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MASTER

PLUTONIA/CURIA COMPATIBILITY TESTING

JULY - SEPTEMBER 1969

QUARTERLY REPORT NO. 4

DR. R. L. ANDELIN AND J. D. WATROUS

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

DATE PUBLISHED—OCTOBER 1969

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Classification cancelled (or changed to) UNCLASSIFIED
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by C. Fitzusky TIE, date 8/5/70

DONALD W. DOUGLAS LABORATORIES
RICHLAND, WASHINGTON

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ABSTRACT

(Unclassified)

The safe use of radioisotope fuels requires knowledge of the capability of materials to contain the fuel. Compatibility testing of isotope-container materials combinations at or above expected use temperatures is currently being performed. Metallography has been completed on refractory metal capsules containing relatively impure $^{238}\text{PuO}_{2.0}$ and $^{238}\text{PuO}_{1.8}$ after exposure at 2000°C for 1000 hours. Most of the metallography has also been completed on refractory metal capsules containing the same $\text{PuO}_{2.0}$ after exposure at 1800°C for 2000 hours.

Fifteen capsules containing a higher purity plutonia have been fabricated. Twelve are being exposed at 1800°C . The remaining three are awaiting receipt of $\text{PuO}_{1.8}$ from Mound Laboratory to provide comparative data.

Plans have been completed and preparations are underway to initiate testing of curium-244 sesquioxide early in the next quarter.

Six plutonia-molybdenum cermets fabricated at Battelle-Columbus Laboratories were encapsulated. One capsule at each of two temperatures, 1800°C and 2000°C , attained 1000 hours exposure and have been removed for examination. The remaining four capsules will continue exposure to 5000 or 10,000 hours.

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Section 1

INTRODUCTION

Principal goals of the DWDL compatibility testing program are to determine the best container materials for both plutonia and curia at high temperature and long times, define kinetics and mechanisms of fuel container reactions, and determine relative reactivities of various plutonia fuel forms. The original plutonia being used in the program contains 0.95% to 1.22% of metallic impurities which, according to information from Mound Laboratory, results in major interactions related to the impurity level and not to a basic plutonia-refractory metal reaction. A higher purity stock of plutonia (~0.23% metallic impurities) has been obtained from Mound Laboratory, encapsulated, and is currently being exposed at 1800°C.

One set of the original plutonia capsules was metallographically examined after exposure at 2000°C for 1000 hours. Results show conclusively that $\text{PuO}_{1.8}$ is less reactive in contact with tungsten, tungsten-25% rhenium, and rhenium than is $\text{PuO}_{2.0}$. Metallographic results of refractory metal- PuO_2 capsules exposed at 1800°C for 2000 hours indicate that W, W-25Re, and Re exhibit very similar behavior to that observed at 2000°C and that Mo is better than either of its two alloys, Mo-50Re and W-30Mo-30Re.

Twelve higher-purity plutonia capsules were fabricated and are currently undergoing exposure at 1800°C. Three higher-purity PuO_2 capsules were also fabricated for exposure at 2000°C but are awaiting delivery of matching $\text{PuO}_{1.8}$ material before starting exposure.

Considerable changes in curia-handling procedures have delayed the start of capsule fabrication. Equipment is being assembled for a start in October.

Six plutonia-molybdenum cermet bodies coated with layers of molybdenum and thoria fabricated at Battelle-Columbus Laboratories were encapsulated at DWDL for exposure at 1800° and 2000°C. Two specimens completed 1000 hour exposure and are awaiting helium-release measurements.

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Section 2

TECHNICAL APPROACH

Compatibility information is needed for both plutonium-238 and curium-244 fuel forms to permit use of these materials for long times at high temperatures. The DWDL program is directed toward testing the oxides of both fuel materials with selected refractory alloys for long times, at higher than potentially useful temperatures. This program is intended to supply basic compatibility information and to supplement information currently being obtained under other Government programs (References 1 and 2). Emphasis is on extremely high test temperatures for long times.

Relatively coarse production-grade $\text{PuO}_{2.0}$ and $\text{PuO}_{1.8}$ powders were selected for initial investigation. Testing with the standard powder PuO_2 was expected to produce the most reactivity. The $\text{PuO}_{1.8}$ is being tested on a comparative basis with the PuO_2 and is expected to be somewhat less reactive. All of the initial plutonia being tested is relatively impure (0.95% to 1.22% metallic impurities) as compared with current feed stock (~0.2% metallic impurities). Test information at Mound Laboratory has shown that the impurities override the effects of the plutonia in reactions with refractory metals at short times.

A new large batch of the higher-purity plutonia being used for testing at Mound Laboratory became available for the DWDL effort during July 1969. Samples currently under exposure or those tests which have been completed with the original plutonia will undergo evaluation, but no new tests will be started. All new tests will use the purer plutonia. The current testing schedule shown in Figure 1 reflects delays encountered in obtaining $\text{PuO}_{1.8}$ from Mound Laboratory.

Curium-244 arrived early in July 1969 in the form of powder CmO_2 . This material will be reduced to the B form of the sesquioxide (Cm_2O_3) prior to encapsulation.

FUEL	ALLOYS	NUMBER OF CAPSULES	TEMP (°C)	TIME (HOURS)	GFY 70												GFY 71							
					J	A	S	O	N	D	J	F	M	A	M	J	J	A	S	O	N	D	J	F
ORIGINAL PLUTONIA																								
PuO _{2.0}	(1)	3	2000	1000	COMPLETED 5/29/69																			
PuO _{1.8}	(1)	3	2000	1000	COMPLETED 5/29/69																			
PuO _{2.0}	(2)	6	1800	2000	COMPLETED 7/8/69																			
PuO _{2.0}	(2)	6	1800	10000																				
PURE PLUTONIA																								
PuO _{2.0}	(1)	3	2000	1000																				
PuO _{1.8}	(1)	3	2000	1000																				
PuO _{2.0}	(2)	6	1800	2000																				
PuO _{2.0}	(2)	6	1800	10000																				
PuO _{x(5)}	(4)	6	2000	5000																				
CURIA																								
Cm ₂ O ₃	(2)	6	2000	1000																				
Cm ₂ O ₃	(3)	3	2000	5000																				
Cm ₂ O ₃	(3)	3	1800	5000																				
BCL CERMET																								
PuO ₂ -Mo	W-25Re	1	2000	1000	COMPLETED 8/26/69																			
PuO ₂ -Mo	W-25Re	1	2000	5000																				
PuO ₂ -Mo	W-25Re	1	2000	10000																				
PuO ₂ -Mo	W-25Re	1	1800	1000	COMPLETED 8/26/69																			
PuO ₂ -Mo	W-25Re	1	1800	5000																				
PuO ₂ -Mo	W-25Re	1	1800	10000																				

NOTES: (1) W, W-25Re, Re
 (2) W, W-25Re, Re, Mo, Mo-50Re, W-30Mo-30Re
 (3) BEST 3 ALLOYS FROM (2) BASED ON 2000°C TESTS
 (4) BEST 2 OR 3 ALLOYS FROM (1)
 (5) COMPOSITION (O/Pu RATIO) AND FORM (MICROSPHERE OR POWDER) BASED ON PRIOR RESULTS

Figure 1. DWDL Current Compatibility Testing Schedule

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Only alloys based on tungsten, rhenium, and molybdenum were selected for investigation based on plutonia compatibility test data from Mound Laboratory, curia data from Oak Ridge National Laboratory, and lanthanide oxide compatibility data from the Donald W. Douglas Laboratories. Molybdenum and its alloys are being tested only at 1800°C because of high vapor pressure and potential vapor-phase transport. An exception is the 1000-hour screening tests with curia at 2000°C in which molybdenum alloys will be included.

A recent addition to the program was investigation of helium release, phase stability, dimensional stability, and compatibility of plutonia-molybdenum cermets fabricated by Battelle-Columbus Laboratories. Individual fuel bodies are being exposed up to 10,000 hours at 1800° and 2000°C prior to examination. Two cermet bodies are being stored at room temperature for possible future use either as comparison specimens or future exposure under, as yet, undefined conditions.

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Section 3

EFFORT DURING CURRENT QUARTER

Metallography was completed on those specimens exposed at 2000°C for 1000 hours and is near completion on those exposed at 1800°C for 2000 hours. Fifteen pure plutonia capsules were fabricated. Six plutonia-molybdenum cermet capsules were assembled.

3.1 Original Plutonia

Comparison of the behavior of W, W-25Re, and Re in contact with $\text{PuO}_{2.0}$ and $\text{PuO}_{1.8}$ after exposure at 2000°C for 1000 hours was completed. Damage to each refractory metal caused by $\text{PuO}_{2.0}$ was greater than that caused by $\text{PuO}_{1.8}$. This is shown in Figures 2 and 3 and summarized in Table 1. Tungsten sustained gross intergranular solution attack and loss of complete grains in contact with PuO_2 , while contact with $\text{PuO}_{1.8}$ resulted in surface pitting limited in depth to approximately 1 mil (Figure 4). There is also one large intergranular crack through the thin disc which could have been caused by thermo-mechanical means such as rough machining coupled with a rapid thermal transient instead of a direct reaction with the plutonia.

Tungsten-25% rhenium had subsurface oxide precipitates and deeper unknown precipitation after contact with $\text{PuO}_{2.0}$ while only showing surface pitting (or precipitation) after contact with $\text{PuO}_{1.8}$. Rhenium showed a definite surface roughness caused by solution and intergranular attack by $\text{PuO}_{2.0}$ but was not attacked by $\text{PuO}_{1.8}$. The Re- PuO_2 capsule showed some very interesting structures (Figure 5). The upper photomicrographs show vapor-transported rhenium on the capsule wall adjacent to the large disc. This transport apparently occurred in the presence of a small thermal gradient, with plutonia as the source of oxygen. The thin disc under the plutonia diffusion bonded to the bottom of the capsule is shown in the lower left photomicrograph. Solution and intergranular attack are shown in the two lower photos. A disc from a companion rhenium capsule exposed under the same conditions is shown in Figure 6.

69-1468

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W (100X)

W-25Re (100X)

69-1470

69-1469

W-25Re (500X)

Re (100X)

Figure 2. Refractory Metal Surfaces After Contact with $\text{PuO}_{2.0}$ at 2000°C for 1000 Hr

69-1471

W (100X)

69-1472

W-25Re (100X)

69-1473

W-25Re (500X)

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Re (100X)

Figure 3. Refractory Metal Surfaces After Contact with $\text{PuO}_{1.8}$ at 2000°C for 1000 Hr

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Table 1

REFRACTORY METAL ATTACK BY PLUTONIA
AFTER EXPOSURE AT 2000°C FOR 1000 HOURS

Refractory Metal	PuO _{2.0} Damage		PuO _{1.8} Damage	
	Type	Depth (mils)	Type	Depth (mils)
Tungsten	Gross intergranular	10	Intergranular Surface pitting	30* 1
Tungsten-25% Rhenium	Subsurface oxide Precipitation	1 10	Surface pitting	1
Rhenium	Solution and intergranular	2	None detected	-

*May be result of thermo-mechanical history rather than plutonia attack.

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Figure 4. Tungsten After Contact with PuO_{1.8} at 2000°C for 1000 Hr (500X)

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(50X)

(200X)

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(1.5X)

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(50X)

(200X)

Figure 5. $\text{PuO}_{2.0}$ -Re Capsule After Exposure at 2000°C for 1000 Hr

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Figure 6. Rhenium Exposed at 2000°C for 1000 Hr Not in Contact with Plutonia (100X)

Figure 7 shows the appearance of $\text{PuO}_{2.0}$ and $\text{PuO}_{1.8}$ after exposure at 2000°C for 1000 hours in contact with tungsten. Limited appearance of a second phase in the $\text{PuO}_{2.0}$ indicates that some loss of oxygen occurred but not to any great extent. The structure of the $\text{PuO}_{1.8}$ is typical of that material. The large number and size of the voids in both fuels are typical of relatively low-density cold-pressed and sintered fuel pellets. To a much lesser extent, some pore formation may result from helium release. Metallic precipitates ranging from 0.05 to 0.5 mil in size are present in the $\text{PuO}_{2.0}$, indicating some solubility of tungsten in plutonia at 2000°C.

Table 2 lists the depths of various forms of attack observed on the refractory metals after exposure at 1800°C for 2000 hr in contact with $\text{PuO}_{2.0}$. Values reported for general solution should be considered as minimum values because the location of the original surface cannot be ascertained accurately. Other values are maximums observed on the metallographically observable plane and may also be somewhat less than an absolute maximum.

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PuO_{2.0}

PuO_{1.8}

Figure 7. Plutonia After Contact with Tungsten at 2000°C for 1000 Hr (50X)

Table 2

ATTACK BY PuO_{2.0} AFTER EXPOSURE
AT 1800°C FOR 2000 HR.

Alloy	Type of Attack (Depth in Mils)			
	General Solution	Intergranular Penetration	Oxide Precipitation	Intermetallic Precipitation
W	2	10	None	None
W-25Re	0.5	None	7	None
Re	5	3	None	None
Mo-50Re	None	8	1	None
Mo	None	6	None	None
W-30Mo-30Re	1.5	2	4	2

Tungsten attack at 1800°C and 2000 hours as shown in Figure 8 was somewhat less extensive than that observed after the 2000°C 1000-hour exposure, probably due to the lower solubility of oxygen in the tungsten. However, there was still extensive intergranular oxide penetration and loss of grains from the surface. Plutonia in contact with the tungsten as shown in Figure 9 exhibits a slightly substoichiometric two-phase structure similar to that shown in Figure 5.

Tungsten-25% rhenium (Figure 10) and rhenium (Figure 11) behaved identically under the two different exposure conditions. Vapor transport of rhenium from the large disc to the capsule wall was again observed with complete bonding in one area (Figure 12). Molybdenum-50% rhenium exhibited both a deep intergranular oxide penetration and a shallower oxide precipitation as shown in Figure 13. Molybdenum appeared unattacked except for one isolated case of a 6-mil intergranular penetration as shown in Figure 14. The tungsten-30% molybdenum-30% rhenium alloy exhibited four forms of attack to varying extents as shown in Figure 15. Some general solution at the surface was coupled with somewhat deeper intergranular penetration, precipitation of an unknown intermetallic phase, and oxide precipitation.

Figure 8. Tungsten After Exposure at 1800°C for 2000 Hr in Contact with $\text{PuO}_{2.0}$

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Figure 9. PuO_{2.0} After Exposure at 1800°C for 2000 Hr in Contact with W (100X)

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Figure 10. W-25Re After Exposure at 1800°C for 2000 Hr in Contact with PuO_{2.0} (500X)

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Figure 11. Re After Exposure at 1800°C for 2000 Hr in Contact with $\text{PuO}_{2.0}$ (200X)

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Figure 12. Re Large Discs (at Left) and Capsule Wall (at Right) Joined Together (100X)

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Figure 13. Mo-50Re After Exposure at 1800°C for 2000 Hr in Contact with $\text{PuO}_{2.0}$ (500X)

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Figure 14. Molybdenum After Exposure at 1800°C for 2000 Hr in Contact with $\text{PuO}_{2.0}$ (200X)

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Figure 15. Tungsten-30% Molybdenum-30% Rhenium After Exposure at 1800°C for 2000 Hr in Contact with $\text{PuO}_{2.0}$ (200X)

Six more original plutonia capsules continue undergoing exposure at 1800°C and will attain 10,000 hours in July 1970.

3.2 Pure Plutonia

Ten grams of a purer form of PuO_2 arrived from Mound Laboratory in August. A portion of this batch, containing approximately 2300 ppm metallic impurities, is also being used in the Mound Laboratory compatibility testing program. This powder appeared to be somewhat more granular than the prior batch resulting in some early difficulty in obtaining sound pellets. Consistently good pellets were made by using a drop of water as a powder and die lubricant during pressing. The moisture was subsequently removed during the sintering step.

Fifteen refractory metal capsules were loaded with 0.25-gram PuO_2 pellets, electron-beam welded with no external contamination, and subsequently further encapsulated in tungsten secondary and tantalum tertiary containers. Twelve

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capsules (two each of W, W-25Re, Re, Mo, Mo-50Re, W-30Mo-30Re) are currently being exposed at 1800°C. The remaining three capsules (W, W-25Re, Re) are being stored until Mound Laboratory can supply equivalent $\text{PuO}_{1.8}$ pellets for simultaneous exposure at 2000°C.

3.3 Curia

Ten grams of curia arrived in July from Oak Ridge National Laboratory. A trip was made to Savannah River Laboratory and Oak Ridge National Laboratory to discuss handling techniques and compatibility test results. Procedures have been formulated and facilities are being readied to prepare and encapsulate six Cm_2O_3 pellets. Two new Lucite gloved boxes with once-through argon flow are being fabricated for use in place of a larger stainless-steel inert-gas gloved box with a recirculating purification system. This change-over will minimize program costs and lessen contamination potential in the hot laboratory. A moisture and oxygen monitoring system will be used to measure gas purity.

3.4 Plutonia-Molybdenum Cermets

Six plutonia-molybdenum cermet pellets coated with a 2-mil layer of molybdenum and a 4-mil layer of thoria fabricated at Battelle-Columbus Laboratories were encapsulated at DWDL in tungsten-25% rhenium containers. Each pellet was approximately 0.3 in. in diameter and 0.38 in. tall and weighed between 4.2 and 4.7 grams.

Each primary container was further encapsulated in a tungsten secondary container and a tantalum-10% tungsten tertiary container.

Two cermets attained the scheduled 1000 hour exposure, one at 1800°C, the other at 2000°C. These two cermets are awaiting shipment to Battelle-Northwest Laboratories for helium-release measurement prior to return to DWDL for further examination.

The remaining four cermets are continuing exposure at 1800° and 2000°C.

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Section 4

EFFORT DURING NEXT QUARTER

Major effort will be directed toward fabricating, exposing, and initial examination of the first curia specimens. Lesser effort will be involved in fabricating three pure $\text{PuO}_{1.8}$ capsules and examining the initial cermet specimens.

4.1 Original Plutonia

Final metallography will be completed on the first two sets of specimens. Exposure will be continued on six specimens scheduled for 10,000 hours at 1800°C .

4.2 Pure Plutonia

Pellets of pure $\text{PuO}_{1.8}$ should be available from Mound Laboratory by the end of October for assembly and exposure starting shortly thereafter with the already-fabricated set of PuO_2 pellets. Early November fabrication will result in completion of 1000 hour exposure at 2000°C before the end of the quarter. In addition, six specimens will complete a 2000 hour exposure at 1800°C .

4.3 Curia

The 10-gram batch of curia will be divided into two 2-gram batches with 6 grams remaining in the original container. One 2-gram batch will be used for experimentation in obtaining the B (monoclinic) form of Cm_2O_3 , experimental pressing into pellets and sintering, and for making the first batch of 6 pellets for encapsulation. Pellets will be encapsulated into one each of W, W-25Re, Re, Mo, Mo-50Re, W-30Mo-30Re containers for exposure at 2000°C for 1000 hours.

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4.4 Plutonia-Molybdenum Cermets

Helium-release measurements will be completed on the two specimens exposed for 1000 hours. Visual, dimensional, and metallographic examination will also be completed.

The remaining four cermets will continue exposure at either 1800° or 2000°C.

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