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AEC RESEARCH AND DEVELOPMENT REPORT

OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

PROGRAM 4700

SEPTEMBER 1958

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OAK RIDGE NATIONAL LABORATORY
STATUS AND PROGRESS REPORT
PROGRAM 4700

September 1958

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OAK RIDGE NATIONAL LABORATORY

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

September 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 Facility (TSF) Experiments

In the supplementary 2π measurements being performed at the TSF at the request of GE-ANPD, fast-neutron and gamma-ray dose rates and thermal-neutron fluxes were measured in the GE crew compartment as a function of altitude. The crew compartment rear shadow shield contained borated water at all times, while the other tanks were either empty or contained plain or borated water. One series of measurements was obtained in air at an altitude of 8 ft as a function of the horizontal reactor orientation angle. Measurements were also made around the reactor shield at an altitude of 150 ft in a vertical plane perpendicular to the reactor shield horizontal centerline.

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

A temporary assembly of the annular fuel elements was completed, and six of the control mechanisms were assembled. The first critical experiment will be performed with the elements as soon as the control mechanism support assembly is completed.

The second model of the electrohydraulic transducer which will operate the control mechanism is now being fabricated and should be ready for testing the first week in October.

Preparations are being made for an experiment to investigate the friction factor versus Reynolds number between the parallel plates of the TSR-II annular fuel elements with the proper entrance and exit flow regions. The experiment will demonstrate whether or not the 1000-gpm flow rate is adequate.

[redacted]

Design modifications of the hydraulic flow test unit which will be used to study the water flow in the central cylinder region were nearly completed. Vendors are being contacted to supply pitot tubes for measuring the flow rates between the plates of the central fuel elements.

The cubicle for the reactor controls was delivered. It will be completely wired and then moved into position in the underground building at the TSF when the TSR-I control console is removed.

A method for fabricating the control and safety plates was developed. Cadmium wire 1/8 in. in diameter is placed in Inconel tubes and the entire assembly is rolled to a 70-mil total thickness. The ribbon thus formed is rolled to a given radius, sections are cut to the proper length, and the studs are welded. The short sections are then tack welded to a frame to form a grid.

The boral segments for the lead-boral shield were delivered and were found to meet the specifications. The 1/4-in.-thick aluminum hemispheres were therefore reordered, this time from the company (Superior Products Co.) that furnished the 1/8-in.-thick aluminum hemispheres for the lead-boral shield.

Final assembly of the reactor tank is being made except for the hemispherical bottom section, which has not yet been received. Assembly welding of the central cylinder is under way.

Corrosion Tests of Molybdenum-Lithium Systems

A series of six seesaw-furnace corrosion tests of molybdenum in contact with lithium was completed. The results indicate that molybdenum has excellent resistance to attack and mass transfer at hot-zone temperatures as high as 1900°F. Anomalous results were obtained in one test at 1700°F, however; significant weight changes occurred in specimens held in both the hot and cold zones of the test capsule. The reasons for these weight changes have not yet been ascertained.

The molybdenum test capsules containing the lithium were encapsulated in Inconel with sodium serving as a heat transfer bond. Dissimilar-metal mass transfer of Inconel to molybdenum was observed in these tests. No failures of the molybdenum capsules, either in the base material or in weld zones, occurred in the six tests.

Lithium Purification Studies

Studies of the various methods of purifying lithium with respect to oxygen and nitrogen were continued. Hardness increases and vacuum-fusion analyses indicate that yttrium getters both oxygen and nitrogen from lithium.

Oxidation Rate Studies of Columbium

An apparatus for studying the oxidation rate of columbium at various pressures and temperatures was developed. The range of pressures which can be studied with this apparatus is 1×10^{-5} to 5×10^{-3} mm Hg and perhaps higher. The temperatures may range from room temperature to 1500°C. Preliminary data obtained with this apparatus at 850°C indicate that the oxidation rate of columbium is greatly affected by pressure. The weight gain in 150 min varied from 1.40 mg/cm^2 at 3×10^{-3} mm Hg to 0.06 mg/cm^2 at 5×10^{-5} mm Hg.

The data show that the oxidation rate of columbium at 850°C not only decreases rapidly with the pressure but it also undergoes a transition from a parabolic to a linear rate function at about 2×10^{-4} mm Hg. Data obtained at 1000°C indicate that the oxidation rate increases with temperature for a given pressure.

Columbium Tubing Fabrication and Evaluation

Composite tubing from four columbium extrusions was received in a shipment that consisted of about 50 ft of 0.870-in.-OD, 0.095-in.-wall, type 446 stainless steel-clad columbium tubing. The type 446 stainless steel cladding has a thickness of 0.060 in., and the columbium has a thickness of 0.035 in. Nondestructive tests are to be made of this material.

Approximately 59 ft of 0.620-in.-OD, 0.090-in.-wall duplex tubing from a previous shipment is being evaluated. This tubing consists of a 0.030-in.-wall columbium tube clad with a 0.060-in.-wall type 446 stainless steel tube. The chief inspection methods used are pulse-echo and resonance-ultrasound. The use of the resonance-ultrasonic technique for the evaluation of bond quality has revealed almost 100% lack of bond between the layers, and the pulse-echo technique has detected several discontinuities. Therefore metallographic studies are in process to determine the nature of the discontinuities. Since there is a separation between the layers, it is highly improbable that any inspection was achieved of the inner (columbium) layer with the pulse-echo technique. Therefore an x-ray examination was made to provide more complete information concerning the quality of the tubing. Many discontinuities were noted, some of which seemed to be laps or folds in the columbium. Metallographic sections of typical defective areas were also made for examination.

Creep and Tensile Tests of Columbium Between 1800 and 2500°F

An apparatus was developed for creep and tensile tests of columbium in a controlled gaseous environment in the temperature range of 1800 to 2500°F that utilizes a platinum-wound resistance furnace. Tests proved the apparatus to be satisfactory except for difficulties encountered in

making accurate strain measurements. This problem is under consideration at the present time.

The columbium being used in these tests was extruded at 1200°C from a 3-in.-dia ingot to a 1-in. rod. The rod was then swaged to 5/8 in. to give a total reduction of about 80%. Samples of the material are being annealed at various temperatures between 1100 and 1300°C in order to determine the recrystallization temperature and to investigate the rate of grain growth. This information will be utilized to determine a pretest anneal that will yield a reasonably stable fine-grained structure.

Refractory-Metal-Base Brazing Alloy Studies

A number of binary and ternary refractory-metal-base alloys have been arc-melted and evaluated for joining columbium and molybdenum for service in high-temperature lithium. Tests of columbium and molybdenum T-joint specimens brazed with a 95% Zr-5% Be alloy were promising. This brazing alloy exhibited very good flow and wetting on zirconium and titanium, as well as on columbium and molybdenum. Of the ternary alloys tested, the 60% Zr-25% V-15% Cb, 65% Zr-25% V-10% Cb, and 63% Ti-27% Fe-10% Mo alloys appear to be the best as far as wettability and flowability are concerned. The alloy containing titanium has an added advantage of a low flow point of 1180°C.

Yttrium Zone-Refining and Hydriding Studies

Another attempt was made to zone-refine yttrium. The specimen from the first attempt was machined in the region where the failure occurred and used in this run. The first two passes were successfully made with a zone travel rate of 10 mm/min. On the last pass, the zone travel rate was reduced to ~2.5 mm/min. During this pass the zone temperature became too high and the liquid zone collapsed against the quartz tube; the quartz broke and exposed the metal to air at an elevated temperature. The specimen was about 7 in. long and 1/2 in. in diameter. The top end was threaded and the bottom end was tapered. The break occurred about 2 in. above the taper.

The specimen was cut into 20 samples, beginning about 1 in. below the threaded section and ending 1 in. above the taper. These samples were submitted for metallographic and chemical analyses. The chemical analysis was made in the hope of finding some specific impurity distribution which might have been set up by the zone-refining operation, but no definite conclusions could be drawn.

The hydriding furnace was put into operation, and preliminary experiments are being set up to study the properties of yttrium hydride. The first experiment is designed to give a rough estimate of the effects of various impurities in the base metal on the hydride and the hydriding characteristics.

Yttrium Preparation Research

Operation of the yttrium preparation facility was initiated. In the first run, an attempt was made to prepare yttrium metal directly from a binary mixture of YF_3 and LiF. Five pounds of lithium and 50 lb of the fluoride mixture were successfully transferred into the furnace, with only minor difficulties. Vaporization of the lithium proved troublesome at temperatures over 800°C. However, this difficulty, by itself, is not expected to seriously hamper future experiments. In this first run both thermocouples became ineffective at ~1075°C, either because of condensation of lithium, which caused shorting of the thermocouple wires, or, more probably, because of thermocouple well failure. Heating was continued with the temperature estimates being made with an optical pyrometer. The lithium fumes made these readings unreliable, however. Therefore an attempt was made to pour off the slag and excess lithium before continuing to heat the metal to above the melting point. The slag pour was not successful, and at this time metal or slag deposits were observed on the floor of the furnace. The operation was then halted and the furnace and its contents were allowed to cool. When the furnace was opened, it was discovered that the tantalum crucible had failed and allowed the reactant product mixture to leak through to the graphite susceptor and fire-brick lining. Preparations were begun immediately for the next run which will involve the preparation of the yttrium-magnesium alloy. This reduction will be made at around 1050°C, instead of 1650°C, and therefore many of the problems encountered in the first run should be eliminated or minimized.

Chemical Studies Related to the Production of Yttrium

Additional experiments were run to determine the feasibility of purifying lithium metal by extraction of the impurities with an LiF-LiCl eutectic mixture. A batch of lithium metal of uncertain purity was contaminated with an additional 9180 ppm of oxide by adding CuO. Titration of the salt phase after extraction (1.12 milliequivalents/g) indicated removal of 11,400 ppm of oxide from the lithium metal. A subsequent extraction of the same batch of lithium removed an additional 544 ppm of oxide. It is possible that this remnant was picked up during the mechanical handling involved in the second extraction. This experiment appears to indicate that the partition coefficient favors the concentration of oxide in the salt phase in preference to the metal. The difficulties in analyzing lithium metal for oxygen preclude reliance on direct chemical analyses for oxide concentrations in the metal phase.

A salt mixture suitable for the production of yttrium that does not include magnesium was found by phase equilibrium studies of the system LiF-YF_3. A single eutectic exists in the system at 19.5 mole % YF_3, mp 682°C. The solid phases occurring at this invariant point are LiF, a LiF-YF_3 compound of as yet undetermined composition, and a liquid. The

melting point of the eutectic is low enough to permit convenient processing during the removal of oxide from the salt.

Two 50 lb batches of the $LiF-YF_3$ mixture (81.0 mole %-19.0 mole %) were processed for use in the reduction of low-oxide yttrium metal directly from the salt by lithium metal. The reduction was unsuccessful, as mentioned above. The ternary mixture $LiF-MgF_2-YF_3$, which has been used successfully on a small scale, will therefore be utilized in future reductions, or at least until a greater familiarity with the induction furnace is gained. Five 50-lb batches of the ternary mixture have been processed and are available for the reduction. The feasibility of eliminating the direct hydrofluorination of solid Y_2O_3 by converting the oxide to fluoride in a melt of $LiF-MgF_2$ is being investigated.

Bearing and Seal Tests

The centrifugal pump rotary assembly that is being operated under gamma irradiation in the MTR canal continued to function satisfactorily. The region of the oil-lubricated lower seal and journal bearing had accumulated on September 18, 1958, a gamma-ray dosage of 9.2×10^9 roentgens during 4307 hr of operation. The corresponding estimated accumulated dosage to the bulk oil is 2.24×10^8 roentgens.

A centrifugal pump with a bellows-mounted shaft seal has operated for more than 8300 hr with NaK at 1200°F, a shaft speed of 2500 rpm, and a flow rate of 1200 gpm. Operation continues to be satisfactory.

Edited by A. W. Savolainen

Approved by Alvin M. Weinberg, Director

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