[redacted]

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION

57A

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES
3 4456 0361341 8

ORNL-2646
C-85 - Reactors-Aircraft Nuclear Propulsion Systems

AEC RESEARCH AND DEVELOPMENT REPORT

DECLASSIFIED
CLASSIFICATION CHANGED TO: UCO 5.7.70
AUTHORITY OF: C. Gedberg 2.11.71

# OAK RIDGE NATIONAL LABORATORY

## STATUS AND PROGRESS REPORT

PROGRAM 4700

NOVEMBER 1958

[redacted]

OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

CENTRAL RESEARCH LIBRARY
DOCUMENT COLLECTION
LIBRARY LOAN COPY
NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to see this document, send in name with document and the library will arrange a loan.

[redacted]

[redacted]

[redacted]

C-85 Reactors - Aircraft
Nuclear Propulsion Systems

ORNL-2646

This document consists of 10 pages.
Copy 57 of 58, Series A.

Contract No. W-7405-eng-26

# OAK RIDGE NATIONAL LABORATORY
# STATUS AND PROGRESS REPORT
# PROGRAM 4700

November 1958

Date Issued: DEC 5 1958

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
Operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

[redacted]

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES
3 4456 0361341 8

[redacted]



OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

TABLE OF CONTENTS


| | Page |
|---|---|
| PROGRAM 4700 - REACTOR DEVELOPMENT | 3 |
| AIRCRAFT NUCLEAR PROPULSION PROJECT | 3 |
| Tower Shielding Reactor II (TSR-II) Development and Fabrication | 3 |
| Shielding Theory and Design | 4 |
| Tower Shielding Facility Experiments | 4 |
| Corrosion Tests of Columbium-Lithium Systems | 5 |
| Oxidation Studies of Columbium | 6 |
| Fabrication Problems | 6 |
| Yttrium Preparation Research | 6 |
| Chemical Studies | 7 |
| Analytical Chemistry Studies | 8 |
| Pump Bearing and Seal Tests | 9 |


Reports in this series issued during the past year:

| | |
|---|---|
| November 1957 | ORNL-2438 |
| December 1957 | ORNL-2459 |
| January 1958 | ORNL-2472 |
| February 1958 | ORNL-2488 |
| March 1958 | ORNL-2507 |
| April 1958 | ORNL-2526 |
| May 1958 | ORNL-2547 |
| June 1958 | ORNL-2564 |
| July 1958 | ORNL-2580 |
| August 1958 | ORNL-2596 |
| September 1958 | ORNL-2624 |
| October 1958 | ORNL-2637 |

[redacted]

[redacted]



OAK RIDGE NATIONAL LABORATORY
STATUS AND PROGRESS REPORT
November 1958

This monthly report covers those AEC activities which fall under program 4700, categories C-84 and C-85, classified SECRET. Some of the topics are reported every month, but the majority are reported bimonthly.

## PROGRAM 4700 - REACTOR DEVELOPMENT

### AIRCRAFT NUCLEAR PROPULSION PROJECT

#### Tower Shielding Reactor II (TSR-II) Development and Fabrication

In the initial critical experiment it was discovered that the clean, water-moderated TSR-II fuel assembly was subcritical. By lowering the upper central fuel elements 1/2 in., sufficient excess reactivity was realized to make flux traverses, to evaluate the reactivity worth of the control grids, to measure the temperature coefficient of reactivity, and to determine the proportionality constant between $\Delta m/m$ and $\Delta k/k$. These measurements are continuing so that an accurate determination of the fuel required for the 5-Mw reactor may be obtained.

The reactivity calculations for the reactor, with adequate allowances for burnup and poison, were originally made using a three-group three-region Oracle code which has yielded correct answers on several critical assemblies. These results were later confirmed by means of calculations with a multigroup multiregion code. In addition, some experiments were performed with BSF reactor elements. Efforts are now being made to determine why the calculations were in error and to develop a method of calculation which furnishes useful estimates of critical mass for this type of reactor.

In one of the experiments about 1% excess reactivity was obtained by replacing water in the 17.5-in.-dia internal reflector with a 12-in.-dia aluminum sphere. With such a scheme the present elements may be useful for low-power experiments with the TSR-II.

Since calculations and an experiment have shown that the 1000-gpm flow rate is sufficient for cooling with transition-region flow, an exploratory attempt to redistribute the flow in the annular region was made with the use of screens. The test with the first set of screens did not reduce the flow in the first eight channels sufficiently, but the second set of screens, which is now being installed, is expected to produce nearly correct results. The screens which were used to disperse the flow from the helical tubes in the central cylinder region adjusted the velocity distribution below the tubes to a value of 2:1. One more adjustment should be sufficient to produce an acceptable distribution.

[redacted]

The pitot assembly for measuring the flow ratio between the fuel plates in the central cylinder was received, and the modifications to the hydraulic flow test stand required so that the fuel elements, the control grids, and the pitot assembly can be mounted to it were nearly completed.

An investigation is under way to determine the temperature distribution in the core region from afterheat, as a function of time, with various amounts of water in the core region. The 5-Mw heat-removal system was tested under a static pressure head and also under flow conditions. In general, the system was acceptable, but some minor corrections must be made. The demineralizer will be checked out later while a representative of the vendor is present.

The additional conduit required for the TSR-II is now being installed and wiring of the reactor cubicle is under way. The sling boxes for terminating the aerial cables are being fabricated. The 3/4-in.-thick hemisphere which will complete the pressure vessel was received and inspected. It is being machined prior to welding to the cylindrical section. The central cylinder assembly and the ionization guide assembly were assembled and are now being welded. The bearings for the coaxial water swivel were received, and final assembly is under way. The first of the shim control grids was fabricated, and the remaining ones will be completed shortly.

## Shielding Theory and Design

Work was begun on a project to apply the "Cornpone" consistent $P_1$ reactor code to the calculation of centerline neutron fluxes in shielding configurations at the Lid Tank Shielding Facility. Preliminary results for thermal-neutron fluxes in pure water and pure oil configurations indicate surprisingly good agreement in both shape and absolute magnitude between the calculated and experimental curves. The behavior of the thermal-neutron flux in the region close to the source assembly was predicted extremely well (within experimental accuracy), and the calculated and experimental curves agree to within $\sim$12% at distances as large as 50 cm from the source assembly. Work is in progress to attempt to incorporate inelastic scattering within the framework of the machine code. It is hoped that the code may eventually be used to determine the distribution of secondary gamma-ray sources in actual laminated shielding configurations.

## Tower Shielding Facility Experiments

The latest group of tests in the second series of experiments performed at the TSF for the General Electric Company was directed toward determining the source of the gamma rays detected in a crew compartment 64 ft from a heavily gamma-ray-shielded reactor. For these tests the TSR-I was encased in the GE No. 1 shield which consists of 5 in. of lead

and 17.5 in. of water. There was also a thick shadow shield between the reactor and the crew compartment (the ORNL compartmentalized detector tank). Since with this shielding the dose in the crew compartment from reactor gamma rays was essentially eliminated, the previously observed dose rate must have resulted from secondary gamma rays produced by neutron capture either in the air or in the crew shielding. In an attempt to identify the source, gamma-ray spectral measurements were made in the crew compartment with a 3- by 3-in. NaI crystal while the thickness of the water side shield was varied. Both plain water and borated water were used. These tests were carried out at an altitude of 177 ft. The data are being analyzed.

Corrosion Tests of Columbium-Lithium Systems

Extensive metallographic examinations were completed on columbium specimens to which oxygen or nitrogen had been added by treating arc-cast annealed sheet at high temperatures in either oxygen or nitrogen at very low partial pressures and which had been exposed to static lithium for 500 hr at 1500°F. The results of the metallographic examinations prove fairly conclusively that the oxygen contaminant is responsible for the grain-boundary corrosion which has been detected in practically every weld and heat-affected zone of a columbium system following testing in lithium at ORNL. The proposed mechanism of the corrosion is the reduction of columbium oxide in the grain boundaries by lithium and the formation of lithium oxide plus columbium metal. Such a reduction would lead to a volume expansion of $\sim$47% and would account for the many grain-boundary cracks which have been observed on the surfaces of unstressed columbium weld specimens following exposure to lithium.

Static tests were conducted on various types of columbium tubing in lithium at 1500°F and at 1800°F for 100 hr. All the tubing specimens were tested in a common columbium container at the two test temperatures. The cladding material was removed from the clad tubes prior to the test and both the inner and the outer surface of each specimen were exposed to lithium. The attack on any one piece of tubing was quite erratic, with the inner surface being attacked in some cases but not the outer surface, and in other cases the reverse was found. The heaviest and most consistent attack occurred on the tubing of highest oxygen analysis, and no attack was found on the specimen of lowest oxygen content. These results indicate that contamination of the surfaces during certain of the processing stages led to oxygen contamination of the surface areas and that subsequent exposure to lithium led to attack in the contaminated regions.

Examination of the latest shipment of clad columbium tubing (50 ft of type 446 stainless steel cladding, 0.87 in. OD with a 0.060-in. wall, on 0.75-in.-OD, 0.035-in.-wall columbium tubing) revealed extensive cracks through the entire wall of the columbium tubing. In some areas, a 0.5-mil

columbium oxide film could be seen on the inner surface of the columbium near the cracked areas. The fabrication procedure for tubing will be reviewed critically from the standpoint of the oxygen pressure used in the annealing furnace and the number of annealing cycles.

## Oxidation Studies of Columbium

Studies of the rate of reaction between columbium and very low-pressure oxygen were continued. Five runs were completed at 1000°C and an oxygen pressure of $5 \times 10^{-4}$ mm Hg. A parabolic type of oxidation curve describes the reaction rate under these conditions. An average weight increase of 1.5 mg/cm$^2$ was found after 200 min, and a tenacious black oxide had formed which is assumed to be columbium oxide.

## Fabrication Problems

A quantity of 85% Zr-15% Cb wire developed for columbium welding at ORNL was made at the request of Pratt & Whitney Aircraft. The alloy was prepared by arc-melting arc-cast columbium and iodide zirconium in an atmosphere of purified argon. The finger castings were cold swaged to 0.25 in. in diameter and annealed under vacuum. The wire was then swaged to 0.035 in. in diameter and cleaned.

The finished wire was analyzed for contaminants and found to contain 0.026% oxygen and 0.0069% nitrogen. Half of the wire was shipped to Pratt & Whitney, and the remaining portion was retained for experimental use at ORNL.

Development work on poisoned beryllium oxide for GE-ANPD was completed with the delivery of six 3- x 3- x 1-in. hot-pressed $ZrB_2$-BeO blocks (5.22 wt % $ZrB_2$) of 3.01 g/cm$^3$ average density, and four cold-pressed and sintered bars 1 1/4 x 3/4 x 4 in. of the composition 94% BeO-5% MgO-1% $B_4C$ and 2.7 g/cm$^3$ density.

## Yttrium Preparation Research

The problem of separating LiF slag and Y-Mg alloy in the reduction process was successfully solved by pouring both reactor products together into a titanium mold after completion of the reaction cycle. The alloy and slag separated cleanly into discrete layers and were easily removed from the mold. An attempt to increase the yield, while at the same time decreasing reaction time, to minimize contamination from the crucible was not successful. Helium gas was introduced through a titanium tube and used to agitate the molten reactor mixture. The yield was increased somewhat, but the alloy and slag adhered very tightly to the titanium mold, making it necessary to tear the mold away.

The Y-Mg alloy batches produced in runs OR-3, OR-4, OR-5, and OR-6 were vacuum heat treated to remove the magnesium with various degrees of success. The alloy was handled in large pieces to minimize surface oxidation effects, but this caused difficulties with spattering when the temperature was increased too rapidly and incomplete removal of the magnesium because of insufficient heating time. The spattering was eliminated in treatment of the OR-6 batch by keeping the temperature below 900 C, but even after 48 hr the removal of the magnesium was not complete. However, it does appear that vacuum distillation of large pieces can be made to work by proper control of the heating cycle.

Portions of yttrium sponge from batches OR-3, OR-4, and OR-5 were melted in an inert-arc button furnace. The following vacuum-fusion oxygen analyses were obtained: one melt from OR-3, 1100 ppm oxygen; two melts from OR-4, 920 ppm and 1100 ppm; and one melt from OR-5, 880 ppm. Analyses by the Leco oxygen analyzer agreed quite well with the above, with the difference ranging from 5 ppm to 180 ppm. The fluoride content of the samples went from a low of 135 ppm to 2080 ppm.

The major portion of the sponge from batch OR-4 was pressed into electrodes for consumable-arc melting. The first melt was successfully made under a reduced argon pressure. The resultant ingot was machined to remove surface defects and quartered lengthwise. These sections were welded together into an electrode for a second melt under vacuum. This melt was not successful. A hot spot that developed in the upper portion of the electrode caused it to melt through and drop into the molten-arc pool. Analyses of samples of the ingot after the first melt indicated an oxygen content of $\sim$900 ppm, but this result is suspect because of the high magnesium content.

## Chemical Studies

Thermal-gradient quenching experiments have shown that the previously reported compound in the system $LiF-YF_3$ has the formula $LiF \cdot YF_3$. This compound is a member of the hexagonal crystal system, and it melts incongruently to $YF_3$ and liquid at 815 $\pm$ 5°C.

It was established that reaction of $Y_2O_3$ with an excess of molten $NH_4F \cdot HF$ and removal of the excess bifluoride by volatilization yields anhydrous $(NH_4)_3YF_6$ and that heating of the $(NH_4)_3YF_6$ to 600°C produces $YF_3$ whose $Y_2O_3$ concentration is too low to detect with the petrographic microscope. This process is being used to supply high-grade $YF_3$ for phase studies and other research work, and it may be of value in supplying $YF_3$ for reduction to metallic yttrium.

The $LiF-MgF_2-YF_3$ (58.3-18.6-23.1 mole %) mixtures that have been prepared on a 50-lb scale with the individual components used as starting material have shown 400 to 700 ppm oxygen, in contrast to oxide contents

of less than 200 ppm in similar 5-lb preparations. It is anticipated, however, that longer treatment with HF vapor will lower the oxygen content of the 50-lb batches to less than 200 ppm oxygen.

Preliminary experiments on a 5-lb scale have shown that $LiF-MgF_2-YF_3$ mixtures containing less than 200 ppm oxygen can be obtained if LiF, $MgF_2$, and $Y_2O_3$ are used as starting materials. The separate step in which the $Y_2O_3$ is converted to $YF_3$ by direct hydrofluorination is eliminated in this process. While the use of $Y_2O_3$ rather than $YF_3$ as a starting material has been found to be feasible, container corrosion problems may limit the usefulness of this process.

In studies of the purification of lithium, preliminary experiments indicated that $Li_2O$ is extracted from molten lithium by a mixture of LiCl and LiF. To test this extraction further, a small forced-circulation loop was designed and is being assembled. The loop is made of stainless steel and consists of a closed circuit that includes a lithium receiver, an electromagnetic pump, two lithium-salt extractors, and a lithium-metal sampler. The lithium metal will be circulated continuously until the alkalinity of the salt in the extractors reaches a constant value. Salt samples are to be obtained by means of samplers which will contain liquid bismuth and which will provide contact between the bismuth and salt phases as the salt samples are withdrawn from the extractors.

A study of procedures for preparing pure BeO was initiated as part of a program for developing improved beryllia ceramics for fuel elements. In one series of trials beryllium oxalate of high purity, sufficient to yield 300 g of BeO upon calcination, was produced by the reaction of commercial BeO with molten oxalic acid hydrate, followed by recrystallization. According to reports from GE-ANPD, beryllium oxalate has been found to yield BeO of good sintering characteristics.

## Analytical Chemistry Studies

Two methods for the determination of oxygen in yttrium were evaluated: vacuum fusion and Leco conductometric analyzer. Statistical analysis of the available data indicates that no bias exists between the methods and that the over-all agreement is of the order of 10%. The precision of the Leco method, as obtained by analysis of standard iron samples, is 5% and that of the vacuum fusion method on similar standards is also 5%.

The presence of about 3% magnesium in yttrium was found to produce drastically low results in the analysis of yttrium for oxygen. Magnesium volatilizes readily at 1800°C, the combustion temperature, and apparently acts as an efficient getter for the carbon monoxide produced in the combustion. It will be necessary to analyze such samples, as well as the yttrium-magnesium alloy (80-20 wt %), by the mixed fluoride ($KBrF_4$) technique.

Pump Bearing and Seal Tests

The oil-lubricated pump rotary element that was operating in a gamma-ray field was removed from the MTR canal and disassembled. Oil was found in the can surrounding the test piece. The mechanical face seal, which received a gamma irradiation of approximately $10^{10}$ r, showed an even wear pattern; no buildup of radiation-damaged lubricant was evident. The hardnesses of the Buna N O-rings removed from various parts of the pump ranged from brittle to normal. A pressure check on the coolant-oil system indicated leakage through the hardened O-ring seals, and thus some of the oil accumulation in the outer can may be attributed to O-ring leakage.

Edited by A. W. Savolainen

[redacted]



C-85 Reactors - Aircraft
Nuclear Propuslion Systems

ORNL-2646

## DISTRIBUTION

### Oak Ridge Operations Office

1-10. Research and Development Division (Attn: H. M. Roth)
11. Technical Information Service Extension

### External

12. Atomic Energy Commission, Washington
13. Lockland Aircraft Reactor Operation Office
14. Hartford Area Office
15. Wright Air Development Center (Attn: Materials Laboratory, SCORTO)
16. Wright Air Development Center (Attn: Power Plant Laboratory, WCLPW)
17. General Electric Company (ANPD)
18. Pratt & Whitney Aircraft Division (CANEL)
19. ANP Project Office, Convair, Fort Worth
20. AFPR, Lockheed, Marietta
21. Los Alamos Scientific Laboratory
22. University of California Radiation Laboratory, Livermore

### Internal

23. D. S. Billington
24. E. P. Blizard
25. A. L. Boch
26. G. E. Boyd
27. C. E. Center
28. R. A. Charpie
29. C. E. Clifford
30. A. P. Fraas
31. J. H. Frye
32. B. L. Greenstreet
33. W. R. Grimes
34. E. Guth
35. T. Hikido
36. C. S. Harrill
37. H. W. Hoffman
38. W. H. Jordan
39. G. W. Keilholtz
40. F. C. Maienschein
41. W. D. Manly
42. H. J. Metz
43. E. J. Murphy
44. G. J. Nessle
45. H. W. Savage
46. A. W. Savolainen
47. W. H Sullivan
48. J. A. Swartout
49. E. H. Taylor
50. A. M. Weinberg
51. J. C. White
52. C. E. Winters
53. W. Zobel
54-55. Laboratory Records
56-57. Central Research Library
58. ORNL-Y-12 Technical Library
Document Reference Section

[redacted]