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PLUTONIA/CURIA COMPATIBILITY TESTING

APRIL - JUNE 1972
QUARTERLY REPORT NO. 15
J. D. WATROUS

PREPARED FOR THE ATOMIC ENERGY COMMISSION
DIVISION OF SPACE NUCLEAR SYSTEMS
UNDER CONTRACT NO. AT(45-1)-2138

DATE PUBLISHED - JULY 1972

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Donald W. Douglas Laboratories
Richland, Washington

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ABSTRACT

Compatibility tests with plutonia-238 in refractory metal capsules have shown that Mo-46Re is the best container material in exposures at 1800°C for 15,000 to 18,000 hours. Rhenium is also adequate but results in greater transport of plutonia within the container. Compatibility tests of plutonia-molybdenum cermets (PMC) and plutonia-thoria-molybdenum cermets (SSC) in double layer refractory containers at 1500°, 1800°, and 2000°C resulted in complex interactions. The SSC fuel form was equivalent to the PMC fuel form in helium release, degraded more structurally, but indicated less aggressive attack on the container materials. TZM and Mo-46Re appeared to provide the best structural containment at 1500° and 1800°C. T-111 appeared inadequate in all tests conducted at these temperatures. Plutonia and PMC fuel aging specimens with curium-242 additions to accelerate helium generation will complete exposure and be examined during the next quarter. Systems oriented tests are being prepared for the Multi-Hundred Watt Thermoelectric Generator Program with exposure to commence during the next quarter and initial results before the end of the calendar year.

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Section 1

INTRODUCTION

Compatibility between a radioisotope fuel form and its container material is one criterion which must be established for safe use of radioisotope heat sources. Reactions and interaction increase whenever additional materials are included in the system to be evaluated. Materials migrate at elevated temperature by vapor transport and solid state diffusion. Mechanisms and amount of material migration is influenced by temperature, thermal gradients, accessibility, and relative area or volume ratios of various system components. Testing has evolved from simplistic oxide/monometallic systems capable of providing research-oriented results to complex multiple-material, thermal-gradient configurations incorporating mechanical test specimens designed to provide engineering data on specific system requirements. Simplistic tests provided a starting point for selection of more representative and complex combinations to be tested as a partial substitute for high-cost, full-size, heat-source system tests.

Five tasks have been established in this program. Task 1 effort is to study simple plutonia-238 or curia-244 pellets in single containers and determine the stability of a plutonia-molybdenum cermet. Task 2 will determine stability of PMC and SSC fuel forms and the interrelationship with double containment materials combinations. Task 3 studies plutonia with noble metal containers coupled with barriers and external oxygen sinks. Task 4 was added to study the effects of fuel aging on structural stability of plutonia and PMC fuel forms. Task 5 will study the MHW-RTG systems interrelationships mocked up in a sub-scale configuration.

Original goals for this program were to define the best refractory alloy containers for both plutonia and curia under accelerated testing conditions at 1800° and 2000°C, and to determine the rates at which plutonia and curia

interact with these container materials. The intent was to extrapolate reaction rates to lower temperatures and long times. The basic program was devised to test plutonia and curia in contact with a monometallic capsule containing discs in intimate contact with the fuel.

During the initial effort of this program, eighteen plutonia-refractory alloy capsules were fabricated using production-grade plutonia containing approximately 1% nonactinide cationic impurities. All 18 specimens have completed evaluation, with the exception of microprobe examination. A matched set of higher-purity ($\sim 0.23\%$ nonactinide cationic impurities) plutonia/refractory metal specimens was added to the program because it was believed that the original lower-purity plutonia could produce erroneously high attack. Effort on the higher purity plutonia specimens has been completed with the exception of microprobe examination. Eighteen curia-refractory metal capsules were fabricated and exposed; 12 have been evaluated while the remaining six were contingency capsules whose evaluation was not required by the results obtained from the first twelve.

A supplemental effort was added in 1969 to evaluate high-temperature stability of plutonia-molybdenum cermet fabricated at Battelle-Columbus Laboratories. Six cermets were encapsulated in W-25Re for high-temperature exposure. Exposure and evaluation have been previously reported. Two extra unexposed specimens were also evaluated to determine the effect of long-term storage. This work and the original effort to define the best refractory alloy containers for plutonia and curia and determining interaction rates have been identified as Task 1. Task 1 effort has been completed, with the exception of microprobe examination.

Task 2 was designed to evaluate the structural stability of double-containment refractory-alloy materials combinations for PMC (plutonia-molybdenum cermet) using a more systems-oriented testing configuration. Tests with SSC (plutonia-thoria-molybdenum solid-solution cermet) were included to provide comparative information as to differences in stability and compatibility between the two fuel forms. Sixteen PMC and four SSC test specimens have been exposed at 1500° , 1800° , and 2000°C for times from 500 to 5000 hours. Evaluation has been completed, with the exception of microprobe examination.

Task 3 involves evaluating barrier materials between pressed plutonia (PPO) and the noble metals for use in the Multi-Hundred-Watt Radioisotope Thermoelectric Generator (MHW-RTG) program. A set of 10 complex test capsules has completed 1000 and 5000 hour exposures at 1500°C. Evaluation of four test capsules exposed for 1000 hours has been reported (MDC G3129 and MDC G3136). Metallographic results of the six specimens exposed for 5000 hours was reported last quarter (MDC G3142). Limited microprobe work remains to be completed.

Task 4 is an experiment to determine the effects of plutonia fuel aging. The intent of this effort is to produce an equivalent of approximately 2-1/2 years of helium release from the plutonium in 6 months by adding shorter-lived curium-242. A systems-type capsule configuration containing PPO and PMC fuel forms is being thermally treated at 850° and 1200°C for seven months prior to heating to simulate reentry thermal pulses. The original plan included impact testing at Los Alamos Scientific Laboratory, but variable fuel quality negated the potential value of the impact effort. A reduced-scope post-test activity is currently planned for completion starting in the first quarter of GFY 73. Plans are being formulated to start with known-quality fuel using information on the current effort as a basis to develop the follow-on program.

Task 5 was initiated late in the previous quarter to provide a systems-oriented test configuration analogous to the MHW-RTG heat source. Heat source materials volumes and thermal conditions are to be reasonably simulated. Four PPO-fueled and three control (non-fueled) thermal-gradient test specimens containing iridium micro-size tensile specimens, some adjacent to the fuel and others in contact with either the graphitic impact liner or graphite ablator materials will be evaluated. Three PPO-fueled and three control uniform-temperature test specimen containing sub-size iridium tensile specimens will also be evaluated. Maximum effort will be made to provide results to the MHW-RTG program office prior to the end of calendar year

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Section 2

RESULTS

Metallographic evaluation was completed on the original and higher purity plutonia in Task 1 and on the PMC and SSC specimens in Task 2. No effort was expended on the Task 3 MHW-RTG Barrier Tests. Exposure continued on the Task 4 fuel aging experiment. Capsule procurement and hardware development for the Task 5 MHW Mockup Test effort was well underway during the quarter.

2.1 TASK 1 - CURRENT PROGRAM

A status summary of all Task 1 compatibility capsules is given in Table 2-1.

2.1.1 Original Plutonia (~1 w/o metallic impurities) and Improved Purity Plutonia (~0.23 w/o metallic impurities)

Twelve long-term exposures were completed late in the last quarter, six being original plutonia exposed at 1800°C for 18,000 hours and six being improved purity plutonia exposed at 1800°C for 15,000 hours. The as-assembled test configuration had a monometallic inner capsule, small disc, and large disc surrounding the plutonia pellet. A wire wrap around the inner capsule was used as a standoff to minimize solid-state transfer from the tungsten secondary capsules. All capsules were sealed by electron-beam welding and remained intact throughout the extended exposure. Because of the very low plutonia-to-container volume ratio, little difference was noted between the two purity levels. The six alloys investigated each behaved differently, with behavior generally consistent with that reported in the interim report (MDC G2034) for the short-term tests of 1000 hours at 2000°C and 2000 hours at 1800°C.

Tungsten was grossly damaged as shown in Figure 2-1. The small disc originally in contact with the plutonia pellet had a very irregular surface and large internal voids. Mass transport occurred to the colder surfaces such as the capsule corners. The attack to the walls was less than that observed for the short-term tests. The plutonia had migrated from its original location

Table 2-1

TASK 1 - CAPSULE TEST STATUS

DWDL Capsule Designation	Fuel Form		Container Material	Exposure		Status (7/1/72)
	Material	O/Pu or O/Cm Ratio		Temp (°C)	Time (hr)	
1-03	Original Plutonia ↓	2.0	W	2000	1,000	Reported in MDC G2034 ↓ Metallography Complete ↓
2-04		2.0	W-25Re	2000	1,000	
3-04		2.0	Re	2000	1,000	
1-05		1.8	W	2000	1,000	
2-05		1.8	W-25Re	2000	1,000	
3-05		1.8	Re	2000	1,000	
1-01		2.0	W	1800	2,000	
2-03		2.0	W-25Re	1800	2,000	
3-01		2.0	Re	1800	2,000	
4-02		2.0	Mo-46Re	1800	2,000	
5-04		2.0	Mo	1800	2,000	
6-03		2.0	WMoRe	1800	2,000	
1-02		2.0	W	1800	18,000	
2-01		2.0	W-25Re	1800	18,000	
3-02		2.0	Re	1800	18,000	
4-03		2.0	Mo-46Re	1800	18,000	
5-02		2.0	Mo	1800	18,000	
6-01		2.0	WMoRe	1800	18,000	
1-12	Purer Plutonia ↓	2.0	W	2000	1,000	Reported in MDC G2034 ↓
2-08		2.0	W-25Re	2000	1,000	
3-11		2.0	Re	2000	1,000	
1-08		1.8	W	2000	1,000	
2-14		1.8	W-25Re	2000	1,000	
3-14		1.8	Re	2000	1,000	
1-09		2.0	W	1800	2,000	
2-06		2.0	W-25Re	1800	2,000	
3-10		2.0	Re	1800	2,000	
4-06		2.0	Mo-46Re	1800	2,000	
5-06		2.0	Mo	1800	2,000	
6-05		2.0	WMoRe	1800	2,000	

Table 2-1 (Continued)
TASK 1 - CAPSULE TEST STATUS

DWDL Capsule Designation	Fuel Form		Container Material	Exposure		Status (7/1/72)
	Material	O/Pu or O/Cm Ratio		Temp (°C)	Time (hr)	
1-10	Purer	2.0	W	1800	15,000	Metallography Complete ↓
2-07	Plutonia	2.0	W-25Re	1800	15,000	
3-09	↓	2.0	Re	1800	15,000	
4-05		2.0	Mo-46Re	1800	15,000	
5-05		2.0	Mo	1800	15,000	
6-06	↓	2.0	W-MoRe	1800	15,000	
Cm-62	Curia	1.5	W	2000	1,000	Evaluation complete ↓
Cm-64		1.5	W-25Re	2000	1,000	
Cm-61	↓	1.5	Re	2000	1,000	
Cm-63		1.5	Mo-46Re	2000	1,000	
Cm-59		1.5	Mo	2000	1,000	
Cm-60		1.5	W-MoRe	2000	1,000	
Cm-68		1.5	W	2000	10,000	In Storage
Cm-72		1.5	W-25Re	2000	10,000	Evaluation Complete
Cm-67		1.5	Re	2000	10,000	In Storage
Cm-98		1.5	Mo-46Re	2000	10,000	Evaluation Complete
Cm-97		1.5	Mo	2000	10,000	In Storage
Cm-70		1.5	W-MoRe	2000	10,000	In Storage
Cm-66		1.5	W	1800	12,000	In Storage
Cm-65		1.5	W-25Re	1800	12,000	Evaluation Complete
Cm-99		1.5	Re	1800	12,000	In Storage
Cm-94		1.5	Mo-46Re	1800	12,000	Evaluation Complete
Cm-95		1.5	Mo	1800	12,000	Evaluation Complete
Cm-96	↓	1.5	W-MoRe	1800	12,000	Evaluation Complete
AA-52	BCL PMC	~1.85	W-25Re	2000	1,000	Completed ↓
AA-56	↓	~1.85	W-25Re	2000	5,000	
AA-57		~1.85	W-25Re	2000	10,000	
AA-55		~1.85	W-25Re	1800	1,000	
AA-54		~1.85	W-25Re	1800	5,000	
AA-53		~1.85	W-25Re	1800	10,000	
AA-58A		~1.85	Ta-10W	1500	120	
AA-59B	↓	~1.85	None	Ambient	~23,500	

and was present as a meniscus above the upper disc and co-deposited with tungsten along the walls. The plutonia was very substoichiometric and had no metallic precipitates. There was no discernible difference between the two plutonia purity levels. Microhardness values listed in Table 2-2 indicate that, for some unexplainable reason, hardening of the tungsten occurred with higher purity plutonia but not with the lower purity level.

Tungsten-25% rhenium had a large number of subsurface oxide precipitates previously identified as plutonia. Precipitate depth ranged to 10 mils for the original plutonia as shown in Figure 2-2 but only to about 4 mils for the purer plutonia with somewhat fewer precipitates. Hardness of both plutonia containers was harder than the matched control specimen as listed in Table 2-2. Again, the alloy adjacent to the purer plutonia was harder than that adjacent to the lower purity plutonia. The purer plutonia had many small metallic precipitates, while the original plutonia had none. The plutonia appeared less substoichiometric than in the tungsten capsules, probably caused by a lower permeability for oxygen.

Rhenium appeared virtually unaffected except for mass transport to the colder surfaces. This is shown in Figure 2-3 where the small disc at the top of the photo has thinned considerably with material transported to the bottom of the capsule along with "fingers" of oxide. Rhenium hardness appears to be a function of grain orientation, thereby obscuring the effects of plutonia with those caused by orientation. However, it is apparent that the purer plutonia caused some hardening of the rhenium. Capsule walls had a small amount of what appeared to be oxides. There was no plutonia body visible in the capsule either as the original pellet or as thick deposits along the wall. These were the only two capsules where this occurred.

Molybdenum-46% rhenium exhibited the lowest level of detrimental behavior. The thick upper disc and the capsule wall are shown in Figure 2-4 with plutonia between the two surfaces. The only attack to the alloy is a row of small subsurface voids probably caused by the agglomeration of recoil alpha particles into helium bubbles. Hardness of the two capsules was unaffected by the plutonia as indicated in Table 2-2. The behavior of both the alloy and plutonia was identical for the two capsules. The plutonia had no metallic precipitates and was very substoichiometric.

Table 2-2
MICROHARDNESS VALUES FOR ORIGINAL AND
PURER PLUTONIA CONTAINERS
(Vickers diamond indenter; 100-g load; values in kg/mm²)

Alloy	Fuel	Time at 1800°C (hr)	Small Disc	Large Disc	Capsule		
					Inside	Center	Outside
W	Control	18000	409, 403	397, 397	370	382	409
	Control	12000	413, 413	417, 390	390	405	388
	Original	18000	----	*433, 405, 454	380	387	389
	Purer	15000	----	----	420	493	514
W-25Re	Control	18000	514, 519	493, 503	500	508	503
	Original	18000	*536-493, 511		525	530	548
	Purer	15000	542	----	554	606	566
Re	Control	18000	330, 281	Lt grns 464, 394	425	550	554
				Dk grns 268, 264	309	322	357
	Original	18000	----	----	357	348	413
	Purer	15000	*566, 585, 473	----	530	681	554
Mo46Re	Control	18000	351, 329	373, 372	342	345	357
	Original	18000	*363, 370, 359	----	363	360	405
	Purer	15000	----	*356, 380, 366	330	354	394
Mo	Control	18000	193, 183	182, 181	187	182	189
	Control	10000 at	188, 187	182, 190	192,	187	197
	Original	18000	----	*182, 166, 165	182	185	186
	Purer	15000	*176, 177, 182	----	174	177	206
W-30Mo 30Re	Control	18000	459, 417	459, 473	483	454	493
	Original	18000	*450, 443, 464	----	473	483	501
	Purer	15000	*488, 464, 483	----	464	459	464

*Side toward fuel
at 2000°C

Molybdenum exhibited voids, precipitation, and mass transport as shown in Figure 2-5. Additionally, voids were present along the outer surface of the capsule which was adjacent to a secondary tungsten capsule. Hardness measurements showed no major effect attributable to plutonia. Plutonia was present at both the upper disc and along the bottom corners. The original plutonia had fine metallic precipitates as shown in Figure 2-6, while the purer plutonia did not.

Tungsten-30% molybdenum-30% rhenium had oxide precipitates to a significant depth and was the only alloy to exhibit compound formation as shown in Figure 2-7. The compound appeared in both capsules with somewhat greater compound being present in the purer plutonia capsule. There was no major effect of plutonia on microhardness. The original plutonia pellet remained intact after exposure, the only one to do so in this test series. The plutonia in both capsules had extensive metallic precipitates and had about the same appearance as in the Mo-46Re capsules.

Six specimens will be sent to Westinghouse-Hanford for microprobe examination which will be performed in July or August. A topical report will document this portion of the program.

2.1.2 Curia-244

Further work has been deferred to allow concentration on MHW tasks.

2.1.3 Battelle-Columbus Laboratories Plutonia Molybdenum Cermet

Metallography has been completed on two cermet specimens aged at room temperature for almost three years. One specimen was heated to 1500°C for 120 hours to remove helium generated during storage. Figure 2-8 shows the edge of the room temperature aged specimen. The plutonia spheres appear cracked but are generally intact and slightly substoichiometric. The molybdenum network surrounding the plutonia remains completely intact. The molybdenum (white) and thoria (multi-layer gray) coatings over the cylindrical surface also remained intact. Heating damaged the cermet structure near the molybdenum coating as shown in Figure 2-9. The plutonia fragmented and was lost near the surface. The molybdenum network became porous. The surface molybdenum coating reacted to form

a nonmetallic appearing layer. The thoria coating was fractured and developed a completely different appearance.

Total degradation of the aged and thermally treated material was considerably greater than for a comparably heated new cermet. The structure, particularly at the specimen periphery, was far more porous than that exhibited in a specimen heated by BCL for 1000 hours at 1500°C. Fragmentation of the microspheres also occurred throughout the bulk of the specimen which also did not occur in fresh material. Helium release measurement of the aged material indicated that 88% of the stored helium was released by the thermal treatment. An expected value for a relatively new cermet heated for comparable conditions would be less than 20% release. This indicates that a cermet stored at or near room temperature for a long time will release the large majority of the stored helium in one burst during a reentry pulse resulting in major structural degradation.

2.2 TASK 2 - PMC AND SSC TESTING PROGRAM

Twenty refractory alloy capsules containing PMC (plutonia-molybdenum cermet or SSC (solid-solution cermet) fuel pellets have been exposed at 1500°, 1800°, and 2000°C for times from 500 to 5000 hours. Each specimen has a cylindrical PMC or SSC fuel pellet within a thin-wall primary container and a thick-wall secondary container. Polished thin discs of the primary capsule material were placed in direct contact with the fuel ends in an attempt to provide more precise information as to penetration depth into the primary container. Evaluation of six of the shorter-term specimens have been reported in the 11th (MDC G3124) and 12th (MDC G3129) quarterly reports and two 5000 hour specimens were reported in the 14th (MDC G3142) quarterly report. Task status is listed in Table 2-3. Helium release measurements were completed on three of four attempts. The fourth specimen was too hard to drill successfully. Results are reported in Table 2-4. It appears that PMC and SSC are comparable, based on the behavior of the two specimens heated at 1500°C for 5000 hours with both having released approximately 75% of the total helium present. A second comparison can be made between the SSC heated at 1800°C for 5000 hours and PMC specimens fabricated by Battelle Columbus Laboratories and tested previously. The SSC released 91% of its total helium whereas the PMC exposed under the same conditions released

Table 2-3
TASK 2 - PMC-SSC TEST CAPSULE STATUS

DWDL Capsule Designation	Fuel Form		Container Material		Temp (°C)	Time (hr)	Status (7/1/72)
	Material	O/Pu Ratio	Liner	Structural Member			
PU266	PMC	1.85	Ta-10W	T-111	1500	500	MDC G3124, MDC G3129
PU267	PMC	1.85	Ta-10W	T-111	1500	5000	MDC G3142
PU268	PMC	1.85	TZM	T-111	1500	5000	Metallography Complete
PU272	PMC	1.85	W-25Re	T-111	1500	5000	Metallography Complete
PU276	PMC	1.85	Mo-46Re	T-111	1500	5000	MDC G3142
PU269	PMC	1.85	TZM	TZM	1800	1000	MDC G3124, MDC G3129
PU273	PMC	1.85	W-25Re	T-111	1800	1000	MDC G3124, MDC G3129
PU277	PMC	1.85	Mo-46Re	TZM	1800	1000	Metallography Complete
PU270	PMC	1.85	TZM	Mo-46Re	1800	5000	Metallography Complete
PU274	PMC	1.85	W-25Re	TZM	1800	5000	Metallography Complete
PU278	PMC	1.85	Mo-46Re	TZM	1800	5000	Metallography Complete
PU280	PMC	1.85	W-MoRe	T-111	1800	5000	Metallography Complete
PU271	PMC	1.85	TZM	W-25Re	2000	1000	MDC G3124, MDC G3129
PU275	PMC	1.85	W-25Re	W-25Re	2000	1000	Metallography Complete
PU279	PMC	1.85	Mo-46Re	W-25Re	2000	1000	MDC G3124, MDC G3129
PU281	PMC	1.85	W-MoRe	W-25Re	2000	1000	MDC G3124, MDC G3129
PU262	SSC	>1.85	Ta-10W	T-111	1500	5000	Metallography Complete
PU263	SSC	>1.85	TZM	T-111	1800	5000	Metallography Complete
PU264	SSC	>1.85	W-25Re	T-111	1800	5000	Metallography Complete
PU265	SSC	>1.85	W-MoRe	W-25Re	1800	5000	Metallography Complete

Table 2-4
HELIUM RELEASE VALUES FOR PMC AND SSC

DWDL Specimen Designation	Fuel Form	Exposure Temp (°C)	Conditions Time (hr)	Helium Released (cc)	Helium Generated (cc)	Helium Stored as Fabricated (cc)*	Total Helium (cc)	Helium Release (%)
PU268	PMC	1500	5000	0.85	0.93	0.19	1.12	76
PU262	SSC	1500	5000	0.76	0.86	0.15	1.01	75
PU263	SSC	1800	5000	0.82	0.75	0.15	0.90	91
AA-54**	BCL							
	PMC	1800	5000	1.96	1.64	0.41	2.05	96
AA-56**	BCL							
	PMC	2000	5000	2.06	1.70	0.40	2.10	98

*Battelle-Columbus Laboratories has estimated that 0.1 cc of helium is stored per gram of cermet at the time of fabrication

**Specimens fabricated by Battelle-Columbus Laboratories and reported previously (MDC G2055)

96% of its total helium. The values are comparable, particularly considering the difference in fabrication techniques for the dissimilar specimens. The major factor open to question is the value used for helium stored at the time of fabrication. Battelle-Columbus has estimated a storage after hot pressing of 0.1 cc helium per gram of cermet. This may have been reduced somewhat in the Los Alamos produced PMC and SSC specimens which underwent 1200°C outgassing operation subsequent to hot pressing. Reduction of stored helium to a lower value such as 0.05 cc helium per gram of cermet would not substantially affect these observations. Measurements were made at room temperature after exposures were completed and a substantial pressure of helium was present at temperature, possibly inhibiting a higher level of helium release from the structure. The amount of inhibition, if any, is unknown.

Three specimens, PU262, PU268, and PU272 have been examined metallographically after exposure at 1500°C for 5000 hours. All specimens had T-111 structural or secondary containers which had large amounts of oxide precipitation throughout the structure. PU262 containing SSC, a Ta-10W liner, and a T-111 structural member exhibited less attack to both the liner and structural member than did a matched PMC specimen (PU267) discussed in the 14th quarterly report (MDC G3142). The Ta-10W exhibited both intergranular attack to a depth of approximately 3 mils, as well as subsurface void formation as shown in A, Figure 2-10. Precipitation is evident throughout the Ta-10W as shown in both A and B, Figure 2-10. An oxide interface is present between the Ta-10W and the T-111. Large oxides are precipitated throughout the T-111. Hardness of both alloy layers, as listed in Table 2-5, has not been affected by the presence of the SSC. Structurally, however, degradation is evident. Only a slight sliver of the SSC remained in the capsule after sectioning resulting in insufficient area for interpretation.

PU268 including PMC, TZM liner, and T-111 structural member had the best appearing fuel-liner combination of this group. The TZM liner had no attack at the interface with the PMC. A grain size gradient occurred across the liner as shown in A, Figure 2-11, probably caused by earlier precipitation of oxides near the surface thereby inhibiting grain growth. Reaction occurred

between the TZM and T-111 as shown in B, Figure 2-11. It appears that molybdenum is diffusing into the T-111 and that oxide precipitation occurred along grain boundaries within the TZM. Heavy oxide precipitation throughout the T-111 aligned primarily in stringers along the rolling direction. This form of precipitation infers that microinhomogeneity is still present in current commercial T-111 bar stock. Hardness results, as listed in Table 2-5, are inconclusive. The PMC had a large crack and also some porosity but retained both the metal network and molybdenum overall coating.

PU272 containing PMC, W-25Re, and T-111 appeared to have the highest oxygen content in the PMC of any of the PMC specimens, but the W-25Re and T-111 were seriously degraded. The PMC had lost its coating, but the network remained intact. The W-25 Re had oxide precipitates and voids throughout as shown in Figure 2-12. The T-111 had gross precipitation. The W-25Re and T-111 hardnesses were only slightly above comparable control specimens. Coating from the PMC pellet appeared to have transferred to the W-25Re surface. Structure of the PMC remained intact with a relatively large level of porosity.

Examination was completed during the quarter on the last specimen exposed at 1800°C for 1000 hours after considerable delay in attempting to perform helium release measurement. PU277 containing PMC, Mo-46Re, and TZM showed no attack to the Mo-46Re but massive precipitation in the TZM. The surface of the Mo-46Re appears irregular because of molybdenum transport from the cermet fuel to the container surface. The surface irregularity is shown in Figure 2-13 along with the precipitation in the TZM. The Mo-46Re shows no hardness change, but the TZM has been hardened somewhat by the precipitation, as indicated in Table 2-6. Transfer of the PMC coating to the capsule wall along with some plutonia is evident in Figure 2-13. The PMC body remains intact, except for the material transfer. The molybdenum network remained intact but the plutonia was quite porous with a relatively high oxygen content.

Seven specimens were evaluated after exposure at 1800°C for 5000 hours. Two of three SSC fuel bodies lost their metal network during exposure. The networks were lost in W-25Re and W-30Mo-30Re containers but was retained in a TZM container. Similarly, the molybdenum fuel body coating was lost

Table 2-5
PMC-SSC CONTAINER MICROHARDNESS
EXPOSED AT 1500°C FOR 5000 HOURS

(Vickers diamond indenter, 100-g load; values in kg/mm²)

DWDL Capsule Designation	Fuel Form	Capsule		Location in Capsule	Location in Specimen		
		Layer	Alloy		Adjacent to Fuel	In Center	Away from Fuel
PU262	SSC	Primary	Ta-10W	Wall	222	221	209
		Secondary	T-111	Wall	235	206	233
8-9B	Control						
PU268	PMC	Primary	TZM	Wall	212	205	195
		Secondary	T-111	Wall	233	253	225
PU272	PMC	Primary	W-25Re	Wall	498	560	548
		Primary	W-25Re	Bottom Disc	517	511	517
		Secondary	T-111	Wall	258	256	258

Table 2-6
PMC CONTAINER MICROHARDNESS
EXPOSED AT 1800°C FOR 1000 HOURS
(Vickers diamond indenter, 100-g load; values in kg/mm²)

DWDL Capsule Designation	Fuel Form	Capsule		Location in Capsule	Location in Specimen		
		Layer	Alloy		Adjacent to Fuel	In Center	Away from Fuel
PU277	PMC	Primary	Mo-46Re	Wall	351	351	317
		Primary	Mo-46Re	Bottom Disc	325	357	360
		Secondary	TZM	Wall	212	206	206
4-7A	Control	Primary	Mo-46Re	Wall	354	351	357
		Secondary	TZM	Wall	198	184	186

in all PMC specimens contained in W-25Re, Mo-46Re, and W-30Mo-30Re capsules but retained in a TZM capsule. It is apparent that TZM containers result in enhanced cermet stability for some, as yet undefined, reason. Reactions in the double containers were too complex to be summarized other than to say that the Mo-46Re liner with a TZM structural member appeared to behave best under these test conditions. Results for the seven 1800°C, 5000 hour specimens follow.

PU263 with SSC, TZM liner, and T-111 structural member partially retained the metal network in the SSC, but the T-111 had massive oxide precipitation. There was no attack to the TZM surface, but the interface between the TZM and T-111 was identical to that exhibited at 1500°C in Figure 2-11, except for an increase in extent. Neither the damage to the TZM or the precipitation is evidenced in the hardness measurements listed in Table 2-7. Surface molybdenum coating remained relatively intact during exposure as did the molybdenum network, as shown in Figure 2-14. The molybdenum has some porosity and appears to be globularizing and dissolving in the oxide matrix. The oxide phase appears to be predominately single phase with unusual crack and multi-phase formation in erratic regions. The reason for this remains unknown until microprobe examination results have been obtained.

PU264 containing SSC, W-25Re liner, and a T-111 structural member exhibited major damages to both alloys. The W-25Re had massive oxides to approximately 30 mils deep in some areas. Lesser attack is shown in A, Figure 2-15, along with the hard (508 VHN) compound layer between the two alloys and the massive precipitation in the T-111 coupled with oxides in the grain boundaries. The W-25Re is somewhat softer adjacent to the SSC, as listed in Table 2-7, probably because of the dissolution and diffusion of molybdenum into the W-25Re. The SSC shown in B, Figure 2-15, has no residual molybdenum network. The structure appears typical of substoichiometric plutonia. Porosity appears less than that exhibited by PMC bodies exposed under the same conditions.

PU265 containing SSC, a W-30Mo-30Re liner, and a W-25Re structural member was also grossly attacked. The W-30Mo-30Re liner had oxides to about 15 mils deep. The W-25Re structural member had voids and oxide precipitates to about 10 mils deep. The SSC remained intact but had neither

Table 2-7
 PMC-SSC CONTAINER MICROHARDNESS
 EXPOSED AT 1800°C FOR 5000 HOURS
 (Vickers diamond indenter, 100-g load; values in kg/mm²)

DWDL Capsule Designation	Fuel Form	Capsule		Location in Capsule	Location in Specimen		
		Layer	Alloy		Adjacent to Fuel	In Center	Away from Fuel
PU263	SSC	Primary	TZM	Wall	188	195	195
		Secondary	T-111	Wall	249	215	209
PU264	SSC	Primary	W-25Re	Wall	468	464	483
		Primary	W-25Re	Bottom Disc	376	493	450
		Secondary	T-111	Wall	281	206	240
2-9B	Control	Primary	W-25Re	Wall	498	496	514
		Primary	W-25Re	Disc	500	498	498
		Secondary	T-111	Wall	225	232	233
PU265	SSC	Primary	W-30Mo 30Re	Wall	454	464	514
		Primary	W-30Mo 30Re	Bottom Disc	441	450	420
		Secondary	W-25Re	Wall	493	493	498
6-2A	Control	Primary	W-30Mo 30Re	Wall	473	483	468
		Primary	W-30Mo 30Re	Disc	450	459	446
		Secondary	W-25Re	Wall	498	483	530
PU270	PMC	Primary	TZM	Wall	230	230	242
		Primary	TZM	Bottom Disc	188	198	217
		Secondary	Mo-46Re	Wall	272	330	405
7-4	Control	Primary	TZM	Wall	189	182	187
		Primary	TZM	Disc	193	345	201
		Secondary	Mo-46Re	Wall			
PU274	PMC	Primary	W-25Re	Wall	420	493	394
		Primary	W-25Re	Bottom Disc	446	508	488
		Secondary	TZM	Wall	183	192	201

Table 2-7 (Continued)

PMC-SSC CONTAINER MICROHARDNESS
EXPOSED AT 1800°C FOR 5000 HOURS

(Vickers diamond indenter, 100-g load; values in kg/mm²)

DWDL Capsule Designation	Fuel Form	Capsule		Location in Capsule	Location in Specimen			
		Layer	Alloy		Adjacent to Fuel	In Center	Away from Fuel	
2-7	Control	Primary	W-25Re	Wall	514		508	488
		Primary	W-25Re	Disc		464	473	
		Secondary	TZM	Wall	189	192		202
PU278	PMC	Primary	Mo-46Re	Wall	314		327	274
		Secondary	TZM	Wall	185	180		186
4-7B	Control	Primary	Mo-46Re	Wall	340		345	351
		Primary	Mo-46Re	Disc		317	314	
		Secondary	TZM	Wall	188	192		188
PU280	PMC	Primary	W-30Mo- 30Re	Wall	429		409	493
		Secondary	T-111	Wall	225	210		215
6-9	Control	Primary	W-30Mo- 30Re	Wall	446		459	468
		Primary	W-30Mo- 30Re	Disc	446		450	450
		Secondary	T-111	Wall	205	222		221

the original molybdenum coating or network. The structure appeared somewhat different than that in PU264, but this may have been caused by a lesser cladding permeability to and sink for oxygen in the fuel.

PU270 contained PMC, TZM, and Mo-46Re. The TZM had precipitation throughout and oxides to a depth of 6 to 7 mils. Hardness was increased by this precipitation. As shown in A, Figure 2-16, the TZM remained intact and bonded to the Mo-46Re structural member along the line where precipitation ceased. The Mo-46Re was softened along the interface because of molybdenum diffusion. In one area, voids occurred in the Mo-46Re. The PMC remained intact, including both the overcoat and molybdenum network as shown in A, Figure 2-16. The PMC had several large cracks, many large voids, and had what appeared to be the lowest oxygen content for the three PMC specimens.

PU274, which included PMC, W-25Re, and TZM, exhibited a substantial damage within the W-25Re as shown in Figure 2-17. Voids and oxide precipitates were present to a depth of 20 mils or more. The TZM appears relatively undamaged, with no apparent hardening attributable to precipitation. The two layers bonded together with void formation but had no compounds, as expected. The PMC remained intact but had lost the overcoat. The oxygen content appeared to be highest of the PMC specimens because of the lower permeability of W-25Re to oxygen.

PU278 containing PMC, Mo-46Re, and TZM exhibited the least attack of this group of specimens. The Mo-46Re had deposited material on the surface but exhibited no attack, as shown in Figure 2-17. The Mo-46Re was hardest in the center because of molybdenum diffusion from the deposit on the inside and contact with the TZM on the outside. The two layers were well bonded. There was no attack to the TZM shown either metallographically or by microhardness change. The PMC remained intact with the least number of voids and had an intermediate oxygen content, which is expected from oxygen permeability/sink characteristics of the container materials.

PU280 had PMC, W-30Mo-30Re, and T-111 as contents. The W-30Mo-30Re liner had porosity on the inside extending to a depth of approximately 10 mils accompanied by some oxide precipitation as shown in A, Figure 2-19. A large intermetallic phase was formed at the interface between the alloys as

shown in B, Figure 2-19. The T-111 exhibited large amounts of oxide precipitation in stringers along the working direction of the original barstock. The PMC coating was gone, apparently transferred to the capsule walls, along with several mils of plutonia. The metal network was degraded considerably.

One remaining 2000°C, 1000-hour specimen was examined after unsuccessfully attempting to measure helium release. PU275 containing PMC and two W-25Re containers showed considerable degradation to the PMC and the inner W-25Re container but no attack to the outer W-25Re container. The edges of the PMC were very porous, but the core remained intact as shown in Figure 2-20. Oxygen content remained relatively high, having two low-permeability containers surrounding the fuel. The inner W-25Re container was as bad as that shown in Figure 2-17 from PU274. There was some oxide penetration in the weld area of the secondary container, however, there was no attack elsewhere. Hardness of the W-25, as listed in Table 2-8, showed no degradation caused by the plutonia. The double W-25Re combination appeared the most satisfactory of the 2000°C containment systems investigated.

The only remaining experimental effort is to complete microprobe examination of selected specimens. A final report will be written upon completion of that examination.

2.3 TASK 3 - MHW-RTG BARRIER TESTS

Microprobe examination by LASL of the plutonia from MHW-31 failed to disclose any evidence of extraneous material in the plutonia. No further attempts will be made to determine whatever the cause for the unusual structure reported in the last quarterly report (MDC G3142).

Further microprobe examination at Westinghouse Hanford was held in abeyance until the end of the fiscal year.

2.4 TASK 4 - PLUTONIA FUEL AGING

Fuel aging specimens currently being exposed at 850° and 1200°C will complete a seven-month exposure on July 3 and will be terminated at that time for evaluation. Table 2-9 lists the specimens and their thermal treatment prior to examination. Helium release will be performed on eight specimens selected

Table 2-8
 PMC CONTAINER MICROHARDNESS.
 EXPOSED AT 2000°C FOR 1000 HOURS
 (Vickers diamond indenter; 100-g load; values in kg/mm²)

DWDL Capsule Designation	Fuel Form	Capsule		Location in Capsule	Location in Specimen		
		Layer	Alloy		Adjacent to Fuel	In Center	Away from Fuel
PU275	PMC	Primary	W-25Re	Wall	464	514	519
		Primary	W-25Re	Disc	420	493	483
		Secondary	W-25Re	Wall	516	516	514
2-2	Control	Primary	W-25Re	Wall	530	508	503
		Secondary	W-25Re	Wall	468	493	493

Table 2-9
TASK 4 - FUEL AGING CAPSULE TEST STATUS

DWDL Capsule Designation	Fuel Form	Container Materials		Exposure		Reentry Simulation (1)	Status (7/1/72)
		Liner	Structural Member	Temp. (°C)	Time (mo)		
420	PMC	Ta-10W	T-111	Ambient	7	T/P	Exposure begun 12/3/71
416				850		None	
417				850		T/P	
418				1200		None	
419				1200		MHW	
425	PMC + Cm			Ambient		T/P	
422				850		None	
421				850		T/P	
423				1200		None	
424				1200		MHW	
428	PPO (HP)			Ambient		MHW	
427				850		None	
426				850		T/P	
430				1200		None	
429				1200		MHW	
431	PPO + Cm			Ambient		MHW	
433				850		None	
432				850		T/P	
434				1200		None	
435				1200		MHW	
436	PPO (CPS)		Ta-10W	850		TBD	
437	PPO (CPS)		Ta-10W	1200		TBD	

(1) T/P is a Transit/Pioneer thermal reentry transient;
MHW is a Multihundred Watt thermal reentry transient.

to represent more likely potential use of the PPO and PMC fuels. Examination will be completed next quarter.

2.5 TASK 5 - MHW-RTG MOCKUP TESTS

Agreement has been reached among AEC-SNS, GE-IPSO, and DWDL as to a final test matrix to be used for the MHW-RTG Mockup Tests. The major concern was with the small size of the DWDL micro-tensile specimens and the ability to correlate test results with the comprehensive GE program to characterize iridium mechanical properties. The test matrix is listed in Table 2-10.

Four fueled "T" thermal gradient tests will be run in conjunction with three matched unfueled "C" control tests. Each of these assemblies includes a LASL-supplied production-grade plutonia pellet, four DWDL micro-tensile specimens adjacent to the fuel, a vented iridium can, impregnated Thornel-50 graphite with four more micro-tensile specimens, POCO graphite, a larger iridium container, and two guard containers for the vacuum furnace. The present proposed thermal gradient is 1370°C for the PPO/Ir interface with the upper end of the POCO graphite to be approximately 100°C cooler. Temperatures are subject to review and modification prior to startup. The ultra-high vacuum Varian VF400 furnaces to be used have been modified to produce measured, controllable differential temperatures from about 50° to 400°C.

Three fueled "M" uniform temperature test assemblies will be run in conjunction with three unfueled "MC" control tests. Each assembly contains an iridium subassembly containing six GE sub-size iridium tensile specimens and MRC production-grade plutonia powder which is then placed in a vented iridium can, impregnated Thornel 50 and POCO graphites, a larger iridium can, and two guard containers for the vacuum furnace. Plutonia powder surrounding the iridium tensile specimens will produce a worst-case situation for potential degradation.

The current schedule required completion of evaluation of the short term (1500/3000 hour) tests sometime prior to the end of 1972. Longer-term tests will then permit corroboration of the short-term test data. The 1850°C thermal ramp is included to determine whether a reentry thermal pulse has any effect on iridium properties and system interactions.

Table 2-10
MHW-RTG MOCKUP TEST MATRIX

Fuel Test No.	Matched Control No.	Fuel Form	Tensile Specimen	Time (hr)	1850°C Ramp	Temperature (°C)
T-1	C-1	Pellet	DWDL Micro	1500/3000	No	1370/1270*
T-2	---	Pellet	DWDL Micro	Optional	No	1370/1270*
T-3	C-3	Pellet	DWDL Micro	1500/3000	Yes	1370/1270*
T-4	C-4	Pellet	DWDL Micro	Optional	Yes	1370/1270*
M-1	MC-1	Powder	GE Subsize	1500/3000	No	1370 Uniform
M-3	MC-3	Powder	GE Subsize	1500/3000	Yes	1370 Uniform
M-4	MC-4	Powder	GE Subsize	Optional	Yes	1370 Uniform

*Final thermal gradient to be provided by SNS prior to startup.

A tensile impact machine (Figure 2-21) is being fabricated for testing the radioactive iridium specimens at temperatures to 1370°C. The basic framework has been fabricated. Checkout of the testing system has demonstrated the ability to measure load and grip movement in room temperature testing of nickel-270 micro-size specimens. Grip separation has been measured to be in the range of 6 fps by use of both proximeters and high speed (~4000 frames per second) motion pictures. The next task is to obtain the required temperature. Direct resistance heating will be attempted first. It is expected that considerable difficulty will be encountered in obtaining a reasonably uniform temperature ($\Delta T \sim 100^\circ\text{C}$) across the gage length of the micro-size specimen. Initial heating will be performed in air and changed to inert gas only if necessary to protect the heated grips. Initial testing will be performed on the DWDL micro-tensile specimens shown in B, Figure 2-22, although it is expected that similar capability will be shown for the GE sub-size specimens shown in A, Figure 2-22.

Equipment has been designed and fabricated for measuring helium flow through the iridium capsule vents prior to assembly and subsequent to exposure. Comparisons will be made as to plugging propensity.

All hardware for test assemblies is on order. Iridium capsule components will be available early in July. Plutonia pellets and powder will be available at the end of July. The first iridium tensile specimens will be available early in August with continued delivery through the month. POCO graphite parts will be available in early August, but the impregnated Thornel-50 graphite may be delayed to near the middle of August. Guard capsules will be completed by mid-August. Component assembly will begin in early August with fueling to proceed upon receipt of the final components.

Figure 2-1. Tungsten/Original Plutonia, 1800°C, 18,000 hr

Figure 2-2. W-25Re/Original Plutonia, 1800°C, 18,000 hr

Figure 2-3. Rhenium/Purer Plutonia, 1800°C, 15,000 hr

Figure 2-4. Mo-46Re/Original Plutonia, 1800°C, 18,000 hr

50x (20 MILS)

Figure 2-5. Mo/Purer Plutonia, 1800°C, 15,000 hr

200x (5 MILS)

Figure 2-6. Mo/Original Plutonia, 1800°C, 18,000 hr

Figure 2-7. W-30Mo-30Re/Purer Plutonia, 1800°C, 15,000 hr

Figure 2-8. Room-Temperature Aged BCL-PMC

(100x (10 MILS))

Figure 2-9. Aged and Heated BCL-PMC

A. Ta-10W SURFACE
200x (5 MILS)

B. Ta-10W/T-111 INTERFACE
200x (5 MILS)

Figure 2-10. Ta-10W and T-111 from PU262, 1500°C, 5000 hr

50x (20 MILS)

A. PMC/TZM/T-111

200x (5 MILS)

B. TZM/T-111 INTERFACE

Figure 2-11. PMC/TZM/T-111 from PU268, 1500°C, 5000 hr

50x (20 MILS)

Figure 2-12. PMC/W-25Re/T-111 in PU272, 1500°C, 5000 hr

Figure 2-13. PMC/Mo-46Re/TZM in PU277, 1800°C, 1000 hr

Figure 2-14. SSC in PU263, 1800°C, 5000 hr

A. W-25Re/T-111

50x (20 MILS)

B. SSC

50x (20 MILS)

Figure 2-15. PU264, 1800°C, 5000 hr

A. TZM/Mo-46Re

100x (10 MILS)

B. PMC

50x (20 MILS)

Figure 2-16. PU270, 1800°C, 5000 hr

Figure 2-17. W-25Re/TZM from PU274, 1800°C, 5000 hr

Figure 2-18. PMC/Mo-46Re/TZM from PU278, 1800°C, 5000 hr

50x (20 MILS)

A. PMC/W-30Mo-30Re/T-111

200x (5 MILS)

B. W-30Mo-30Re/T-111 INTERFACE

Figure 2-19. PU280, 1800°C, 5000 hr

Figure 2-20. Macrograph of PU275, 2000°C, 1000 hr (3x)

Figure 2-21. Tensile Impact Machine

A. GE SUB-SIZE

ALL DIMENSIONS IN INCHES

B. DWDL MICRO-SIZE

Figure 2-22. Tensile Specimens

Section 3

CONCLUSIONS

Results from the plutonia/refractory alloy container tests are relevant for preliminary judgement as to satisfactory containers for radioisotope heat sources. However, more complex configurations such as used in the PMC and SSC testing are required for typical engineering systems, and even then, must be evaluated as to applicability for use in real heat sources.

Long-term plutonia-refractory alloy tests at 1800°C show that Mo-46Re is the best overall container alloy. Small subsurface voids related to alpha recoil from the plutonia appear to be the only minimal damage. Rhenium appears to be totally unaffected by exposure to plutonia at 1800°C but apparently enters into a mass transport of plutonia phenomenon resulting in movement of plutonia throughout the container. Movement appears to be inhibitable by reduction in oxygen content of the plutonia and lowering the temperature. Rhenium would be the best material to inhibit transport of oxygen through a barrier to the structural member. The major problem is the ability to produce and maintain a completely sealed rhenium system. Tungsten, W-25Re, molybdenum, and W-30-Mo-30Re all undergo degradation in excess of that in Mo-46Re reducing desirability of their use with plutonia.

Because of the relatively small volume of plutonia present in the capsules, there was no observable difference in behavior caused by the two plutonia purity levels. In subsequent tests, care must be taken to produce a fuel volume to cladding volume relationship equivalent to the potential system being studied in order to fully define a possible effect of fuel purity.

Plutonia-238 is more aggressive in contact with refractory metals than is curia-244, based on exposure at 1800°C for times of 10,000 hours or more. Curia transports to a greater extent than plutonia. Total containment of curia is more difficult because of the more rapid generation of helium and the need to either contain or vent the helium. Containment requires stronger alloys. Venting requires means to prevent vent plugging.

Helium release in PMC and SSC fuel forms appear to be equivalent after exposures of 5000 hours at 1500° and 1800°C. Release into sealed systems was about 75% at 1500°C and greater than 90% at 1800°C. An aged PMC specimen released almost 90% of the stored helium in a relatively short term exposure at 1500°C indicating structural damage during long-term aging at room temperature.

PMC and SSC testing at 1500° and 1800°C for 5000 hours indicates that either TZM/Mo-46Re or Mo-46Re/TZM is the best liner/structural member combination tested. In both cases, the structural member remained undamaged by the presence of the plutonia fuel form. All other combinations tested, particularly with T-111 as the structural member, were degraded substantially. It was established that 1500°C is too high a temperature to protect T-111 from oxygen transport from the plutonia and resulted in massive oxide precipitation with the greatest amount oriented in stringers in the direction of rolling.

Combinations of molybdenum alloys and T-111 resulted in formation of inter-metallic compounds at the interface during exposure at 1500°C and above. The extent of these reactions would appear to be detrimental to the behavior of both materials in a heat source. Either a barrier must be developed or these combinations are to be avoided down to 1500°C and probably much lower.

TZM liners resulted in the best fuel form behavior. The only PMC specimens retaining the molybdenum coating over the surface were exposed in TZM liners. The best appearing SSC specimen retaining the greatest amount of molybdenum network was exposed in a TZM liner. The reason is not evident, but the results are.

SSC fuel forms degraded badly at 1800°C. Those contained in W-25Re and W-30Mo-30Re completely lost the original molybdenum network. It appeared that the molybdenum had dissolved in the plutonia-thoria matrix. The metal network had degraded considerably in an 1800°C exposure in TZM but partial retention was observed.

PMC behavior depended on liner material and temperature. Retention of the best structure occurred in TZM with only slightly less adequate behavior in Ta-10W. All other liners resulted in molybdenum surface coating and

plutonia transport to the liner walls. Oxygen content of the plutonia depends on the permeability and gettering characteristics of the container alloys. The highest oxygen content was observed in the W-25Re/W-25Re system which has the lowest permeability for oxygen coupled with low gettering. The molybdenum containing alloys and Ta-10W acted as windows for helium permeation at 1500°C and above allowing movement to the T-111 which acted as a getter for oxygen. Oxygen loss is not bad if the density decrease associated with it can be accommodated. Random changes in porosity of the various specimens did not permit an estimation of the overall effect of exposure on the behavior of the PMC.