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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA ○ LOS ALAMOS NEW MEXICO

QUARTERLY STATUS REPORT ON LAMPRE PROGRAM
FOR PERIOD ENDING NOVEMBER 20, 1962

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LOS ALAMOS SCIENTIFIC LABORATORY
OF THE UNIVERSITY OF CALIFORNIA LOS ALAMOS NEW MEXICO

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All LAMS reports are informal documents, usually prepared for a special purpose. This LAMS report has been prepared, as the title indicates, to present the status of the LASL LAMPRE program. It has not been reviewed or verified for accuracy in the interest of prompt distribution. All LAMS reports express the views of the authors as of the time they were written and do not necessarily reflect the opinions of the Los Alamos Scientific Laboratory or the final opinion of the author's on the subject.

LAMPRE I

Operation

The system modifications to reduce reactor cell temperatures to acceptable values appear to have been successful, and routine operation of LAMPRE I (Core II) at 1 MW was initiated. On September 15, while the reactor was at 1 MW power level, a rapid loss of reactivity ($\sim 1\%$ in 20 min) was observed. This was thought to be due to a leak occurring in a fuel capsule and so the reactor was shut down. After removal of the defective capsule for examination, as described below, operation was resumed on November 15 with a temporary limitation on the power level of 500 kW while the cause of the capsule failure was investigated. Meanwhile, additional irradiation of the four Pu-Co-Ce containing capsules in the reactor can be continued. The rate of reactivity loss at 500 kW continues to be about $1\%/h$.

The LAMPRE control elements proved to be satisfactory. Scram time for the four control rods appears to be constant to within 0.01 sec for all operating conditions, and the shim rundown time during a scram is also satisfactory.

A summary of the LAMPRE I operations during the current report period and, in less detail, of the prior history of Core II is given in Table I.

TABLE I
Summary of LAMPRE Operations, Core II

<u>Run No.</u>	<u>Start Date</u>	<u>Stop Date</u>	<u>Power, MW</u>	<u>Accumulated, MW-h</u>
42 through 57	4/23/62	8/27/62	0-1	767
58	8/30/62	9/7/62	0.5-1	946
59	9/13/62	9/15/62*	1	988
60	9/17/62	9/17/62	10 ⁻³	---
61	9/18/62	11/5/62**	0-10 ³	988
62	11/15/62	----	0.5	---

*Run terminated by leak in capsule No. 1257.

**This run was used to locate the defective capsule; after its removal periodic checks on criticality were made.

Examination of Defective Capsule

In order to identify the defective capsule, a high subcritical multiplication was established by adjustment of the control elements and the change produced by raising individual capsules 10 in. was observed. An abnormally small multiplication change occurred when the defective capsule (the 76th) was raised. This capsule (No. 1257) was in the second row out from the core center. When it had been pulled up into the fuel handling machine, it was observed and photographed through a periscope. There was clear evidence of a hole in the wall below the normal level of the molten fuel. Drops of what appeared to be fuel were observed on the exterior surface near the hole and also at the top and at the bottom ends of the capsule.

An adjacent capsule (No. 1273) was also removed from the core. Splattered fuel was observed on that part of its surface which had faced the leaky capsule. A third capsule (No. 1290) was removed from the position diametrically opposite the core center from No. 1257. It had not been subject to splattering during the leak, and will serve as a control in the metallurgical examinations.

The location of the fuel level in an irradiated capsule is characterized by a concentration of La^{140} , probably in conjunction with its Ba parent. In the case of Capsule No. 1257, three distinct areas of La^{140} concentration were observed: (1) at the position corresponding to the normal level of the molten fuel during initial irradiation, August 20-November 20; (2) at a position slightly below that of (1); and (3) at the still lower or final fuel level which was determined by the hole in the capsule wall. It is possible, therefore, that leakage in capsule No. 1257 took place in two stages, the first at such a slow rate that it was unnoticed in comparison with the reactivity loss associated with the normal operation, whereas the second occurred more quickly.

Actual measurement of fuel level after removal of the capsule top indicated that 2.1 in. (~ 70 g) of fuel had leaked into the sodium system. Transverse slices for metallographic inspection were removed from the capsule in the following areas: weld zone, gas phase, defect area, final gas-fuel interface, and fuel phase near the bottom tip.

A hole about 0.004 in. in diameter was found in the apparent defective area. This hole could have been somewhat larger because of the uncertainty that the plane of polish was not on the diameter of the hole. The capsule wall was dished in from the outside, but the inside diameter of the capsule was not altered. Two blobs of fuel were found on either side of the hole and the outside of the capsule was coated with fuel to the edge of the hole. The fuel blobs contained a bright constituent identified as tantalum.

The microstructure of the tantalum near the hole indicated precipitation at the grain boundaries and a lattice oriented precipitate in the grains. About 0.1 in. (longitudinally) away from the hole, the tantalum appeared much cleaner.

A longitudinal section through the new liquid level indicated the fuel was in bubble form for the whole extent of the metallographic sample (about $1/4$ in.). The streak of fuel on the outside appeared to be made up of two layers; a dark layer on the tantalum surface with a lighter

layer over it. The light layer matched the appearance of the fuel inside. The dark layer was also found inside, between the fuel and the tantalum capsule.

The bottom tip area and vapor phase sections showed no evidence of attack of the tantalum by the irradiated fuel. However, reaction layers were found in these two sections and there were important differences between the reaction zones found: (1) The reaction layer at the tip dissolved during etching, while that in the vapor phase did not. (2) The reaction layer in the vapor phase penetrated the capsule intergranularly, completely through the wall thickness and was manifest by a discontinuous bright phase which was evident on the unetched specimen. This phase could not be found after etching. The reaction layer on the inside surface was refractory to the etchant and there was considerable spalling on the fuel side.

The weld specimen showed evidence of severe, localized intergranular penetration through the weld adjacent to the heat-affected zone. Plutonium was clearly evident in the grain boundaries in this area, even during rough grinding on 300 grit paper. A thin film of fuel was found on the outside and a large drop of fuel was noted about 30° removed from the region of penetration. The microstructure of the drop appeared to be that of pro-eutectic plutonium grains surrounded by intergranular Pu-Fe eutectic. Many platelets, resembling tantalum oxide, were found around the periphery of the weld and, to a lesser extent, in the heat-affected zone. There was a conical void in the heat-affected zone, adjacent to the weld, which appeared to extend about two thirds through the wall. This void may have gone completely through the wall at a location other than the plane of polish; however, this cannot be definitely established.

In summary, it appears that the failure was initiated by severe, localized intergranular penetration through the weld. The fuel then leaked out the weld, ran down the side of the capsule and, by dissolution of the capsule wall below the initial liquid level, created a hole in the capsule to permit subsequent leakage of fuel. While the sequence of failure

has been tentatively established, there is not as yet a detailed understanding of the mechanism of failure.

Radiography of the adjacent capsule (No. 1273) showed that the capsule wall had suffered thinning at the level of the fuel surface. This result is subject to confirmation by metallographic examination. While no evidence of external attack was observed in capsule No. 1273, it was nevertheless decided to replace all the remaining capsules which were immediately adjacent to the one which failed before resuming reactor operation. All five of these capsules were seen to have Pu alloy on their exterior surfaces, but a detailed examination has not yet been made.

Examination of Coolant

Samples of Na removed from the coolant loop contained Pu and Fe, apparently in particulate form, but the proportion of Fe was higher than in the Pu-Fe fuel alloy. A sample of Na was taken from the core region for analysis and comparison with samples obtained earlier. A preliminary gamma scan indicates the presence of I^{132} , Te^{132} , and Ce^{137} , but no La^{140} .

The presence of uncontained fuel alloy in the coolant system has not yet caused any trouble. A cursory gamma scan of the coolant loop revealed the concentration of activity tentatively identified as Ce^{136} and Ce^{137} at the highest (and coldest) point in the gas space of the system surge tank. The interior of the fuel handling machine is now contaminated with Pu; this will make future maintenance operations more tedious.

Capsule Fabrication

Efforts are being made to improve the interior and exterior surfaces of finished capsules. Unless materials and procedures can be controlled so as to produce satisfactory surfaces, it may be necessary to machine the impacted piece and to polish as a final step to achieve the desired finish.

Methods by which capsules can be made from tubing are being examined. To secure end closure, spinning of the tube with subsequent welding may offer a solution. If tubing can be used to form capsules, considerable savings in fabrication costs should result. As an added advantage, tubing would provide a better surface than is presently available from the impact extrusion and ironing process if surface finish is indeed important. There is, however, no experimental evidence to indicate that surface finish is a factor in capsule integrity in the reactor.

RESEARCH AND DEVELOPMENT

Core Calculational Program

The objective of the core calculational program is to develop satisfactory methods and cross sections to permit two-dimensional transport theory description of core characteristics. Small, high-density cores of the LAMPRE I type as well as the more dilute and larger cores containing Pu-Co-Ce fuels will be treated.

A preliminary study has been made of the appropriate cross sections, number of space points, etc., for use in the two-dimensional neutron transport code (TDC). LAMPRE I has been used as the system on which test calculations are performed, since experimental results are available for comparison. Tentatively, a seven-group set of cross sections appears to be satisfactory. The six upper energy groups are taken from the 16-group cross section set in use at IASL, and the seventh group values are derived by collapsing (on the basis of flux integral weighted) groups 7 through 16; the flux values are derived from one-dimensional S_4 problems.

Two-dimensional test problems include "shim up" and "shim down" configurations of the LAMPRE system. The critical experiment, known as LCX III, was performed before the system had been filled with Na; the core contained solid Pu-Fe alloy fuel in tantalum capsules. A comparison of the experimental and computed values is given in Table II.

TABLE II
Comparison of Calculated and Observed Critical Masses

<u>Case</u>	<u>Configuration</u>	<u>Calculated, kg</u>	<u>Observed, kg</u>	<u>% Difference</u>
a	LCX III, shim up	25.80	25.03*	3
b	LCX III, shim down	33.40	31.36*	6.5
c	Shim worth, LCX II case b minus case a	7.60	6.33*	20

*These values were obtained by extrapolations of neutron multiplication data. It is thought that the "shim down" M_c value is too small by at least 0.2 kg.

Additional work has been done on a Fortran code (DPC) to convert reactor engineering specifications into data suitable as input for neutronic codes. Mixture subroutines compute the elemental atomic densities from a specification of the material and temperature. The volumetric expansion with temperature for several standard regions may also be obtained. By organizing the order in which regions are treated, DPC can calculate the relative displacements due to thermal expansion of the various component regions. This feature will be useful in estimating clearances required to permit free movement of control elements, etc.

Preparation of Pu-Co-Ce Alloys

The following procedure has been developed for preparing Pu-Co-Ce fuel alloys. Machined ingots of pre-cast Pu, filtered Ce and Co are alloyed by heating at 900°C for 30 min in a CaF_2 -coated Ta crucible in a vacuum furnace. Homogeneity of the alloy is assured by two additional heats and one pour. The final ingot is bottom poured into a CaF_2 -coated graphite (or steel) multiple rod mold heated to 325°C. The rods are given a heat treatment.

Fuel material containing 3 and 5 g Pu/cm³ have been prepared. Chemical analyses demonstrate homogeneity of the material within each rod and between the rods of a batch within the precision of the analyses. Preparation of an 8-g Pu/cm³ lot is in progress. The products are very brittle, but the heat treatment reduces cracking in the as-cast rods. Further, the alloys oxidize readily in moist air and must be stored in extremely dry air. The use of Linde Co. Molecular Sieve Type 5A as a desiccant has proved to be satisfactory.

The compositions of the fuel lots prepared are as follows:

g Pu/cm ³	Weight Percent		
	Pu	Ce	Co
3	33.5	56.3	10.2
5	49.3	40.8	9.9
8	68.0	23.7	8.3

Reaction of Pu-Co-Ce Alloys with Tantalum

Metallographic examination of the Ta-Co reaction zone has been supplemented by electron microprobe analysis, x-ray diffraction, and chemical analysis in an effort to understand the mechanism of film formation. The primary compound formed is tentatively identified as Ta₆Co₇ with a hexagonal or rhombohedral unit cell. Interspersed in the film are crystals of pure Ce metal and CeCo₂.

The rate of film formation on tantalum in contact with Co-Ce binary alloys increases markedly with increasing Co content. In tests run at 800°C for 96 h with capsules containing binary alloys of 5.7 w/o Co, 12.8 w/o Co, and 18.1 w/o Co, respectively, the reaction layer occurred in the vapor phase with the 12.8 w/o Co alloy, and in both vapor and fuel-tantalum interface with the 18.1 w/o Co alloy. No film was observed with the 5.7 w/o Co alloy.

In general it has been found that heavy reaction layers in the gas phase adjacent to the fuel-gas-tantalum interface are accompanied by a thinning of the capsule wall immediately below the fuel level.

Preliminary results indicate that the formation of the Ta_6Co_7 reaction layer can be greatly reduced by a 1-mil TaC coating on the internal surface of the capsules. Investigation is continuing to establish the mechanism of formation of this reaction product and how to prevent, or greatly minimize, its formation.

A re-evaluation has been made of tantalum-fuel mass-transfer experiments conducted during the period 1958-1960. Quantitative analysis of the Ta capsule and Pu-Co-Ce fuel data yielded estimates of tantalum solubilities in the fuel, diffusivities, dissolution rate constants, and reaction rate constants of film formation. Although these results are useful in analyzing this fuel system, they are estimates and in many cases were based on subjective analyses of limited data. In order to establish the validity of these results a series of experiments has been initiated to study tantalum mass-transport in Pu-Co-Ce alloys.

U-Base Fuel Alloys

Scoping tests of the U-Mn-Pu system have been continued. Crucibles of TaC, NbC, and ZrC were exposed to an alloy consisting of 78 a/o U-22 a/o Mn for 210 h at 1100°C. The results are given below.

<u>Carbide Composition</u>	<u>Free Carbon (w/o)</u>	<u>% Theoretical Density</u>	<u>Metallographic Observations</u>
TaC with at. ratio of $\frac{\text{Combined C}}{\text{Ta} + \text{Nb}} = 1.00$ }	< 0.05	94	1.5 mils of inter- granular attack
Nominal NbC	< 0.05	~ 90	10 mils of inter- granular attack
ZrC with at. ratio of $\frac{\text{Combined C}}{\text{Zr}} = 0.92$ }	0.58	98.5	13 mils of inter- granular attack

No solution attack was observed in any of the above tests. Further work will be done with the carbide crucibles because of their greater resistance to thermal shock as compared with other ceramics, especially oxides.

PLUTONIUM ALLOY SYSTEMS

Plutonium-Carbon System

Elucidation of details of the Pu-C phase diagram has proved to be difficult, because oxygen and, possibly, other impurities, alter the phase relationships. Some of the results obtained so far are given below.

Zeta Phase: The composition of the zeta phase is approximately 40 a/o C; this phase probably has a solubility range of less than 1% on both the high and low C sides.

Monocarbide: The solubility limit of PuC on the low C side at 610°C is between 42 and 44 a/o C, as determined by microstructure and volume fraction analysis. The solubility limit of PuC at 555°C, also on the low C side, is between 43 and 44 a/o C. The maximum solubility of C in PuC at 610°C is 48.2 a/o C determined by volume fraction analysis; at 555°C the maximum solubility is 47.7 a/o C.

The hardness of PuC varies over a considerable range, from about 600 to about 1000 DPH; it increases with increasing C content.

Examination of cast alloys shows that the PuC peritectic horizontal (at 1652°C) extends from Pu₂C₃ to at least 46 a/o and perhaps as far as 44 a/o C, but not as far as the 35 a/o C composition indicated in earlier work.

Sesquicarbide: Volume fraction analysis indicates that Pu₂C₃ is not completely stoichiometric but has a solubility range. On the low C side, the solubility limit was found to be 58.5 a/o C at 610°C and 59.4 a/o C at 555°C. Since these values differ from earlier ones, the Pu₂C₃ phase boundary will be rechecked at several higher temperatures.

Plutonium-Carbon-Nitrogen System

Preliminary results indicate that PuC and PuN are completely soluble in the solid state.

Plutonium-Carbon-Oxygen System

Preliminary investigation of the pseudo-binary section PuC-PuO shows that a significant degree of mutual solid solubility exists between these two compounds.

Plutonium-Indium System

The eutectoid decomposition of the δ -phase in Pu-In alloys occurs slowly, and the effect of increasing the indium content well beyond 0.6 a/o is to increase the sluggishness of the reaction. After extended heat treatment at 450°C, the phases present were identified by x-ray examination of alloy filings that had been heat treated in evacuated silica capillary tubes at temperatures between 200° and 300°C. Alloys containing from 0.5 to 5.0 a/o In were studied. From data obtained with a sample containing 1 a/o indium and heat treated successively at 250°, 275°, and 300°C, the equilibrium temperature of the $\delta = \gamma + \zeta$ (Pu₃In) eutectoid reaction has been found to lie between 275° and 300°C.

Plutonium-Nitrogen System

Oxygen contamination has been found to decrease the apparent melting temperature of PuN. The true, congruent melting point of PuN is higher than 2770°C and the decomposition pressure exceeds 375 psi.

Plutonium-Rare Earth Systems

It was mentioned in the preceding Quarterly Report (IAMS-2753, p. 12) that no intermediate phases had been found in the binary systems of Pu with Sm, Eu, Gd, and Tb and that Pu and Eu form immiscible liquids. In addition, Dy, Ho, Er, Tm, Yb, and Lu do not form intermediate phases

with Pu, and Yb forms immiscible liquids with Pu. Eu, Yb, and Gd appear to be the only rare earths that are not dissolved sufficiently in δ -Pu to permit retention of that phase to room temperature.

X-ray data indicate that the maximum terminal solid solubility of all the rare earths in Pu, from Pr to Lu (with the exception of Eu and Yb which form immiscible liquids), is from 2 to 3 a/o, and that of Pu in the rare earths is of the order of 20 a/o, with the solubility limit increasing somewhat with increasing atomic number.

The change in solidus temperature with increasing atomic number of the rare-earth alloying element, at the 5 a/o rare-earth composition, is an interesting feature of these alloys. The addition of Pr to Pu lowers the melting point to a eutectic temperature of 627°C . The solidus temperature then rises to a maximum of about 645°C with Ho and Er, but then decreases to 634°C with Lu. It is also of interest that the solidus changes from a eutectic to a peritectic horizontal at Gd, in which the 4f shell is half filled.

CORE TEST FACILITY

Phase A

Title II Review

A review of the Norman Engineering Co. Title II drawings was conducted from October 1 through October 10, 1962. "IASL Title II Evaluation and Review," containing comments on Norman Engineering drawings was transmitted to the AEC October 29, 1962. IASL comments on the specifications followed November 20, 1962.

A Title II cost estimate was received from Norman Engineering September 18, 1962 that was 13% larger than the previous estimate and 40% larger than the Title I estimate. On December 3, 1962, Norman Engineering will present the AEC with a revised facility cost estimate reflecting modifications generated during the Title II Review. Modifications

to drawings are now held in abeyance, except for drafting and engineering errors, pending approval by IASL and the AEC of a revised facility cost estimate. Upon approval of such estimate, Norman Engineering will proceed to make the necessary modifications and resubmit the drawings and specifications for a compliance review.

Phase B

Safety Analysis Report

Printing of the Safety Analysis Report has been completed and copies were mailed to ALO and DRD on August 30, 1962. There is a distinct possibility that the ACRS will be asked to consider Core Test Facility safety, and final approval for start of construction may not be received before March 1963.

Sodium Systems

Intermediate Sodium Heat Exchangers: General arrangement drawings showing overall dimensions of the two intermediate heat exchangers have been checked and returned to the vendor with a request for minor modification in pipe connections, support details, etc. No detailed fabrication drawings have yet been received.

Heat Dump Systems: The vendor (Struthers Wells Corp.) is continuing to submit drawings of heat dump components for approval. However, design work appears to be progressing slowly and some action may be required if all drawings are to be completed and approved by February 1, 1963 as scheduled.

Piping: Vendor check prints of the Intermediate Heat Exchangers have been received and reviewed. The heat exchanger dimension changes made by Griscom-Russell after a re-evaluation of the thermal design will necessitate minor relocation of some of the sodium loop piping. These drawing modifications are under way and two-line drawings of the radioactive 4-in. and 6-in. loop piping have been initiated.

Piping System Capability: An evaluation has been made of the 15-MW radioactive sodium system to determine pressure drop available for the core using the Seawolf E.M. pump. Values were determined for pressure drop vs flow for 6-in. and 8-in. pipe loops. The 6-in. system pressure drop was calculated from the present design layout, and the 8-in. data were derived from a velocity comparison with the 6-in. system. Two limitations were placed on pump performance; one was the 400 A output limitation of the Superior Electric power supply, the other was an allowable pump winding temperature of 200°C specified by the pump manufacturer, corresponding to a maximum allowable pump current of 890A. It was determined that, with power factor corrected to unity, the pump current limit was restrictive.

5-MW Radioactive Sodium Pump: General Electric Co., supplier of the 5-MW radioactive sodium pump, is considering a modification of their induction pump which incorporates a double helix. Sodium flows through an outer helix, turns around, and leaves the pump through a concentric inner helix. The addition of a magnetic core within the inner helix should improve efficiency. Final design of the pump is being delayed while data are being obtained on the performance of similar pumps in use at the G.E. Flight Propulsion Laboratory.

E.M. Pump Control System: A study of the 3300-gpm E.M. pumps, which are to be used in the 15-MW loops, has been started to determine the requirements for a suitable control system. Two systems have been proposed for the E.M. pumps: one employs a motor generator set and the other requires a variable transformer or Powerstat. The latter system is the most economical; however, before a selection is possible, further studies must be made of the E.M. pump characteristics.

Cold Traps: The new set of specifications for cold traps have been released. While requests for proposals were to be sent to some six companies, it is expected that only Liquid Metals and MSA will submit concrete proposals. The cold trap system has been redesigned to incorporate cooling by gaseous nitrogen rather than NaK, and flow rate has been

reduced from 20 gpm to 5 gpm. Specifications also require the cold trap, economizer, plugging indicator, and all necessary valves and piping to be pre-assembled into a complete package.

Sodium Purity Indication: A small bypass loop will be installed on the 15-MW radioactive sodium system for use in making purity analyses. Equipment for such measurements will be installed in the nonradioactive sodium dump tank room. Techniques under consideration are rhometer, sample distillation, and metallic coupon.

Cover Gas System

A revised layout of penetrations between the radioactive and non-radioactive sodium rooms was submitted to Norman Engineering at Title II Review. This latest layout has a greatly reduced number of individual penetrations. It is felt that this new arrangement will minimize the last-minute changes to be negotiated with the contractor. The revision consists mainly of the substitution of a single 3 x 3 ft hole in the nonradioactive-sodium room floor for some 20 single penetrations. Cover gas lines will be routed through this large penetration and shielding installed after all lines are in place.

The remotely operated sodium vapor barrier valves in the gas lines have been eliminated. These lines will be heated as far as the valves in the nonradioactive-sodium room which serve both as tight shutoffs and sodium vapor barriers. Local shielding of the valves will be necessary because of the possibility of condensation of radioactive sodium vapor.

Core Cover Gas Cleanup System

The requirements for chemical cleanup of the core cover gas are currently being investigated. If hydrogen, nitrogen, and methane are generated at a rate comparable to that of LAMPRE I and are continuously removed, the removal rates will be ~ 5 ppm/h for hydrogen, 6 ppm/h for nitrogen, and 1.2 ppm/h for methane.

The removal of hydrogen and methane is straightforward, using a hot copper oxide bed followed by a molecular sieve bed. Methods of nitrogen removal are being studied to find a procedure which will operate at room temperature.