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MOLTEN PLUTONIUM ALLOYS AS FAST REACTOR FUELS*

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ABSTRACT

Molten plutonium alloys are being investigated by LASL as fuels for fast reactors. LAMPRE-I, a 1 Mw reactor experiment, utilized a Pu-Fe alloy of 16 g Pu/cc and demonstrated the feasibility of using molten alloys as reactor fuel. Current effort is being directed toward developing the less corrosive Pu-Co-Ce alloy system which will be employed in the LASL Molten Plutonium Burnup Experiment (MPBE). Results of current research and development regarding the Pu-Co-Ce fuel system are summarized.

INTRODUCTION

Molten metal alloys for utilization as fuel in fast reactors have several attractive properties, such as: capability of release of the bulk (approaching 100%) of volatile fission products; a large thermal expansion coefficient ($-90 \times 10^{-6} \frac{\Delta V}{V}/^{\circ}\text{C}$); the potential for simplified fuel handling and low fabrication costs; and the elimination of fuel slump as an accident consideration.

LAMPRE-I, a 1 Mwt reactor experiment, employed Pu-Fe fuel of 16 g Pu/cc and demonstrated, in a small system, the feasibility of using a molten alloy as a fast reactor fuel. However, Pu-Fe fuel is not a suitable fuel for large power reactors (~1000 Mwe) because of its high plutonium density (and consequently low allowable specific power).

An alternate fuel alloy, Pu-Co-Ce, will be utilized in the Molten Plutonium Burnup Experiment (MPBE)^{1,2} which is presently being designed. This fuel alloy offers two major advantages over Pu-Fe; (1) the Pu concentration is variable and over the entire range of alloys from 0 g Pu/cc - 13 g Pu/cc the melting point is less than 450°C (see Fig. 1), and (2) the fuel is far less corrosive to the selected containment materials than is Pu-Fe at equivalent test conditions.

Coupled with the advantages of molten alloys listed above, three areas are of concern. The first, and most obvious, is the potential for corrosion of the container and loss of the fuel. Present experience indicates that Ta or Ta - W alloys are the most likely container materials. The development effort which has been expended on this facet has resulted in improvements to the extent that reliable long term fuel containment may be anticipated.

The second problem area is that Pu-Co-Ce fuels exhibit large volumetric expansions during freezing of from 1 - 3% for plutonium concentrations from 0 - 8 g Pu/cc (Fig. 2). This situation poses a potential operational nuisance in that the core may have to be maintained in a molten condition or under controlled conditions if mechanical damage to the container is to be avoided.

The third area is associated with the formation of a Ta-Co intermetallic

Fig. 2. Volumetric Expansion During Freezing of Pu-Co-Ce Alloys as a Function of Pu Concentration.

reaction layer which may be damaged during irradiation and cause undesirable mass transport effects. Preliminary evidence obtained by radiotracer experiments in the absence of irradiation indicate that no major difficulties will be presented by this phenomenon.

The following sections will summarize the major results of current research on the various aspects of the molten alloy development problem and define areas which require in-pile evaluation.

Corrosion Behavior

One of the most important aspects of developing a molten-alloy fuel system is demonstrating the capability of containing the molten metal for extended periods of time. It was established early in the LAMPRE program that the corrosion-failure mechanism involved with liquid plutonium alloys contained in refractory metals was that of intergranular penetration (IGP) of the plutonium through the grain boundaries of the container.³ It was also realized, although somewhat later, that corrosion behavior as measured by length of time before a fuel element showed detectable plutonium on the surface was statistical in nature with a ratio of shortest observed lifetime to longest of <0.1 . Figure 3 shows the fraction failed versus test time for Pu-Fe and Pu-Co-Ce under all test conditions up to 850°C plotted according to the Weibull distribution.

Because the time interval required to obtain meaningful corrosion data at temperatures of interest in reactor applications (500 - 750°C) is in the thousands of hours, test equipment designed to obtain lifetime data at temperatures up to 1150°C has been recently employed. Figure 4 shows mean lifetime data obtained at elevated temperatures in vacuum ($\sim 1 \times 10^{-7}$ torr) for both Pu and Pu-Fe eutectic alloy. It is significant that for the Pu-Fe fuel, the high temperature data and the lifetime data obtained for sodium environment may be represented by the same straight line, which lends support to the validity of extrapolating high temperature data to obtain corrosion behavior information at lower temperatures.

Tests to determine the lifetime of Pu-Co-Ce fuels of interest to the MPBE are in progress. Corrosion tests have encompassed fuel concentrations from 5 - 8 g Pu/cc in contact with Ta and Ta - W alloys (up to 10 w/o W) and at temperatures up to 1100°C. Since there have been few failures

associated with tests carried out at 850°C and below, no definite conclusion can be drawn concerning expected lifetime of this fuel system. However, several generalizations can be drawn from data available:

- a. Pooling all Pu-Co-Ce alloy corrosion tests (all combinations of fuel composition, container material, and test temperature), it is observed that 135 tests have resulted in 27 failures of which 26 have been in welds.
- b. The results of these tests indicate that container walls are not penetrated by Pu-Co-Ce (in dramatic contrast to Pu-Fe fuel) but that only welds are subject to penetration, up to the maximum observation time accomplished to date of 7500 hours at 850°C (Fig. 3).
- c. When welds are removed from consideration, there is an indication that significant corrosion of Ta or Ta - W alloys will not occur at temperatures up to 850°C.
- d. The lifetime prior to failure of Pu-Co-Ce alloys contained in Ta or Ta - W alloys will be significantly better than for Pu-Fe alloy (see Fig. 3).

It has been found that a carburized layer on the internal surface of the container or the diffusion of carbon into the tantalum grain boundaries is exceptionally effective in preventing plutonium penetration with the highly corrosive Pu-Fe fuel. Evidence also indicates the latter treatment to be effective in prevention of Pu penetration of welds by Pu-Co-Ce fuel. For example, when carbon was allowed to diffuse into the tantalum grain boundaries from the external surface of the container in a run with Pu-Fe fuel, the tantalum capsule did not fail after 380 hours at 1100°C, whereas without the carburization treatment the lifetime is less than 48 hours (Fig. 4). Under similar conditions, two tests with 8 g Pu/cc Pu-Co-Ce fuel did not exhibit failure after 700 hours at 1100°C, and one test lasted 1500 hours without failure (Fig. 4). All of these tests were discontinued before failure was observed. It is anticipated that exploitation of the pronounced deterrent effect on Pu penetration of C in the Ta grain boundaries will eliminate IGP in a reactor system.

It should be pointed out that in all the corrosion work mentioned in this paper a "failure" due to IGP is defined as detectable plutonium

Fig. 3. Failure Analysis of Pu-Fe and Pu-Co-Ce Fuels. (Weibull Distribution Plot)

Fig. 4. Mean Lifetime of Pu and Pu Alloys Contained in Unalloyed Ta. Note the Effect of External Carburization of the Ta on Lifetime of PuFe and Pu-Co-Ce Alloys.

(by alpha counting) on the surface of the container following a given test. In general, the failures obtained with Pu-Co-Ce fuels have been on the order of $<10,000$ cpm or $<10^{-7}$ g Pu on the outer surface of the containers. It is obvious that the presence of plutonium on the surface of a capsule in the magnitude described will not constitute a "failure" in the sense of being of operational importance in a reactor. No evidence at this time indicates that a "failure" of $\sim 10^{-7}$ g Pu will ever progress to a magnitude that will allow liquid fuel to flow from the containers into the sodium coolant. In fact, some evidence exists that in certain instances so called "failures" may become non-failures if the test is allowed to continue after initial detection of Pu. Table I shows data obtained on two test capsules that are under current investigation. Further studies are in progress concerning the conditions which govern the size of plutonium leaks.

Mass Transfer

The transition metals, Fe, Co, Ni form intermetallic compounds with refractory metals Nb, Ta and W. This intermetallic compound formation has been observed when both Pu-Fe⁴ and Pu-Co-Ce⁵ fuels are in contact with Ta or Ta alloys. Fig. 5 shows a reaction layer formed between 6.2 g Pu/cc Pu-Co-Ce fuel and Ta. The formation of this layer presents two potential problems with respect to long term behavior at high temperatures:

First, since these compounds; TaCo or Ta₂Co, have lower Ta activities than does pure Ta, it might be possible to establish a "short circuit" pitting corrosion if spallation of the intermetallic should occur. The pitting would be due to dissolution of bare Ta followed by precipitation of Ta as the intermetallic compound at an immediately adjacent sink.

Direct spallation of the TaCo layer has not been observed in any test run to date although in some cases the precipitated crystals (Fig. 4) have been dislodged from the surface. However, in no case has the diffusion layer been separated from the Ta substrate.

No pitting corrosion due to preferential TaCo crystal removal has been observed. However, tests at 1100°C of Pu-Fe fuel (which forms the similar reaction layer TaFe have definitely indicated pitting and subsequent IGP through the unprotected container wall in the pit (see Fig. 6).

Table I
Surface Plutonium Alpha Activity of
Two Possible Capsule Failures*

<u>Test Condition</u>	<u>Test Time (hrs)</u>	<u>Pre-Acid Wash</u>	<u>1st Acid Wash</u>	<u>2nd Acid Wash</u>	<u>3rd Acid Wash</u>
Ta container; 8 g Pu/cc Pu-Co-Ce; 850°C	1500	2000 cpm	250 cpm	---	---
	2500	1200 cpm	200 cpm	100 cpm	---
	3500	None	---	---	---
Ta-10W container; 8 g Pu/cc Pu-Co-Ce; 850°C	2500	200 cpm	None	---	---
	3500	10,000 cpm	1900 cpm	500 cpm	500 cpm
	4500	2000 cpm	1100 cpm	500 cpm	300 cpm

*Failure defined as ≥ 500 cpm after 3 acid washes.

Acid wash used is HF:HNO₃:H₂SO₄ in 2:2:5 volume ratio for 5 seconds.

Fig. 5. TaCo Layer Formed on Ta by 6.2 g Pu/cc Fuel.

FUEL

Ta-Co
INTERMETALLIC
COMPOUND

Ta

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Fig. 6. Pitting Corrosion of Ta in Contact with PuFe Alloy for 16 hours at 1100°C. Pit has Occurred where Ta-Fe Intermetallic Compound Spalled off the Ta Wall.

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Since the TaCo intermetallic compound acts as a precipitation sink for Ta in solution in the fuel, mass transfer of Ta is expected due to the free energy differences in different parts of the capsule because of temperature gradients. Thermal gradient mass transfer of Ta has been demonstrated by tracer techniques. Tables II and III summarize the available data. Mass transfer rates of the magnitudes cited should present little difficulty, providing pitting is not initiated.

The second potential problem posed by the formation of the intermetallic compound is that molten-plutonium alloys all exhibit a "climb" phenomenon on metal walls. This phenomenon is manifest by a $<1-3 \mu$ thick fuel layer climbing on Ta to heights of 5 or more inches above the meniscus. As the fuel climbs, the Co in the alloy forms a TaCo or Ta₂Co intermetallic compound and consequently the Pu content of the fuel becomes enriched, thereby giving the fuel increased tendency for intergranular penetration of the Ta wall.

Figure 7 shows the analysis of the fuel film on a capsule wall after exposure for 500 hours at 700°C. Pu was observed on the external surface of this capsule by alpha counting in the region of sections 11 and 12 even though no visible Pu was observed on the internal surfaces of these sections. HCl leaching of the internal surface gave results as shown. The enrichment of Pu is caused by the depletion of Co via intermetallic formation up to approximately 5.5 in. above the fuel level. Since about a 10°C temperature gradient exists along this length of capsule (top ~10° cooler than the fuel level) further enrichment in Pu is caused by sliding along isotherms of the phase diagram (see Fig. 1) caused by precipitation of β Co at approximately 700°C and 4.5 at/o Co.

Although Pu was found by chemical analysis in the section 10, the mass of Pu involved was extremely small, i.e. 11.625 mg. Consequently, it is not expected that a leak in this area would cause macroscopic amounts of fuel to be lost from the capsule. It is known that the presence of a Ta-Co or other intermetallic compound prevents diffusion of Pu in Ta grain boundaries. Consequently, the method that would prevent IGP is to prevent the fuel film from forming TaCo as it climbs.

Preliminary work indicates that the presence of a Ta_xC layer on the

Table II
Mass Transfer Experimental Conditions

Run No.	Fuel Tested	Type of Active Tab and Capsule	Temperature Conditions
3	5 g Pu/cc, Pu-Co-Ce	Ta capsule; annealed Ta tab at bottom	700° ^a to 610° ^b
4	5 g Pu/cc, Pu-Co-Ce	Ta capsule; highly worked tab at bottom	700° ^a to 610° ^b
5	5 g Pu/cc, Pu-Co-Ce	Carbided Ta capsule; annealed uncarbided Ta tab at bottom	700° ^a to 610° ^b
6	8 g Pu/cc, Pu-Co-Ce	Ta capsule; annealed Ta tab at bottom	750° ^a to 650° ^b
7	8 g Pu/cc, Pu-Co-Ce	Ta capsule; annealed Ta ring in wall at gas/liquid interface	750° ^b to 650° ^a

^aTemperature at bottom of capsule

^bTemperature at fuel/gas interface

Table III
Overall Mass Transfer Rates

Run No.	Average Rate Mils/year	Maximum Rate Mils/year	Time of Run Hours
3	0.006	0.014	3300
4	0.025	0.038	3100
5	0.038	0.104	2000
6	0.185	0.95	720
7	0.55	6.20	384 (in progress)

Run No.	Comments
3	No Ta ₂ Co formed on the capsule walls. Ta ₂ Co precipitated at gas/liquid interface. Large quantities of Ta ₂ Co formed in the zone between the tab and the bottom end cap of the capsule.
4	The run was similar to Run 3 except the mass transfer rates were a factor of 3 to 4 higher.
5	No Ta ₂ Co formed on the carbide layer. The carbide layer along the capsule walls was intact after the run was completed. A thin layer

Table III (Continued)
Comments on Run Results

Run No.	Comments
	(less than a micron in thickness) which was probably tantalum carbide formed on the active Ta tab. However, the Ta did dissolve from the tab and mass transferred to the gas/liquid interface where it precipitated as TaC or Ta ₂ C.
6	Ta ₂ Co crystals precipitated only at the gas/liquid interface. No Ta ₂ Co formed on the walls of the capsule. The overall mass transfer rate appeared to be controlled by the rate of precipitation at the interface. Periodically, showers of crystals would form at the interface, and during these periods the mass transfer from the tab would markedly increase.
7	A very large quantity of Ta dissolved from the ring insert during the first 1-1/2 days. Then, no more transfer was noted. Probably Ta ₂ Co crystals precipitated on the Ta surfaces on and around the ring insert and prevented further mass transfer. The formation of the layer and its structure will be checked by post mortum analysis of the capsule.

Visual Film Color
Observation

Gold Color
Stops in Section 4

Blue Color

Internal Surface
Appears to be Clean, Shiny Tantalum

Figure 7

ANALYSIS OF FILM REMOVED FROM TANTALUM

Sample	Height Above Fuel	Mass of Film Removed	Pu		Ce		Co		Pu/Ce	Pu/Co
			w/o	a/o	w/o	a/o	w/o	a/o		
1	Contains 5/16 in. fuel	5.68 grams	54.5	35.9	37.0	41.7	8.4	22.4	0.86	1.6
6	5-3/16 in.	11.115 mg	62.0	47.5	36.8	48.1	1.4	4.43	0.99	10.7
12	11-3/16 in.	11.625 mg	84.0	73.3	14.9	22.2	1.25	4.36	3.30	16.8

All sections 1 in. in length.

* = Samples analyzed.

Fig. 7. Composition Variation of the Pu-Co-Ce Film that Climbed the Ta Alloy Capsule Wall During Testing at 700°C for 500 Hours. Note Pu Enrichment as Co Concentration Decreases due to Ta-Co Intermetallic Compound Formation.

container surface completely prevents tantalum-cobalt intermetallic layer formation and may be self-healing. The carbide layer is established by bulk diffusion of carbon into the tantalum and is not subject to spallation in thin layers (i.e., $<5 \mu$), although spallation of a portion of the layer (at the TaC - Ta₂C interface) has been observed with thick layers (i.e., 10-15 μ). Run 5 (Table III) indicates that although no Ta₂Co formed as a sink for the active Ta in the carburized container run, the dissolved Ta apparently precipitated as Ta_xC and transferred at a rate approximately 4 times that for the uncarburized system. However, the rate observed, i.e., 0.04 mils/yr, is considered not to be significant from a practical standpoint.

The presence of a carburized surface has been shown to enhance the IGP resistance of Ta as well as to prevent Ta₂Co intermetallic compound formation. Therefore, because of the uncertainty associated with the tantalum mass transport phenomenon and pitting corrosion, a carbide layer has been specified for the reference capsule of the MPBE core, until and unless it can be proven by further experimentation that such a layer is not necessary.

Mechanical Properties of the Fuel Container

The mechanical properties of the container, as well as its resistance to corrosion, are important considerations in selecting a suitable container material. Tungsten additions to tantalum have pronounced effects on hardness, ultimate and yield strengths, and total per cent elongation. Increasing tungsten content of the alloy rapidly increases ultimate and yield strengths and moderately lowers percentage elongation. Figure 8 shows yield strength and ultimate tensile strength data for Ta, Ta - 5W, and Ta - 10W alloys. The high temperature yield strength of Ta is markedly improved by W additions. It is seen that for temperatures of interest for fast reactor applications (500°C - 750°C) the yield strength of Ta - 5W is 5 - 7 times that for Ta. Consequently, since container strength is important for preventing capsule distortion in the event of inadvertent freezing of the fuel, and since W additions to Ta may be beneficial to corrosion performance, the material selected for use in the MPBE is Ta - 5W.

Fig. 8. Elevated Temperature Yield Strength Properties for Ta, Ta - 5 W, and Ta - 10 W Sheet.

Irradiation Studies

The effect of irradiation on the mechanical properties of Ta - W alloys is virtually unknown. Room temperature tensile tests on Ta irradiated to $\sim 9.4 \times 10^{19}$ nvt (fast) indicates an increase in yield strength of greater than 200% and an elongation decrease of $\sim 80\%$.⁹ New data on the effect of irradiation on the room temperature tensile properties of Ta and Ta - 10W have been recently reported.¹⁰ However, information concerning the quality of the test material is inadequate, and the data are of little value in predicting the behavior of Ta - 5W during irradiation.

Body-centered-cubic materials show virtually complete recovery of mechanical properties at ~ 0.35 T/T_m (absolute test temperature/absolute melting temperature).¹¹ Based on irradiation data on the high temperature mechanical properties of W, Mo and Nb, it is expected that Ta - 5W will demonstrate an irradiation induced yield strength increase and a reduction of total elongation since the temperature range of the container in the reactor is between 0.235 - 0.30 T/T_m. The magnitude of these mechanical property changes after fast neutron irradiation is under investigation.

The effect of fission product recoil damage on both Ta(W) - Co reaction layers and Ta(W) - C layers is being studied by irradiation testing in the OWR, a 5 Mw thermal flux research reactor at LASL. Because of the fission density profile obtained in such a test, the Ta wall actually is exposed to a total fission fragment generation density that is 5 - 6 times the average for the fuel element. Consequently, such testing is a way of intensifying the fission recoil into the container surface and obtaining large equivalent surface effects of burnup relative to a fast reactor environment which has a flat fission density across the fuel diameter.

The first of these experiments (OWREX-1) designed to investigate wall effects has been completed. The experimental fuel element consisted of an 8 in. long capsule containing ~ 2.2 in. of 6.2 g Pu/cc Pu-Co-Ce fuel [38.5 a/o (58.0 w/o) Pu, 24.6 a/o (9.0 w/o) Co, bal Ce]. The container was an unalloyed, deep drawn Ta capsule. Fuel heat was removed by a naturally convecting Na coolant system with the bottom of the fuel maintained at $\sim 470^\circ\text{C}$ and the top at $\sim 600^\circ\text{C}$.

The average burnup of this capsule was calculated to be 2.5×10^{19} fissions/cc or 0.15 a/o Pu burnup. Radiochemical burnup analysis has not yet been completed. The fission density at the wall was calculated to be

1.25×10^{20} fission/cc. Figure 9 shows a post-irradiation radiograph of this capsule. Significant features shown in the radiograph are the exceptionally clean meniscus and the presence of fission gas bubbles along the capsule wall (similar to those observed in LAMPRE-I fuel capsules). Metallographic examination showed no wall effects other than those expected from an equivalent thermal treatment out-of-reactor (e.g., Ta_2Co being formed at the meniscus/wall interface).

The second capsule (OWREX-2) identical to OWREX-1, was irradiated to ~1 a/o Pu burnup, or 1.6×10^{20} fissions/cc with a fission density at the container wall equivalent to 6 a/o Pu burnup. A post-irradiation radiograph of this capsule is shown in Fig. 10. The radiograph shows this capsule to be substantially the same as OWREX-1. Since post-irradiation examination of this capsule is still in progress, no conclusions can be drawn regarding wall effects. However, although fuel enrichment is expected in the gas phase portion of the capsule by "fuel climb", the capsule was "swipe clean" with respect to Pu contamination indicating Pu did not exhibit intergranular penetration of the capsule during the test (~2400 hours at temperature).

A further significant point is that these two capsules are loaded with the same amount of fuel and that both are the same height (to within a few mils) after irradiation. This result indicates that fission gas release reached a steady state within the 2 week operational period of OWREX-1 and that volume percent of bubbles within the fuel remained constant after an initial transient condition.

Two experiments are being inserted in the OWR that are designed to determine the irradiation effect on Ta - 5W containers with and without a carbide coating. It is anticipated these capsules will be run to at least 3% average Pu burnup.

Conclusions

Our studies of potential fast reactor core materials may be summarized as follows. Molten Pu-Co-Ce fuel alloys appear to be much less corrosive than the Pu-Fe eutectic alloy used in LAMPRE. Ta - 5W alloy, selected as a core container, has at least a factor of 5 higher yield strength than Ta in the unirradiated condition and it is expected that this increased strength will minimize or eliminate capsule distortion if the fuel is frozen. Although several major questions regarding fuel irradiation performance

Fig. 9. Post-irradiation Radiograph of OWREX-1 Irradiation Capsule.
Fuel Composition 6.2 g Pu/cc; Average Burnup; ~0.15 a/o Pu.

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Fig. 9

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Fig. 10. Post-irradiation Radiograph of Fueled Region of OWREX-2 Irradiation Capsule. Fuel Composition 6.2 g Pu/cc; Average Burnup; 0.9 a/o Pu.

still must be answered: e.g., the effect of high fission products/cc on the fuel's chemical behavior, the rate of fission gas release at high power densities, etc., Pu-Co-Ce alloys offer considerable potential as a practical fast reactor fuel.

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