal expansion.

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sion of this highly oriented structure. The fact that the reaction on the inside dome of the cap appears to be about the same in both cases suggests that gas was leaking around the threads of the cap and block.

Providing a suitable coating or a capsule for the PyC washer appeared to be the next problem. Many approaches were investigated. First, we had to determine to what extent the attack on the threads was due to contact between the threads of the metal alloy cap and the NbC coating on the threads of the block. Therefore, cap 632-4 was tested at 2400°C for 44 min in flowing H₂. The cap was screwed onto the NbC-coated block, and the PyC washer was left out. The results of metallographic examination of this cap are shown in Fig. 12 and should be used for comparison with the results of the subsequent tests to be described.

Cap 632-1 had a Mo-coated PyC washer positioned near its dome (Fig. 13), and was tested in flowing H₂ for 30 min at 2400°C. The NbC coating on the support block held up during the test, but the Mo coating on the PyC washer failed. The corrosive attack on the threads was the least experienced to date. Although cap 632-4 (Fig. 12) showed a greater reaction zone than cap 632-1, it must be remembered that it was run for a longer time (44 vs 30 min) at temperature.

Cap 632-3 had the Mo-coated PyC washer located at the base of the multihole block threads and was tested the same as cap 632-1. The Mo coating on the washer and the NbC coating on the inner bore of the block failed, resulting in catastrophic corrosion of the threads as in shown in Fig. 14. The reaction on the external surface is very pronounced as is shown in Fig. 15 where the outline of its threads can be seen on the outside of the cap.

Caps 632-9 and 632-22 were tested with Re- and Mo-Re-overcoated PyC washers, respectively. Cap 632-9 had a Re spun-preform encapsulated PyC washer, and was first run for three 10-min runs at 2250°C in flowing H₂, amounting to 140 lb/h and a pressure of 550 psig. Visual examination showed the cap to be in excellent condition; therefore, we decided to cycle it six times at 2400°C under identical hydrogen flow and pressure conditions. Again visual examination showed it to look very good. We decided to run it again at 2500°C under identical hydrogen

flow and pressure conditions. The run was terminated after 6 min at temperature because of a furnace abort, and we decided to perform posttest metallographic examination. This examination showed that the carbon had diffused through the Re capsule and resulted in a catastrophic reaction at the threads (Fig. 16), while the Re assumed the shape of the eroded washer (Fig. 17). It should be remembered that this cap ran for a total of 96 min and the reaction at the threads was due not only to the carbon depletion from the washer but also to the contact of the cap threads with those on the NbC-coated support block. On the other hand, cap 632-22 showed only a slight reaction at the threads (Fig. 18). This cap was run for 30 min at 2400°C in hydrogen, as before, and the PyC washer had a chemically vapor co-deposited Mo-Re-overcoat. After the test, the cap looked very good and the Mo-Re overcoat was well bonded to the washer except at the laminations of the basal planes where some (not complete) spalling of the coating occurred (Fig. 19).

Caps 632-24 and 632-30 were run for 30 and 22 min, respectively, at 2400°C in flowing H₂. Both caps contained washers which were encapsulated with Ta spun foil preforms. The reaction at the threads was only slightly more severe than that in the case of the cap with the Mo-Re encapsulated washer. In both cases, the Ta was carbonized and disintegrated on disassembly.

The next cap in this series was 632-18, which had Ta metal vapor deposited on its threads by chemical vapor deposition. The Ta formed a carbide during the hot-gas test; however, the attack (reaction zone) on the threaded region was slight as is shown in Fig. 20A. In this picture one can see that the Ta metal formed a carbide barrier and helped to reduce carburization of the indicated thread. The effect of a nonadherent niobium carbide coating on a graphite support block is also visible in Fig. 20A. The NbC stuck to the threads of the cap on disassembly. It remained stuck to the cap through the cross-sectioning operation, and is visible in the photograph as the looping white band of material somewhat separated from the W-Mo thread. Fig. 20B is another view of the thread area with the parent W-Mo material in the upper right corner and an admixture of TaC + NbC toward the left. Again, the parent material has little evidence of reaction zone with carbon. Figure

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20 C shows a reaction zone at the junction of the last thread where there was no Ta coating.

Attempts to encapsulate the pyrocarbon washer with materials other than the conventional vapor-deposited NbC have not been successful to date. Further attempts to develop a coating for the washer were abandoned because it was demonstrated that with a good coating, normally obtained on the support block, the alloy cap would not suffer adverse corrosion under the proposed operating conditions. This was borne out by the results of test components in the Phoebus 1B reactor test, to be discussed later.

The attack has been attributed to gas leakage around the threads; therefore, the last cap in the series (632-8) was vented with a 1/8 in. hole at the dome to see how this might influence the attack on the threads and on the pyrocarbon washer. The assembly was tested for 30 min at 2250°C with a H₂ flow of 205 lb/h and 625-psig pressure. The pyrocarbon washer was spray-coated with Re. The cap appeared physically undamaged by this test, and we decided to run it again. The second run was for 30 min at 2400°C with a H₂ flow of 140 lb/h and 550-psig pressure. The pyrocarbon washer was encapsulated with spun Ta preforms. Again, the cap looked very good, and we decided to cycle it to 2250°C for three 10-min periods each with the same H₂ flow and pressure conditions. A Ta preform sheet was used for these runs. The cap still looked very good.

We also wanted to mock up a thermocouple probe design for the Phoebus-2 reactor; therefore, we screwed a W-alloy pin into the NbC-coated graphite nut inside cap 632-8, as shown in Fig. 21. The assembly was then cycled twice to 2250°C for 30 min at a H₂ flow of 205 lb/h and 550-psig pressure. This resulted in a total testing time of 2.5 h for this cap. With this in mind, one can evaluate the extent of attack on the cap and pin. Figure 22B shows the extent of the reaction of the threaded region of the pin. This appears to be the only area affected; however, there is a definite separation between the reacted and nonreacted base metal (Fig. 22). The NbC coating on the graphite bolt stuck to the threads of the pin on disassembly.

Figure 22C shows that there has been some carbon reaction with the W-Mo metal in the thread of the cap. However, the thread form is good even though some NbC from the coating of the graphite support block was

stuck to the cap thread. The cap is in remarkably good condition after 2.5 h of hot-gas testing.

PHOEBUS-1B REACTOR TESTS

Excluding the results of cap 632-8 just discussed, a careful study of the data indicated that the W-Mo-ThO₂ cap design would be satisfactory as a heat shield under normal reactor conditions. Therefore, we decided to place six prototype assemblies (two each of three tungsten alloy compositions) into the Phoebus-1B reactor. Uncoated pyrocarbon washers were used in these assemblies. The assemblies were positioned as follows:

Cap Composition, wt %				Core Location, Azimuthal Sector	Radius, Inches from Center
W	Mo	Re	ThO ₂		
70	30	0	+1.5	150 and 330	4.5
59.04	20.54	20.42	+1.5	030 and 210	4.5
59.72	31.17	9.11	+1.5	090 and 270	4.5

The Phoebus-1B reactor ran at planned power of 1500 MW for 30 min on February 23, 1967. All six caps survived and performed the heat-shield job for which they were chosen. Upon disassembly, hot-cell metallographic photos were taken of two of the caps.

Figure 23A shows the microstructure of the threaded region of one of the W-30 wt % Mo + 1.5 wt % ThO₂ caps which was located in sector 150 in the reactor. We believe that the cracking occurred during metallographic preparation. Because of distortion by the hot cell windows, the exact magnification is not known, but it is estimated to be approximately 50x. The reaction zone is not significantly different from that occurring in laboratory tests. Figure 23B shows the microstructure of the threaded region of one of the 59.72 wt % W, 31.17 wt % Mo, 9.11 wt % Re + 1.5 wt % ThO₂ alloy caps which was located in sector 270 in the reactor. The magnification appears to be ~ 60x. The reaction at the threads does not seem to be so severe as that of the W-30 Mo cap; however, it is not known whether the Re is responsible or whether the difference is entirely due to temperature differences in the respective sectors of the reactor. In any case, the results are most encouraging.

SUMMARY

The feasibility of using 70 wt % W, 30 wt % Mo, 1.5 wt % ThO₂ as a metal-alloy protective heat shield in flowing hydrogen at high pressures and temperatures

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for times longer than 30 min has been demonstrated. The metal alloy was shown to be self-supporting up to and above 2400°C. For the times investigated, the outer surface of the metal alloy appeared not to react with parts per million of carbon (hydrocarbons) in the hot flowing hydrogen. The limiting usefulness factor was found to be associated with the reaction zone at the threaded interfaces of the refractory-coated graphite support block and the metal alloy cup. It appeared that carbon transport at this interface had sufficient residence time in which to start carburizing the metal alloy at its threaded area. Nevertheless, for the times involved, the reaction zone did not penetrate the wall, and the metal alloy retained its self-supporting characteristics both in the laboratory and during the actual nuclear rocket engine reactor test. Rhenium, as an additive, up to 20 wt % was included in the metal alloy for retention of some ductility after hot-gas cycling tests. It was soon determined that the metal alloy -- 70 wt % W, 30 wt % Mo, + 1.5 wt % ThO₂ -- survived cycling without serious embrittlement. Therefore, the rhenium could be omitted for this application. However, the 20 wt % rhenium additive did seem to re-

duce the amount of the carbon reaction zone at the threads of the metal alloy cap both in the laboratory and in the actual reactor test. The problems associated with encapsulation of the pyrocarbon load-bearing washer remain to be solved; we feel that coating or encapsulation of the washer would be necessary for reliability if longer reactor operating times were used.

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1A

REGULAR SUPPORT BLOCK

1B

SHORT SUPPORT BLOCK

Fig. 1. Graphite support blocks.

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Fig. 2. Manufacturing steps for protective heat shield components.

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Fig. 3. Tungsten - 30 wt % Molybdenum threaded cap design.

- A - Graphite bolt, NbC coated
- B - Graphite support block, NbC coated
- C - Pyro graphite sleeve (heat shield for tie-rod)
- D - Pyro graphite washer (load bearing heat shield)
- E - Graphite nut, NbC coated
- F - Cup (heat shield)
- G - Cap (heat shield)

Fig. 4. Tie-rod and tie-tube hot-end system mockups and typical cup and cap test assemblies.

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Fig. 5. Hot dynamic hydrogen environmental test furnace.

Fig. 6. Cross section of test furnace.

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Fig. 7. Microstructure of W-Mo-Re alloy cup No. 1-1 (no ThO₂) after testing in hot H₂.

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Fig. 8. Microstructure of W-Mo-Re alloy cup No. 4-3; (containing 1.5 wt. % ThO_2) after testing in hot H_2 .

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CUP INTERFACE REACTION ZONE

100 X

250 X

250 X

Fig. 9. Effect of interface barrier on reaction zones.

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NO Re

9.11 w% Re

20.42 w% Re

250X

250 X

250 X

Fig. 10. Effect of Re content on threaded reaction zone.

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Fig. 11. Effect of time on metal corrosion in the presence of bare graphite washer.

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Fig. 12. Corrosion attack on cap thread due to contact with NbC-coated threaded support block. Cap 632-4.

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Fig. 13. Reaction caused by NbC coating on graphite components and Mo-overcoated PyC washer positioned at dome of cap 632-1.

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632-3

632-1

Fig. 15. External evidence of corrosion attack on cap 632-3 as compared with cap 632-1.

Fig. 16. Corrosion attributed to carbon diffusion through rhenium-encapsulated PyC washer. Cap 632-9.

100 X

100 X

7.5X

7.5X

100 X

Fig. 17. Rhenium-encapsulated PyC washer after hot H_2 testing of cap 632-9.

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Fig. 18. Corrosion of cap 632-22 after test involving a Mo-Re-encapsulated PyC washer.

100 X

250 X

100 X

Fig. 19. Mo-Re-encapsulated PyC washer after hot H_2 testing of cap 632-22.

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Fig. 20. Corrosion attack on Ta-coated threads of cap 632-18 after hot H_2 gas testing.

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Fig. 21. Thermocouple probe mockup.

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A

3X

B

3X

C

100 X

D

100 X

E

100 X

Fig. 22. Corrosion of thermocouple mockup after hot H_2 gas testing.

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Fig. 23A. Reaction zone of a W-30 wt % Mo, 1.5 wt % ThO₂ cap after test in the Phoebus-1B reactor, sector 150. ~ 50x.

Fig. 23B. Reaction zone of a 59.72 wt % W, 31.17 wt % Mo, 9.11 wt % Re + 1.5 wt % ThO₂ cap after test in the Phoebus-1B reactor, sector 270. ~ 60x.

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