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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1965

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1965

DATE ISSUED

April 6, 1965

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1965

This Status and Progress Report summarizes the unclassified portion of the Laboratory's work. Some of the topics are included every month, but the majority are reported on a bimonthly schedule.

REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

Fuel-Cycle Costs of a Heterogeneous High-Temperature Gas-Cooled Reactor with Unclad Fuel. — We made a fuel-cycle analysis for a heterogeneous graphite-moderated reactor with unclad fuel in order to determine whether such a high-temperature gas-cooled reactor operated with partially enriched uranium would have fuel-cycle costs comparable with those anticipated for similar reactors operating on a thorium-based fuel cycle. The partially enriched uranium, in the form of 3/8-in.-diam UO_2 pellets, is placed in 19 holes in a triangular array at the center of hexagonal moderator blocks. Coolant holes surround each of the fuel holes in a hexagonal pattern. The range of parameters examined included moderator block dimensions (across the flats) from 9 to 15 in. and uranium enrichment from natural to 5 wt % ^{235}U. Fuel cost calculational methods followed those of the advanced converter study (ORNL-3686). With combined reprocessing and fabrication costs for the fuel fixed at $200 per kg of U + Pu, a minimum fuel cost of 1.3 mills/kwhr was obtained with an enrichment of 3 to 4 wt %. With fabrication and reprocessing unit costs allowed to vary as a function of throughput, according to the schedules given in ORNL-3686, a minimum fuel-cycle cost of 1.0 mill/kwhr was predicted. It thus appears that the heterogeneous high-temperature gas-cooled reactor fueled with low-enrichment uranium would have fuel costs comparable with those predicted for similar reactors operating on a ^{235}U-^{232}Th-^{233}U fuel cycle. Capital cost estimates were not included in the study.

Effects of Deposition Conditions and Heat Treatment on the Properties of Pyrolytic Carbon. — We recently completed a study of the effects of deposition conditions and heat treatment on the properties of pyrolytic carbon deposited from methane onto static substrates. Deposits up to 1 mm thick were formed on resistance-heated graphite coupons over the temperature range 1400 to 2400°C and at deposition rates ranging from 3.2 to 2810 μ/hr. Material deposited at 2200°C was heat treated at 2620°C for periods up to 6 hr. We characterized the deposits by microstructure studies, x-ray diffraction analysis (including preferred orientation), and measurements of density and diamagnetic susceptibility.

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The average density of the columnar deposits increased with deposition temperature from 1.23 g/cm^3 at 1400°C to 2.09 g/cm^3 at 2400°C. The interlayer spacing decreased from 3.423 to 3.370 A over this temperature range, while the crystallite size increased from 100 to 245 A. Values for the specimen that had been heat treated for 6 hr were 3.369 and 220 A respectively.

The anisotropy factors found by x-ray diffraction increased from 2.0 to 14.6 over the deposition-temperature range 1600 to 2400°C, and the specimen that had been heat treated for 160 min gave a value of 24.3. Anisotropy factors determined directly from diamagnetic susceptibility measurements showed a similar trend but were lower by 4 to 10%.

Development of Scaled-Up Fluidized-Bed Coating Apparatus. — We designed and built a fluidized-bed coating apparatus of larger capacity than used in previous studies and placed it in service. This apparatus is of particular interest for determining the extent to which results of the type presented previously (ORNL-3762) can be scaled up in terms of controllable variables such as deposition temperature and specific methane flow rate. The apparatus includes a 2-in.-diam graphite-resistance-heated coating chamber with a 36° cone angle and a water-cooled gas injector. Temperatures in the chamber have been calibrated over the range 1400 to 2000°C.

We selected a fluidizing gas flow rate of 10 liters/min for a 100-g charge of 200-μ-diam spheroidal uranium carbide particles. This flow rate is greater by a factor of 4 than that used for fluidization of 25-g charges of similar particles in the 1-in.-diam coater and is consistent with the fourfold increase in charge size and coating-chamber cross section.

Two coating experiments were completed. The first involved precoating 900 g of 275-μ-diam sol-gel ThO_2 particles with a 10-μ-thick coating in nine runs of 100 g each at 1600°C. By extrapolation of data obtained with the 1-in.-diam coater, a specific methane flow rate of 4.2 cm^3 min^{-1} cm^{-2} (methane partial pressure of 668 torrs) was selected to give the desired deposition rate of 5 μ/min at 1600°C. The coating thicknesses obtained in the 2-min runs ranged from 8 to 11 μ and were essentially the same on each particle within a given run.

In the second experiment, we coated a 100-g batch of uranium carbide particles at 1700°C under conditions designed to produce a 50-μ-thick coating. After 15 min at a specific methane flow rate of 2 cm^3 min^{-1} cm^{-2} (methane partial pressure of 425 torrs), the average coating thickness was 51 μ.

Effects of Heat Treatment on Pyrolytic-Carbon-Coated Oxide Particles. — We have continued to study the effects of heat treatment on pyrolytic-carbon-coated oxide fuel particles. Spheroidal fuel particles, 150 to 250 μ in diameter, of fully dense sol-gel ThO_2 and of conventionally prepared 80 to 85% dense UO_2 are employed in these studies. The particles are coated at temperatures of 900 to 1400°C by pyrolysis of acetylene or methane in fluidized beds.

After heat treatments at temperatures up to 2700°C, no significant reaction between fuel particles and coatings 85 μ or thicker could be detected by metallography, radiography, or x-ray diffraction. All particles having coatings at least 65 μ thick were unchanged by heat treatment at

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1800°C, as well. Some thinner coatings, however, particularly those applied at low temperature (900 to 1100°C), ruptured and reacted with the fuel. The thinner coatings probably ruptured because they could not contain the equilibrium carbon monoxide pressure.

Examination of the coated particles after heat treatments at temperatures of 2000°C or higher revealed that large gaps between fuel particle and coating were produced in several batches of these particles. The as-applied density of the coating, which is controlled by the partial pressure of methane during coating, seemed to be an important factor in determining the size of the gap and, therefore, the void volume. Gaps were clearly visible in microradiographs of coated particles having coatings with an initial density of 1.56 g/cm^3, but they were absent in particles having a high-density coating (1.96 g/cm^3).

Gap formation was accompanied by a decrease in coating thickness, an increase in coating density, and a decrease in the crystallographic c_0 spacing. Measurements made from radiographs of particles before and after heat treatment also indicated that a slight increase in the outside diameter of the coated particles accompanied gap formation. Crushing strength was not determined on all heat-treated samples, but the values obtained showed that the crushing strength decreased, except for the samples with very thick coatings (approx 165 μ), by about 50% after gap formation at 2000°C and by about 70% after gap formation at 2200°C.

Shrinkage of the particle during heat treatment would, of course, influence the size of the gap. The ThO_2 particles were fully dense, however, and were assumed to have no effect on the gap formation. On the other hand, shrinkage of the low-density UO_2 particles during heat treatment could have occurred. Weight-loss determinations showed that little or no carbon was lost due to reaction between oxide particles and the coatings during heat treatment and gap formation, except where coatings ruptured.

In pyrolytic-carbon-coated fuel particles for power reactors, a void volume between fuel particle and coating may be desirable as a fission-gas reservoir and to accommodate fuel swelling. To test this concept, two batches of conventionally prepared, enriched UO_2 particles were coated with low-density pyrolytic carbon at 1400°C and heat treated for 15 min at 2400°C to form a gap of 10 to 20 μ. A second pyrolytic-carbon coating was then applied at 1800°C to one of the batches. These two batches were carefully characterized and submitted for irradiation testing in a sweep capsule facility.

Solid Fission Product Release upon Postirradiation Heating of Coated Particles. — We conducted postirradiation annealing tests on 100 pyrolytic-carbon-coated $(Th,U)C_2$ particles from batch GA-314, which had been irradiated to 0.30% uranium metal burnup in experiment B9-21. Fifty particles were heated for 11 hr at 1370°C and then 7 hr at 1700°C. The other 50 particles were heated for 19 hr at 2000°C. We observed no coating failures. The nonvolatile fission product release rate is given below. A burst of fission products occurred at the start of heating. This is most obvious for the elements with the highest release rates and at the higher temperatures:

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Annealing Temperature (°C)	Hours at Temperature	Fission Product Release Rate (%/hr)					
		^{140}Ba	^{144}Ce	^{137}Cs	^{106}Ru	^{89}Sr	^{95}Zr
1370	1	0.4	0.04	0.03		0.7	≦0.004
	4	0.08	0.01	0.01		0.3	≦0.004
	6	0.1	0.02	0.02	0.01	0.2	≦0.004
1700	1	13	0.5	0.04		11	≦0.004
	6	0.6	0.04	0.02		2	≦0.004
2000	1	30	8	0.2	0.1	38	0.2
	6	6	5	0.3	0.07	7	0.02
	6	2	3	0.2	0.06	4	0.01
	6	2	3	0.2	0.06	3	0.01

Most of the fission products released remained in the graphite crucible at 1370 and 1700°C. Cesium, ruthenium, and zirconium were at or below detectable limits at these temperatures. At 1700°C, approximately 30% of the strontium was transferred to the quartz furnace liner. At 2000°C, 10 to 20% of the barium, >90% of the cesium, 45% of the ruthenium, and 90% of the strontium released were transferred to the quartz liner. Most of the cerium and zirconium remained in the graphite crucible, even at 2000°C.

GCR-ORR Loop No. 1. – Irradiation of experimental fuel assembly 14 continued. The fuel is a mixture of $(Th,U)O_2$ and ThO_2 sol-gel particles coated with pyrolytic carbon and is contained in two capsules, one of which is vented to the loop coolant. We retracted the fuel assembly from the neutron flux region during the ORR refueling shutdown on February 22 to permit decay of short-lived isotopes in preparation for removal of the assembly. However, we subsequently decided to continue irradiation for an additional ORR cycle, and the fuel assembly was returned to the high-flux position during the March 6 intercycle. The central temperature of the fuel in the vented capsule is being maintained at 2500°F with a coolant mixture of 30% neon and 70% helium. There was no significant change in the rate of release of the noble-gas fission products; the R/B (ratio of release rate to birth rate) has been approximately 1 to 2×10^{-5} for xenon and krypton isotopes.

LITR Capsule Irradiations. – Two capsules (L-CP9 and L-CP10), each containing pyrolytic-carbon-coated UC_2 particles in four separate compartments of ATJ graphite, continue to operate satisfactorily. We estimate the burnup in capsule L-CP9 to be 13.9 at. % uranium, and the remaining operable thermocouple indicates a fuel central temperature of 1860°F. Capsule L-CP10 has attained an estimated burnup of 2.7 at. % uranium, and the indicated fuel central temperatures are 1580 to 1860°F.

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ORR Poolside Irradiations. – Capsule 03A-6, containing EGCR-vendor fuel materials, was removed from the reactor after accumulating an estimated burnup of 9476 Mwd per metric ton of UO_2. Irradiation of a similar capsule (04-8) continues. The estimated burnup of this capsule has reached 3980 Mwd/metric ton, and the maximum indicated cladding temperature is 1540°F.

We terminated irradiation of sweep capsule 05-8, which contains three 6-cm-diam fueled-graphite spheres, on March 6, after it had reached an estimated burnup of 6.3 at. % of the heavy metal.

AVR capsule 01A-8 was also removed from the reactor at the March 6 shutdown, after achieving an estimated burnup of 5.0 at. % of the heavy metal. Operation of this capsule was steady and uneventful, with fission-gas release rates (R/B values) of approximately 10^{-4} for krypton and xenon isotopes.

BeO-Graphite Compatibility Experiment. – In support of the TARGET reactor concept, we are conducting an irradiation experiment for investigating the compatibility of BeO in contact with graphite at high temperature (1500°C).

The Mark I in-pile capsule assembly was removed from the reactor on March 12, 1965, after it had been in the reactor for the one planned ORR cycle. It had achieved a maximum temperature of only 1150°C. Before removal, we injected epoxy into the primary system gas lines to test the procedure planned for removal of the Mark II capsule after irradiation. The epoxy serves to seal the gas lines between the capsule and the point at which they are severed during preparation for shipment to the hot cells.

The Mark II capsule was installed in the reactor immediately following the removal of the Mark I. Modifications incorporated in the Mark II capsule were successful in raising the irradiation temperature to the design value of 1500°C. The capsule is presently operating at 1500°C with a control gas consisting of 100% helium and a sweep gas through the primary containment vessel consisting of a mixture of 15% helium and 85% argon.

EGCR Fuel Temperature Coefficient. – A complete reanalysis was made of the results of the experiment conducted in the Physical Constants Test Reactor (PCTR) at Hanford for determining the fuel temperature coefficient of the EGCR lattice. An improved method of analysis was employed that increased the statistical significance of the high-temperature data and gave greater confidence in the temperature dependence of the coefficient. A reinterpretation was also made of the results of Monte Carlo calculations by Westinghouse, which covered a wide range of temperature and surface-to-mass ratio. The survey included, in addition, the calculations by Nordheim and the experiments by Hellstrand and by Palowitch and Frantz.

The interpretation of all calculations and experiments was made on the basis of a ^{238}U Doppler coefficient with a temperature dependence given by

$$\frac{I_{28}(T) - I_{28}(T_0)}{I_{28}(T_0)} = \gamma(T^{1/2} - T_0^{1/2}) , \qquad (1)$$

[redacted]

where γ is a function of the surface-to-mass ratio, S/M, of the fuel element. The results of the Westinghouse calculations and the PCTR experiments were in good agreement and well correlated by Eq. (1). These results gave

$$\gamma = A(S/M)^n , \qquad (2)$$

where $A = 0.0117 \pm 0.008$ and $n = 0.21 \pm 0.04$. (The quoted errors are the rms uncertainties in the coefficients obtained by least-squares fitting.)

The calculations by Nordheim and the measurements by Hellstrand and by Palowitch and Frantz are also consistent with Eqs. (1) and (2) with the same value for n as given above, but the indicated value of the constant A is lower by approximately 20%. No explanation of this discrepancy is offered here, but it may be significant to note that the Monte Carlo calculations and the experiments giving the higher coefficient were all based on the ultimately desired quantity, the reactivity of the lattice. The calculations and experiments giving the lower coefficient were all based on the analysis of single isolated lumps.

For the EGCR, the new correlation of the PCTR experiment gives the same fuel coefficient as before at room temperature and gives a 10% reduction in the coefficient at operating temperature.

Steam-Graphite Reaction Test. — A graphite sleeve dimensionally similar to an EGCR fuel-element sleeve was fabricated from EGCR moderator graphite and exposed to 1500°F superheated steam for 550 hr. We maintained the flow rate over the test specimen at approximately 54 lb/hr. The reaction rate varied with the burnoff, from approximately 3.5 mg/hr per cm^2 of graphite at 1% burnoff to 6.5 mg/hr per cm^2 of graphite at 50% burnoff.

EGCR Fuel-Element-Channel Flow Tests. — Flow tests in air were conducted on a top-dummy fuel-channel section for the EGCR that we had modified by the addition of a 32-ring labyrinth to restrict annulus flow in a graphite surveillance channel. For flow rates in the range of interest for EGCR operation, about 97 lb/hr, the pressure drop across the entire channel for EGCR temperatures and pressures was 9.6 psi. The pressure drop across the top dummy represented the largest fraction of the total, being 8.7 psi. The results are being studied.

REACTOR FUELS AND MATERIALS

Solid Reaction Studies. — We measured self-diffusion coefficients in reactor-grade hafnium (2.1% Zr) over the temperature range 1795 to 1995°C using the radioisotope ^{181}Hf and standard sectioning techniques. The coefficients are approximated by $D = 2.8 \times 10^{-3} \exp(-42{,}000/RT)$ cm^2/sec. The small values of the preexponential factor and the activation energy relative to those expected from diffusion theory show that hafnium belongs to the class of anomalous body-centered cubic metals that includes β-zirconium, β-titanium, and γ-uranium.

Nondestructive Test Development. — Eddy-current testing is an excellent method for the rapid determination of very small discontinuities in

items such as tubing. This is because the use of mechanical systems for moving the tube through or past the eddy-current coil allows electronic discrimination and amplification of the quickly changing signals due to flaws without response to the slower changes due to insignificant variations. However, the more common manual probe systems cannot make this discrimination; therefore they have low sensitivity to defects and suffer from interpretation difficulties. We have developed a manual eddy-current probe that is ten times as sensitive. This probe vibrates rapidly; thus its motion past a small defect produces a quickly changing signal, allowing discrimination and amplification previously possible only with automated mechanical systems.

The eddy-current probe is mounted between two electromagnets. Switching current from one coil to the other vibrates the spring-loaded probe. The signals from the probe are fed to an electronic filter, which is tuned to pass only the high-frequency signals from defects.

Mechanical Properties. — The embrittlement of stainless steels during irradiation at elevated temperature has been attributed to accumulation of helium produced by the reaction $^{10}B(n,\alpha)^{7}Li$ in the boron impurity. To verify this, we purified type 304 stainless steel by electron-beam melting, reducing the boron content from 3.90 ppm to values ranging from 0.015 to 0.130 ppm. Although we found the purified material somewhat more ductile than the unpurified after irradiation in the ORR to 4.5×10^{20} neutrons/cm^2 (thermal) and 3.5×10^{20} neutrons/cm^2 (>1 Mev), the variation was far less than expected from the transmutation of the boron. Because of the appreciable fast-neutron flux in the reactor, another source of helium is the (n,α) reaction in the major constituents of the alloy. When we considered the total helium contents from all sources, the variation in ductility fit. Because thermal reactors inevitably contain fast neutrons, we conclude that boron removal does not solve the problem of elevated-temperature radiation embrittlement of stainless steels.

POWER REACTOR FUEL PROCESSING

Fluoride Volatility Pilot Plant. — The system is being cleaned out and decontaminated, following completion of the processing of short-cooled aluminum-uranium alloy fuels by the molten-salt fluoride volatility method.

The first step in the cleanout process consisted in aqueous flushing of the UF_6 portion of the equipment to recover residual uranium. The uranium so recovered brought the material balance within limits of analytical error.

Ammonium oxalate flushing of the salt-processing portion of the equipment was started. The ammonium oxalate solution should dissolve and remove any residual salt in the system. Decontamination will continue in preparation for examination of the equipment.

Processes for HTGR Fuel. — A promising processing method for HTGR fuel (carbon-coated Th-U dicarbide particles dispersed in a graphite matrix) involves combustion of the fuel in a fluidized bed of inert alumina at 700 to 750°C, followed by acid leaching to recover the uranium and thorium. Five-hour leaching tests were conducted with a bed composed of

67% Norton RR grade Al_2O_3, 25% ThO_2, 6% U_3O_8, and 2% carbon. Uranium and thorium recoveries greater than 99.5% were obtained by leaching with boiling 4 M HNO_3–0.05 M HF–$Al(NO_3)_3$ solutions. Variations in the $Al(NO_3)_3$ concentration between 0 and 0.1 M had no effect on the recoveries. Equally high recoveries were obtained with boiling 13 M HNO_3–HF solutions in which the HF concentration was 0.02 to 0.05 M. In all cases the product solutions were about 0.6 M in thorium.

Processes for Advanced Fuels. – Dissolution of sol-gel ThO_2-UO_2 microspheres in pressurized nitric acid solutions is being studied. Complete dissolution of 97% ThO_2–3% $UO_{2.4}$ microspheres (300 to 400 μ in diameter; density close to theoretical) was achieved in 24 hr at 300°C in 2 M HNO_3 when the NO_3/Th mole ratio was about 100. Pure ThO_2 microspheres (259 to 300 μ in diameter; density close to theoretical) were much less reactive. In 24-hr reactions with 2 M HNO_3 at 300°C, the amounts dissolved were 17, 20, 35, and 49% when the NO_3/Th mole ratios were 10, 30, 50, and 100 respectively.

Shear-Leach Process. – Engineering-scale development and evaluation of a batch shear-leach process for the recovery of fissile and fertile material from spent power reactor fuel assemblies were continued.

The fabrication of mechanical equipment to demonstrate multitube withdrawal of fuel rods from second-generation fuel such as the Consolidated Edison core B assembly was completed. Preliminary tests with the above fuel indicate that the equipment is useful for hot-cell operation. In tests in which fuel tubes were withdrawn from an unirradiated fuel assembly, a total force of about 300 lb was required to withdraw 14 fuel tubes containing UO_2 pellets.

The heat transfer coefficient for sheared, stainless-steel-clad UO_2 is being determined experimentally to evaluate the heat buildup in a basket of sheared fuel during storage. The coefficient for dislodged UO_2 fines (in an air atmosphere) with a mean particle size of 120 μ and 38% voidage increased linearly from 0.256 Btu hr^{-1} ft^{-2} $(°F)^{-1}$ at 200°F to 0.448 at 1600°F.

In a study to determine the safety of shearing zirconium, 3/8-in.-OD Zircaloy-2 rods were cut dry and then with water coolant. A high-speed, medium-hard, abrasive disk-saw blade was used. An extremely brilliant stream of sparks and dense smoke was produced by dry sawing; however, the wet sawing (spray nozzles directed on the saw blade) decreased the sparking to a tolerable level. The amount of Zircaloy-2 particles collected was about 100% greater for wet cutting, indicating that considerably more zirconium is burned when cut dry.

The amount of zirconium particles less than 400 μ in diameter (the size considered hazardous and requiring special handling) was 94.5 and 79 wt % for wet and dry sawing respectively. The volume ratio of blade material to metal particles produced is about 7 to 1. According to the literature, this dilution of metal fines by abrasive particles will assist in preventing spontaneous or static-spark reactions.

Fluoride Volatility Processing: Development of Process for Dissolving Stainless-Steel-Clad UO_2 Fuel Elements. – The solubilities of iron and nickel fluorides in molten fluoride mixtures were studied to see whether an attractive basis can be found for extending the Fluoride Volatility Process to include stainless-steel-clad UO_2 fuel elements, which

comprise the major fraction of the elements in presently operating nuclear power reactors. The phase relationships of the $LiF-FeF_2$ system were examined first; a eutectic melting at 620°C was found for a composition containing 38 mole % FeF_2, and no interfering intermediate compounds were observed. In other studies, NiF_2 was found to be nearly equivalent to FeF_2 in its ability to lower the liquidus temperature of LiF. These results are considered sufficiently encouraging to warrant a more detailed study of a wider range of fluoride mixtures.

Fluoride Volatility Processing: Plutonium Recovery from Molten-Drop Fluorination Tests. — In the molten-salt volatility process, PuF_6 volatilization is hindered by an unfavorable equilibrium that necessitates fluorination times of several hours to even volatilize half the plutonium. The behavior of uranium in molten-drop fluorination tests (gas phase continuous) suggested that plutonium recovery would be faster with this method (ORNL-3718, p. 9). This was confirmed by preliminary results. Four drop-test experiments were made with 50-50 mole % $NaF-ZrF_4$ containing 0.26% plutonium (added as PuF_3). At 540°C, plutonium recoveries were 21 to 69% for drops ranging from 115 down to 58 μ in diameter. At 640°C the removal was 81 and 88% for drops 115 and 75 μ in diameter. The 75-μ-diam drops were estimated to have had a contact time of 7.3 sec in the 52-in.-high fluorination tower. The temperature and diameter of the drops had a smaller effect on volatilization rates with plutonium than with uranium.

Fluoride Volatility Processing: Inhibiting Corrosion. — The use of an inert gas introduced below the surface of the molten salt was studied as a possible way of reducing the severe corrosion that usually occurred with 52-37-11 mole % $NaF-LiF-ZrF_4$ and HF at 650°C. This melt, originally considered for processing stainless-steel-based reactor fuels, was used in the experiments because of its extreme corrosivity — particularly at the salt-gas interface. The experiments (laboratory scale) were conducted at Battelle Memorial Institute.

A letter report (BMI-X-329) was recieved that describes the entire series of tests. Preliminary results of the first eight tests with coupons were reported previously (ORNL-3680, p. 10); an inhibitive effect was noted when argon was used and the coupons were insulated from the container. The last experiments, using individual 2-in.-diam containers and argon, nitrogen, and hydrogen in separate tests, showed very little improvement in the degree of attack at the interface, compared with the attack when no inert gas was used.

This study concluded BMI's corrosion tests with molten salt for the Fluoride Volatility Program.

Fluoride Volatility Processing: Plutonium Recovery Step in Fluidized-Bed Method. — In processing low-enrichment fuel, the recovery of plutonium as well as uranium is necessary. A program of small-scale fluidized-bed tests was initiated to determine the factors involved. The first results, which were obtained with a mini-test unit (ORNL-3771, p. 7), indicated that temperature is an extremely important factor. Plutonium retention, at least in tests of 1 to 2 hr duration, increased markedly at 550 to 660°C over that observed at 450 to 550°C. These tests were made using 5 g of fused alumina (Norton RR 120) with a PuO_2 content of 2%. In 1-hr tests with a fluorine superficial velocity of 0.5 fps, plutonium retentions were 0.36, 0.40, 6.3, and 18.8% at temperatures, respectively, of

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450, 500, 550, and 600°C. In two 2-hr tests, the retentions were 0.58 and 4.2% at 500 and 550°C. In one 0.5-hr test at 550°C, plutonium retention was 3.1%, possibly because of the low F_2/Pu mole ratio of about 1000. At equilibrium an F_2/Pu ratio of 100 is required at 500°C; under dynamic conditions a ratio of 1000 is possibly not sufficient. These results indicate a greater diffusion of plutonium, probably as PuF_3 or PuF_4, into the fused alumina at the higher temperatures, and consequently less efficient fluorination of plutonium to PuF_6.

Small beds of porous NaF (regenerated from NaF·HF and supplied by the Harshaw Chemical Company) were used in all the above tests to trap PuF_6. Material balances were about 100%, due to the effectiveness of NaF in trapping the volatilized PuF_6. The plutonium, however, cannot be easily desorbed from the NaF. The traps were 2-g beds of 12- to 20-mesh porous NaF at 325°C. The PuF_6 was completely sorbed in 0.2 sec. Loading of the beds was characteristically high at their entrances, with a sharp line of demarcation where sorption stopped. The color of the loaded beds was blue-green, in contrast to the usual yellow complex of $2NaF \cdot UF_6$. The capacity of porous NaF is about 100 mg of plutonium per gram of NaF, below that for uranium by a factor of about 7. In one test, an attempt at desorbing the plutonium from a saturated bed in a stream of fluorine at 600°C resulted in only 0.3% desorption in 2 hr.

THORIUM UTILIZATION PROGRAM

Irradiation of Thorium Fuels. — Detailed evaluation of the microstructures of the five vibratorily packed capsules of ThO_2—3% UO_2 under examination has not yet been completed. The dissolution technique for burnup analysis was modified, and new samples from the rods will be analyzed as soon as they can be obtained from the portions of the rods still in the hot cells. Concurrently, the detailed examination of the fourth heat-rating capsule in ETR group I will be completed.

X-ray diffraction data on transverse metallographic specimens indicate very little change in lattice parameters, even for the pellet fuel with a burnup of 120,000 Mwd per metric ton of thorium plus uranium. Since the high-heat-rated ETR group I capsules show columnar grain growth in the central region, additional x-ray diffraction patterns on longitudinal sections are being obtained. With these data, the preferred orientation for such grain growth under irradiation can be established for comparison with the nonirradiated thermal-simulation experiments being planned.

Fabrication of the ETR group III pure ThO_2 rods is complete. Densities ranging from 88.2 to 89.5% of theoretical in all seven rods were obtained. Final inspections, including closure-weld x rays, gamma scanning, autoclave testing, and volume and dimensional determinations, will be completed in time to meet the scheduled start of irradiation in April.

All capsules currently being irradiated are continuing to function normally, with no indications of failures.

^{233}U Distribution Facility. — About 18 kg of ^{233}U containing 50 ppm ^{233}U and 49 kg of ^{233}U containing 250 ppm ^{232}U were received and stored as U_3O_8 in aluminum cans in the dry storage holes of the Building 3019 penthouse. Each can contains about 0.45 kg of ^{233}U.

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Construction of the ^{233}U liquid storage facility by the H. K. Ferguson Company was started.

Thorium-Uranium Recycle Facility (TURF). — Project completion for the TURF is scheduled for December 1966.

Several minor modifications to the facility design and specifications were made during the month to correct errors in the contract package. The Atomic Energy Commission issued an addendum containing these minor revisions of the bid package. In issuing it, they also extended the closing of the bid period from March 17 to March 25. Procurement of the various items to be supplied by ORNL is proceeding. No difficulties have been encountered in meeting the required delivery time, with the possible exception of the buried-ventilation ductwork. Here, there has been some difficulty in obtaining satisfactory sheet metal with which to make the ducts. Also, contracts were signed for obtaining stainless steel valves and eductors. The price was well within the budgeted figure.

As to sol-gel equipment, a solids-level detector was designed and made for installation on top of the vertical tube furnace now being tested. The detector consists of a cone-shaped piece which is lifted up to allow solids to be introduced to the top of the furnace and then is allowed to fall and come to rest on top of the bed. Its position is read out by means of a variable resistor on a rod that extends through the top of the furnace-tube enclosure and is sealed by means of a bellows. It is essential that a detector be provided to prevent overfilling or underfilling of the furnace and to ensure that a blockage has not developed in the furnace.

Also, equipment was designed and built for use in sampling solids and liquids. The solids sampler will continuously remove a portion of the fired oxide stream from the vertical tube (or other) furnace. The liquid sampler, which is to be used for sampling both uranyl nitrate and thoria-urania sol, consists of a plug- or ball-type valve in the stream being sampled. It captures a volume of the liquid and delivers it to a sample receiver when the plug is rotated 90°.

With reference to the sol dryer, preliminary specifications for performance and function were completed and issued to several potential designers and fabricators. The details of design and specification will be discussed with those interested to determine where our specifications and requirements may be unrealistic and to best decide how this device should be specified, and how to proceed with the design and fabrication.

Engineering flowsheets for the sol-gel process are in preparation and will be distributed when completed. They include numbering of all lines and penetrations through which the lines will pass.

A quarter-scale model of the sol-gel process cell for the TURF was made for use in designing the in-cell equipment and its piping system. Preliminary design and layout of the equipment has been under consideration during the month. It is tentatively planned to try to minimize the number of components to be brought in separately; that is, all the liquid-handling equipment will be placed upon or within the same equipment rack if this can reasonably be accomplished. By so doing, the number of connections that must be made in the cell between various items will be minimized, as will the difficulty in achieving registration between components in the cell. The principal connections to be made then will be those

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which run from the equipment rack to the wall of the cell, and which for the most part carry services from outside the cell to the in-cell equipment.

The critical-path schedule for design, fabrication, and testing of sol-gel-process equipment was revised and updated to reflect the latest estimates of manpower and time requirements. The completion date for installation is December 31, 1966. The latest schedule indicates that this target date can be met and that there are about 40 days of "float time" for the entire project.

Coated-Particle Development Laboratory. — Modifications to room 241, Building 4508, to make it into a coated-particle development laboratory, are complete.

The Astro furnace was brought up to temperature six times during the month. A steam leak in the large gas distributor was found and corrected. Two methods of sensing and controlling the temperature were furnished with the furnace systems. To control the lower temperatures, a boron-doped graphite thermocouple is used; for the higher ones, a Radiamatic head is used.

Problems were encountered in achieving reproducible millivolt outputs for given temperatures with the boron-graphite thermocouple. It may be necessary to use the Radiamatic head or other means for low-temperature control. Temporary gas flowmeters were calibrated. Checkout of the furnace-program controller and minor modification continues.

A 4-in.-radius ball joint was ground on one distributor and matching graphite cone. This was tested during the week of March 1, and then thorium oxide particles were fluidized with helium. Coating, using methane, will await the installation of furnace enclosures.

Fueled-Graphite Process Development. — The fluidization experiments have been continued in a Plexiglas model of a 3-1/2-in.-ID fluidization column with 5-in.-high and either 3/8- or 1/2-in.-ID central guide funnels for the particles.

The objective of this design is to make the fluidizing and coating pattern independent of bed-height and bed-volume changes during the coating step. This is achieved by placing a funnel in the center of the column. The fluidizing and coating gases entrain particles upward in this funnel to a distance that can be controlled by proper design. The particles then fall back between the funnel and the chamber wall. The amount of gas-solid contact and the cycle time can be controlled by adjusting the height and diameter of the funnel. So far, experiments have been carried out with a 90° included-angle bottom cone and 3/16- and 1/4-in.-ID single gas inlets. The ends of the guide funnels facing the gas inlet were enlarged to 1 in. in inner diameter, with either 30 or 60° included angles. Iron-shot particles (300 to 500 μ in diameter) were used for the experiments. The influence of the width of the gap between the bottom cone and the edge of the funnel on the amount of particles circulated was studied by varying the gap from 1/8 to 3/8 in., in increments of 1/16 in., and the air flow rates from 30 to 60 standard liters/min, in increments of 5 standard liters/min. With the present configuration, up to 2 kg of 300- to 500-μ-diam iron-shot particles could be circulated per minute.

With respect to coating by the rotating-drum method, experiments on the motion of particles in finned-drum plastic models at room temperature

are being continued. It was assumed that the degree of radial and longitudinal mixing in a horizontal rotating drum could be used as a parameter to define the probability of particles coming to the surface of the bed. This will be essential to coating spherical particles in such a drum. A method was developed to withdraw samples from any position in the charge after any number of revolutions of the drum.

Since no method of labeling for particles of similar physical properties and discrimination after mixing was available, mixing experiments were carried out with iron-shot particles of the same density but of different sizes (417 to 500 μ and 590 to 833 μ in diameter). Thus, discrimination is possible by sieving and weighing.

Experiments were made in a 9-in.-ID, 9-1/2-in.-long Plexiglas drum with vertical end walls. Double-thread-type, 1-1/2-in.-high fins with an 8-in. pitch were inserted. So far, a charge depth equal to 50% of the drum radius, with a coarse- to fine-particle ratio of 4:1, has been studied at 10, 20, 30, and 40 rpm. The fine and coarse particles were initially placed at opposite ends of the drums or in layers, and the degree of mixing was measured after increasing the number of revolutions. Mixing was very poor at 10 and 20 rpm. With increasing rotational speed, it improved. Almost homogeneous distribution could be achieved after 100 revolutions at 40 rpm. This signifies good agitation. More experiments will be carried out with various charge depths and fin configurations.

As to the prototype drum coater, design continued. Heat-transfer calculations to be used for determining insulation requirements are in progress, and detailed design of some mechanical components is under way.

Conversion of ThO_2 to ThC_2. — Studies of the conversion of ThO_2 to ThC_2 were made in a new rotary converter designed to have a balanced mass of graphite parts and heat shields to minimize temperature gradients in the system. The rotating crucible into which the ThO_2 and carbon are charged is 2-1/4 in. in diameter and 3 in. long, compared with the 1-3/4-in.-diam, 5-in.-long crucible used previously.

The results obtained in the new system were comparatively poor under the conditions employed previously (10 g of lampblack and 10 g of 300- to 400-μ ThO_2 particles, heated at 2150°C for 6 hr at a rotational speed of 44 rpm). The increase in the radius of the crucible probably resulted in an increase in the slippage of the particles during rotation; this would decrease the movement of the particles and result in poor contact with the lampblack.

Reduction of the quantity of charge to 5 g of ThO_2 and 5 g of lampblack markedly improved the degree of conversion, but it was still variable and incomplete. By comparison, a 23-g charge of 200- to 250-μ ThO_2 particles coated with 65 μ of porous pyrolytic carbon (17 wt %) was completely converted without benefit of additional carbon; however, the particles were agglomerated. This indicates that variations in conversion, and incomplete conversion, are not due to temperature effects in the new system but rather to poor contact with the carbon.

Development of Metal Fuels. — Thorium powder for use as the matrix material in compatibility studies with selected uranium compounds was prepared from iodide thorium by hydriding, milling, and dehydriding. All dispersoid materials were received and crushed to −170 +325 mesh. Samples of UB_2, UB_4, and (Th,U)C were submitted for x-ray analysis to determine

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the phases. Fabrication of compatibility specimens is in progress, and the heat-treatment phase of the study should be completed during the next report period.

Sol-Gel Microsphere Preparation. — Microsphere-formation studies have continued, using the method of thoria or thoria-urania sol dispersion in an immiscible solvent that sets the sol droplets to gel microspheres by extracting water. Further experiments confirmed earlier results, which showed that the fired density of thoria microspheres is a function of the extent of use of the 2-ethylhexanol drying solvent.

The first thoria microspheres prepared by starting with fresh 2-ethylhexanol have tap densities of about 6.0 g/cm^3, which indicates particle densities of about 10.0 g/cm^3. After 24 hr of accumulated use of the solvent, the tap densities of the product are in the range 4.8 to 5.4 g/cm^3, indicating particle densities of 8 to 9 g/cm^3. There is evidence that the ion exchange resin used to remove nitrate from a side stream of the drying solvent plays an important part in causing density variations. One run, made before the resin was used, gave a product that had a tap density greater than 6 g/cm^3; this was after 24 hr of operation. In addition, three samples of thoria microspheres, recently prepared with 2-ethylhexanol that had not contacted the ion exchange resin, had tap densities of 6.0 to 6.1 g/cm^3.

During future runs, the study of nitrate removal by a simple scrub will be carried out. Results will be compared with those obtained when ion exchange resin is used.

Several rotary dispersers, with 0.010- or 0.016-in.-diam holes in 1-1/4-in.-OD cylinders, were made and tested for dispersing sol into drops. These feeders appeared to work well while discharging the sol directly into the 2-ethylhexanol.

Gas Evolution from Sol-Gel Thoria-3% Urania Fuels. — Exposure of sol-gel material to wet CO_2 at 1000°C caused as much as a sixfold reduction in the gas-adsorptive capacity, together with corresponding reductions in the surface area of the material. (The surface area was determined by nitrogen adsorption.) Exposure to dry CO_2, to water vapor alone, or to air did not cause such a reduction in gas-adsorptive capacity.

Permeability of Vibratorily Packed Fuels. — The previously reported change in effective permeability for sol-gel fuel compacts upon changing from water to gas as the test fluid at room temperature is not observed when the gas is steam. Steam (250 to 300°C) gave permeability values for a particular compact identical with those obtained with liquid water, within the precision of the measurements. Nitrogen yielded values several times that of water, as observed with other samples. The postulated surface interaction with water is apparently equally effective in the vapor phase.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — The program of experiments and shakedown operation with flush salt was concluded, and the reactor was drained and cooled. At the time of shutdown on March 4, flush salt had

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been circulated at 1100 to 1200°F for a total of almost 1000 hr. Coolant salt was circulated more than 1200 hr. Between February 11 (when a partial power failure caused the coolant loop to drain) and the end of the run, there were no equipment failures or malfunctions which interfered with the operation. During the last weeks of the run, we obtained information on entrained gas volumes, accumulation of salt in the fuel pump overflow tank, and krypton behavior in the fuel system.

Response to pressure changes and pump operation indicated that when the salt in the pump was at the normal operating level, entrained gas in the fuel circulating loop amounted to 0.6% by volume. There was no apparent entrainment in the coolant loop, where there is no spray ring in the pump bowl.

We observed a slow accumulation of salt in the overflow tank when the salt level was as much as 2 in. below the lip of the overflow pipe. (The rate was 1 gal/day at 2 in. and was zero at 3 in.) The explanation is not known, but at the level chosen for operation there is no accumulation.

Preliminary analysis of the experiments with ^{85}Kr gave time constants for sweeping the pump bowl, stripping gas from the circulating stream, and transferring krypton between graphite and salt. The stripping effectiveness of the pump bowl spray is apparently less than 50%, somewhat less than we expected. Even so, estimates of ^{135}Xe poisoning based on the observed krypton behavior are still only a few tenths of a percent $\delta k/k$.

While the reactor is shut down, a number of minor repairs and modifications are being made in preparation for the zero-power nuclear experiments. These include replacement of several coolant-air valves, modification of lines that carry coolant air to freeze valves, replacement of two removable heater units, and modification of several other heater units for better remote maintenance.

The sampler-enricher installation is nearing completion, and it will be tied in to the fuel pump before operation resumes.

A four-week advanced training session for the reactor operators was begun on March 15.

Excavation for the vapor-condensing tanks was begun.

Chemical Development. — Throughout the prenuclear test period, specimens of the flush salt and coolant salt were obtained for chemical and spectrographic analysis. Approximately 6783 kg of $LiF-BeF_2$ (66-34 mole %) was charged into the reactor, 4173 kg into the fuel system and 2610 kg into the coolant system. At the end of the prenuclear test period, the flush and coolant salts had circulated for a period of approximately 1000 hr. At this time the mean values of iron, chromium, and nickel concentrations were 45, 45, and 7 ppm in the fuel circuit, and 43, 45, and 7 ppm in the coolant circuit. These results are evidence that excellent procedures were employed during the prenuclear test period. The concentration level of chromium, a sensitive indicator for the presence of oxidizing impurities (including HF produced by hydrolysis), is remarkably low, sufficiently low, in fact, to suggest that no measurable corrosion of the reactor hardware occurred during the prenuclear test period.

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Preparation of MSRE Materials. — The production of all component fluoride mixtures which will make up the MSRE fuel mixture is essentially complete. Approximately 10,500 lb of the barren fuel solvent, ^{7}LiF-BeF_2-ZrF_4 (64.7-30.1-5.2 mole %), is now on hand. The enriched fuel concentrate mixture, ^{7}LiF-$^{235}UF_4$ (73-27 mole %), which contains about 90 kg of ^{235}U, has been prepared in six equally sized batches and is also in storage. The preparation of about 600 lb of the depleted fuel concentrate as a chemically identical mixture, with UF_4 that has been depleted of ^{235}U, is complete. In the next production operation, 154 fuel-enriching capsules will be filled from one batch of the enriched fuel concentrate mixture to provide for initial approach to criticality and sustained nuclear operation of the MSRE. When all components are combined in the fuel drain tank, the reactor fuel mixture will have the approximate composition ^{7}LiF-BeF_2-ZrF_4-UF_4 (65.0-29.1-5.0-0.9 mole %).

Radiation Effects. — Detailed examinations of MSRE fuel, metal, and graphite from earlier in-pile tests such as experiment 47-5 were sometimes interrupted or postponed after the more important results became definite. Some results from additional examinations, however, gradually accumulated, and a completed assessment of all the findings from experiment 47-5 has been made.

The results were in line with the previously established relation of material incompatibilities of fuel radiolysis at room temperature, and were in marked contrast with the favorable results from the more recent experiment 47-6. Heavy uranium deposition, surface roughening, cracking, and fuel penetration were observed in graphite specimens from capsules in which the fuel salt was extensively radiolyzed. Much less damage to graphite was observed in capsules where little or no fuel radiolysis took place. The INOR-8 withstood exposure to fluorine and reduced fuel, as well as extended contact with fissioning fuel, with only slight indications of corrosive attack. Carburization was observed only in two specimens which were in close contact with graphite. The chemical analyses of the salt, though imprecise, indicated no gross chemical changes in the fuel. Gamma scans of salt samples showed a tendency for fission product ruthenium to concentrate in regions exposed to the capsule gas spaces.

MSR Processing. — Construction of the MSRE fuel processing system was completed except for installation of the shielded sampler and the UF_6 absorption equipment. Preliminary testing was started. The heaters were energized, and thermocouple response was observed. A low-flow water-metering and vaporizing system was set up for calibration of the H_2O-HF collection equipment and the in-line moisture analyzer for following oxide removal from the molten salt. The refrigeration unit and brine circulating system for H_2O-HF condensation was put into operation. A portion of the reactor operating group was trained for operation of the fuel processing system.

Studies are in progress to determine the capacity of NaF at 750°F for removal of chromium fluoride from the off-gas stream of the fuel processing system. Six runs to date have shown that this capacity can be improved by greater than a factor of 2 by using NaF pellets with a surface area of 0.07 m^2/g, as compared with 1 m^2/g for the pellets normally used for this purpose.

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Analysis of Radioactive MSRE Fuels: Setting Up Equipment and Training of Personnel. — Checking of the analytical equipment installed in cells 5 and 6 of Building 2026 was continued through the past period. Calibration curves were prepared for the determination of beryllium by the photoneutron method, and these curves indicated that the apparatus was working properly. A calibration curve for the determination of nickel was also prepared, and it indicated that the new-type filter photometer functioned as expected.

The new decoupling device for the transfer container was placed in cell 5 in February. It operated satisfactorily. The design allowed coupling and uncoupling of the transfer container without bringing it into contact with the manipulator fingers in cell 5. This will reduce the probability of contaminating the transfer container during the loading and unloading operations.

Analysis of Radioactive MSRE Fuels: Evaluation of Methods. — The effect of ZrF_4 on the determination of oxide by the hydrofluorination method was studied. Zirconium fluoride was added to an 80-g sample of Li_2BeF_4 so that the ZrF_4 concentration was 5 mole %. Approximately 800 ppm of oxide was added as BeO. Tests were conducted at 600°C, then at 500°C. At 500°C the time required for the oxide in the salt to react with HF was about 3 hr, as determined by successive Karl Fischer titration of the effluent gas. At 600°C about half of the oxide had reacted with HF after 3 hr of hydrofluorination.

An experiment was designed for possible application of a continuous measurement technique to the determination of oxide by the hydrofluorination method. A nickel adsorption tube, 1/2 by 7 in., equipped with nickel frits was made. The tube was filled with about 7 g of NaF and heated to 110°C. A 48% $HF-H_2O$ gas mixture was passed through the column, and the effluent gas was analyzed by successive KOH and Karl Fischer titrations. No water was held by the adsorber; however, even after 30 min at least 60% of the HF was being adsorbed on the NaF.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. — All fuel plates for the first reactor element have been fabricated and have recieved a uranium homogeneity inspection. Those for the inner element have been assembled between the side plates and are now ready for welding.

Both the fuel element and the control rods, which will be used during the flow studies preceding reactor startup, have been completed. It was shown that only minor relaxations will have to be made in the very tight dimensional tolerances which were set as the goal of the element final machining study. No major deviations were required during the machining of this element. For the flow-test inner control rod, the cylinder which had failed by cracking during explosive forming was repaired and resized after being shrunk into the forming die. No additional cracking or recracking of the previous failures was encountered; this indicated a solution to the cracking problem. The dimensional tolerances on the final cylinder were excellent. The radial positions varied from nominal by only +16 and −14 mils, and only a 6-mil difference was found in the average diameters.

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Physics. – Fuel cycle calculations were completed for the second production core (9.4 kg of ^{235}U, 2.8 g of ^{10}B). The results indicate that with nominal loadings and distributions of the fuel and burnable poison, with a minimum-reactivity 300-g plutonium target, and with unpoisoned (new) beryllium, the core lifetime will be about 13 days. With poisoned beryllium the lifetime should be about 9 days.

The results of xenon override calculations for the second production core indicate that the override time is less than previously calculated. For this core, the table below gives typical xenon override times for step changes in power from 100 Mw to 10 and 0 Mw.

Time at 100 Mw (days)	Time to Reach Zero Reactivity (min) After Step Reduction to Specified Power	
	10 Mw	0 Mw
1	35	30
5	20	18
10	7.5	6.5

In view of the large loss in core lifetime associated with the poisoning (buildup of ^{6}Li and ^{3}He) of the beryllium reflector, new fuel and burnable poison loadings are being investigated which will compensate for the reflector poison. Tailoring of cores might also be extended eventually to compensate for reactivity changes associated with the plutonium target and other experiments. It is presently anticipated that cadmium would be satisfactory as a supplementary burnable poison in the tailored cores.

Design and Analysis. – The field measurements at the horizontal beam tubes and the engineering facilities were completed, establishing the relations of the permanent reflector, pressure vessel, and concrete structure. The necessary changes in detail drawings of the various adapter pieces were made, and machining is in progress.

Piping and shielding drawings were completed for the ^{16}N probes in the subpile room. Water samples will be taken from four locations below the core and sent to the ^{16}N monitor station.

Instrumentation and Control. – Design has been completed, and the fabrication of the electronic and mechanical sections of the cladding failure detector is under way. The electronic components have been tested in the laboratory.

Tests have been conducted on the faulty-fuel-element detector in the pipe tunnel of the ORR to determine the required amount of lead shielding to perform on-the-line tests. On the basis of the performance in the ORR, fabrication drawings will be made of the units to be used in the HFIR.

Modifications are in progress to incorporate the Laboratory radiation and contamination alarm system in the HFIR.

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Component Fabrication, Development, and Assembly. — Fabrication of all major reactor components has been completed. These components were installed in the reactor vessel, and measurements, using optical tooling techniques, were made to check the alignment of the components. Concentric and azimuthal alignment of all components is well within the allowable tolerances. Proof testing of control drive rod and safety system parts is continuing.

Preoperational Activities. — Training of the startup crew, which consists of seven experienced engineers and four experienced operators, is continuing at the reactor site. Certification tests will be given to the engineers and the operators in May and July 1965 respectively. Eleven new operators, who were hired in January, are in training at the LITR. This group and the four experienced operators comprise the operator crew for the HFIR.

Testing and balancing of the ventilation system are continuing and are scheduled for completion in April. Dioctyl phthalate tests of the absolute filters in the special building hot exhaust and hot off-gas systems indicate an efficiency of >99.95% for removal of particles larger than 0.3 μ.

Critical Experiments. — Critical experiments associated with handling of the HFIR fuel elements have been completed insofar as experiments with the HFIRCE-2 (8.01 kg of ^{235}U) and HFIRCE-3 (9.4 kg of ^{235}U) cores are concerned. Most of the information obtained from these tests is applicable to anticipated future cores; however, some additional experiments will be conducted when different fuel elements are available. The experiments just completed included the development of an acceptance test that will be used for checking the reactivity of new fuel elements. In addition, the reactivities of the test cores were checked with various combinations of the individual fuel elements and reflector materials (steel, concrete, water, lead). Both the HFIRCE-3 and -2 cores were subcritical when completely submerged in water, the HFIRCE-2 core being about 1% less reactive than the HFIRCE-3 core. A report covering the handling of the critical experiments is being prepared.

Reports. — The HFIR Safety Analysis was transmitted to the USAEC on February 24, 1965, for review by the Division of Reactor Licensing and the Advisory Committee on Reactor Safeguards.

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PHYSICAL RESEARCH

Reactor Operations. — The ORR operated 87.9% and the LITR 90.2% of the time during January and February 1965.

Installation of combination ionization chambers which have an expected lifetime of five years was completed at the ORR. Americium-241 samples are being irradiated in the ORR to produce curium isotopes.

The BSR operated a total of 54.3 Mwhr during this period. Qualification of operators for the BSR has begun.

Reassembly of the Pool Critical Assembly was completed, and the reactor has been operated in preparation for training Laboratory personnel for some nuclear engineering classes for the University of Tennessee.

Waste Disposal. — The strontium removal efficiency of the lime—soda-ash—clay process, used in the treatment of process wastes, has been increased to 90% during the last four months from an average of 63% experienced during the previous year. This improvement was a result of more intensive treatment made possible by a one-third reduction of the volume of waste handled by the Process Waste Treatment Plant. The reduction in volume was accomplished through the use of the two 150,000-gal ponds added to the process waste system which enable the operators to separate and to discharge to the creek, without treatment, the uncontaminated portion of the waste from the main research area, Buildings 4500, 4501, 4505, 4507, and 4508.

Although the process waste system was at one time the main source of strontium activity released to the Clinch River, it contributed only 42% of the total during the year 1964. The remainder came from other sources such as the storm sewers, sanitary sewers, and laundry and from old deposits in the creek bed into which the process waste is discharged. The improvement in the efficiency of the process waste treatment operation will make the contribution of contaminants to the river by the process wastes even less significant.

Hot-Cell Operations. — Operations in the hot cells were normal. One experiment containing eight graphite spheres fueled with coated particles and one ORR capsule containing coated particles were disassembled and examined in detail. One meltdown experiment was assembled with preirradiated UO_2 and closed by welding in the hot cell; after meltdown in the reactor, the experiment was disassembled and examined. Examination of TRU target rod No. 2 was begun; it was removed from the flow holder, visually examined and photographed, gamma scanned, and dimensional measurements made.

Two ORR poolside boxes containing tensile and bend test specimens were disassembled; specimens were recovered from 15 other experiments in the ORR hot cells. About 125 tensile specimens were tested, including nickel, stainless steel, and INOR-8; equipment was installed for tensile testing in a vacuum. Thirty-two SMI-III reactor Izod impact specimens were broken.

The work requested to date on examination of the failed Bonus fuel element was completed. During this period, 11 of the individual fuel rods were gamma scanned, dimensional measurements were made on all the rods, two rods were disassembled by slitting the cladding, and metallography of the welds, cladding, and fuel was completed.

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PHYSICS AND MATHEMATICS

Theoretical Physics: The Reactions $^{48}Ti(n,d)^{47}Sc$, $^{16}O(n,d)^{15}N$, $^{10}B(n,d)^{9}Be$, and $^{6}Li(n,d)^{5}He$ at 14.4 Mev. – At the Institute "Ruder Bošković" the reactions $^{48}Ti(n,d)^{47}Sc$, $^{16}O(n,d)^{15}N$, $^{10}B(n,d)^{9}Be$, and $^{6}Li(n,d)^{5}He$ have been studied at 14.4 Mev. The absolute differential cross sections have been analyzed by using the distorted-wave method and assuming simple proton pickup. The effects of finite range, nonlocality, and radial cutoff have been investigated. Good fits obtained for the reactions $^{16}O(n,d)^{15}N$ (ground state), $^{10}B(n,d)^{9}Be$ (ground state), and $^{10}B(n,d)^{9}Be$ (2.43 Mev) yield spectroscopic factors in close agreement with shell-model predictions. The shape of the angular distribution of the reaction $^{48}Ti(n,d)^{47}Sc$ is well explained, but a discrepancy in absolute magnitude of about a factor of 4 is not yet understood. The agreement between theory and experiment for the reaction $^{6}Li(n,d)^{5}He$ (ground state) is not satisfactory, and this may indicate the importance of other processes such as knock-on.

Theoretical Physics: A Study of the $^{40}Ca(d,p)^{41}Ca$ (Ground State) Reaction at E_d = 14.3 Mev. – At the University of Pittsburgh the proton polarization from the $^{40}Ca(d,p)^{41}Ca$ (ground state) reaction has been measured in the angular range from 15 to 90° at a mean deuteron energy of 14.3 Mev. Also, the $^{40}Ca(d,d)^{40}Ca$ and the $^{40}Ca(d,p)^{41}Ca$ (ground state) differential cross sections were determined at the same bombarding energy and within the same angular range. A preliminary analysis of the data in terms of the distorted-wave Born approximation theory has been presented. Most calculations were performed in the zero-range approximation with local optical potentials, but including the effects of nonlocality in the approximation of Perey. The inclusion of finite range improved the fit to the (d,p) cross sections but had little effect on the polarization. Although a good fit to the polarization data has not been obtained, it is clear that a spin-orbit term is needed in the deuteron optical potential, and furthermore, the indications are that the imaginary part of the spin-orbit potential has to be positive or zero.

Theoretical Physics: Analysis of the Scattering of 40-Mev Protons. – Elastic and inelastic scattering of unpolarized 40-Mev protons from ^{12}C, ^{54}Fe, ^{56}Fe, ^{58}Ni, ^{60}Ni, and ^{208}Pb have been analyzed by using the optical-model potential and its collective-model extension in the distorted-wave approximation. Fits to the inelastic scattering from ^{24}Mg and to the elastic scattering from ^{27}Al, ^{65}Cu, Sn, and ^{181}Ta also have been included. The Woods-Saxon shape parameters for the potential differ from those suggested for scattering at lower energies, and either surface or volume forms of the imaginary part produce good agreement with the data. To best describe the inelastic scattering, it is found necessary to deform the imaginary (as well as the real) part of the central potential. The deformabilities obtained are in reasonable agreement with those found by other methods.

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Theoretical Physics: Hartree-Fock Calculations of Finite Nuclei. — Hartree-Fock calculations are being performed in a truncated harmonic oscillator representation. Since one aim of the work is to examine the convergence of the results as one increases the dimensionality of the representation, it has been necessary to develop a technique capable of calculating oscillator transformation brackets [Nucl. Phys. 13, 104 (1959)] for high radial quantum numbers. This task is simplified by the fact that only relative ℓ's of 0, 1, and 2 contribute appreciably to nuclear matrix elements. The two-body interactions used so far act only in relative s states. Two types are being studied: (1) separable potential, and (2) local potential with velocity-dependent repulsion. Both give the correct energy and density for nuclear matter and have small second-order corrections. Coulomb repulsion has not yet been included. The variation with A of the single-particle levels has been calculated, and various properties of the single-particle potential and wave functions have been determined. The convergence with dimensionality is very rapid, except for the tail of the wave functions.

Low-Energy Neutron Time-of-Flight Spectrometry. — Neutron capture cross-section measurements were made on natural tungsten samples and enriched samples of ^{182}W, ^{183}W, ^{184}W, and ^{186}W with the 1.25-m-diam liquid scintillator detector at the Rensselaer electron linear accelerator. A neutron resolution of ~5 nsec/m was achieved at the higher energies. In the energy region up to 4 kev, 66 resonances were detected in ^{182}W, 47 resonances in ^{184}W, and 45 resonances in ^{186}W; 116 resonances were detected in ^{183}W up to 3 kev. The data have been reduced to neutron capture cross sections, and the integrated capture area for each resonance has been obtained. The results of transmission measurements of the tungsten isotopes have been combined with the capture measurements to determine resonance parameters; Monte Carlo calculations have been carried out, where necessary, to correct for multiple scattering.

Low-Temperature, Nuclear and Solid-State Physics. — Using the Mössbauer effect, the hyperfine structure (hfs) spectrum of the ^{197}Au nucleus has been measured for Au-Fe alloys containing 0.50 and 1.00 at. % gold. The experiments were performed at 4.2°K using a source of ^{197}Pt which decays through the 77.3-kev state of ^{197}Au. The Au-Fe absorbers contained 100 mg/cm^2 of gold. The positions of the six lines of the hfs spectrum could be determined with good precision by a least-squares method. Using a ground-state moment $\mu = 0.1439$ nuclear magneton, we were able to obtain an excited moment $\mu^* = 0.402 \pm 0.025$ nuclear magneton and a value for the effective magnetic field $|H| = (1.29 \pm 0.03) \times 10^6$ oersteds at the gold nucleus. The excited-state splitting $|2\mu^* H| = 12.68 \pm 0.15$ nuclear magnetons/sec. These results are in agreement with previous measurements [Phys. Rev. 129, 664 (1963); Phys. Rev. 133, A1062 (1964)] but are of higher precision.

Compilation of Charged-Particle Cross Sections. — The second section of a compilation summarizing nuclear cross sections for charged-particle-induced reactions has been published in ORNL-CPX-2. This report includes experimental cross-section data available in the literature before January 1964 for the nuclei Ni and Cu.

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High-Voltage Experimental Program: Low-Lying Levels of ^{75}As. — Coulomb excitation of ^{75}As has been effected with 3.5-8.1 Mev alpha particles and 36-38 Mev oxygen ions. The resulting gamma rays were measured with NaI crystals and a lithium-drifted germanium detector. From the gamma-ray yields as determined from singles and gamma-gamma coincidence spectra and from gamma-ray angular distributions, information has been deduced about the properties of 7 levels and 13 transitions deexciting them. The energies in kev, the $B(E2)_{ex}$'s in units of $e^2\ cm^4 \times 10^{-50}$, and the spins and parities established for these levels are: 198.6 ± 0.1, 1.61 ± 0.14, $1/2^-$; 264.6 ± 0.3, 0.54 ± 0.07, $3/2^-$; 279.6 ± 0.3, 4.5 ± 0.4, $5/2^-$; 468.9 ± 0.7, 0.35 ± 0.03, $(1/2)^-$; 572.2 ± 0.3, 7.2 ± 0.7, $5/2^-$; 616.6 ± 0.8, 0.116 ± 0.019, $(1/2, 3/2)^-$; and 821.8 ± 0.4, 10.9 ± 1.0, $7/2^-$. B(M1)'s for the gamma rays ranged from 0.005 to $0.28 \times (e\hbar/2Mc)^2$. Strongly enhanced E2 transitions to the ground state (16 to 29 times the single-particle estimate) were found from a spin 1/2 level, two spin 5/2 levels, and a spin 7/2 level. However, as noted for other nuclei with a ground-state spin of 3/2, the $B(E2)_d$ of a transition from any excited 3/2 state is small.

High-Voltage Experimental Program: Total Cross-Section Measurements of ^{50}V. — The nucleus ^{50}V is an interesting nucleus in a number of respects. It is a neighboring "almost stable" isobar to ^{50}Ti, has a single hole in the $f_{7/2}$ neutron shell, is an odd-odd nucleus, and has the high ground-state spin of 6. It seemed of interest to study the neutron resonance structure of this nucleus. The ^{50}V was in the form of 594 mg of V_2O_5, 44 at. % in ^{50}V, which was fabricated into a sample about 7 mm in diameter and 13 mm long. Transmission measurements were carried out with the ORNL pulsed Van de Graaff over the energy range 2.5 to 60 kev, with energy resolution of about 2%. The following resonance energies were assigned to ^{50}V: 6.3, 6.6, 7.4, 8.5, 9.8, 11.0, 12.0, 15.5, 18.0, 21.0, 23.0, 26.0, 29.5, 34.5, 37.5, 41, 44, 46, 50, and 54 kev. Parameters for these resonances were determined.

CHEMISTRY

Effects of Radiation on Analytical Methods. — The study on the effects of high temperature on the ferrous sulfate dosimeter has yielded the following:

Ferrous sulfate solutions purged with argon gas and sealed in tubes were found to give the same G value (the number of molecules changed by 100 ev of radiation) on irradiation with ^{60}Co gamma rays at 150°C as that obtained with irradiation at room temperature. The G value at both temperatures was 7.5. This solution, therefore, seems to be suitable for chemical dosimetry at 150°C. It is expected that this value will show little change with increasing temperature as long as phase changes are avoided. In this work it has also been observed that the cleansing of glassware with hot perchloric acid followed by steam yields more consistent results than those obtained when such items are cleaned with chromic acid followed by steam.

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A short study has been carried out on the stability of the Karl Fischer reagent (water determination) in a radiation field. Solutions containing, in one case, an excess of this reagent and, in another case, an excess of sample were exposed to various levels of gamma radiation up to ~10^6 r, with no deleterious effects being noted.

Chemical Engineering Research: Stacked-Clone Contactor Development. – Additional mass-transfer and flooding studies were conducted as part of a continuing program to evaluate the operability and effectiveness of the stacked-clone contactor. Fluids of various physical properties and chemical composition were studied.

The system 1 M $NaNO_3$–H_2O–5% tributyl phosphate–Amsco was tested, and flow capacities and mass-transfer efficiencies (using uranyl nitrate) were similar to those obtained with the higher tributyl phosphate (TBP) concentrations (up to 53%) reported previously.

The system 1.25 M Na_2CO_3–38% TBP–Amsco was recently tested for flow capacity. The higher aqueous-phase viscosity (1.3 centipoises) and the lower interfacial tension (6.7 dynes/cm) of this system, compared with those for an $NaNO_3$ system (0.7 centipoise and 11.1 dynes/cm), contributed to the lower throughput of 1.43 liters/min, compared with 3 liters/min for the $NaNO_3$ system (A/O = 3).

Flooding studies with 0.8 M $Al(NO_3)_3$–0.5 M HNO_3–38% TBP–Amsco, which has intermediate properties of aqueous-phase viscosity (1.2 centipoises) and interfacial tension (8.9 dynes/cm), indicated a flow capacity of 1.86 liters/min (A/O = 3).

Flow capacities for a Purex-type flowsheet were 1.63 and 2.18 liters/min for extraction and stripping modes respectively, at normal Purex-process flow ratios. These capacities represent uranium throughput rates of 140 and 95 kg/day.

Separations Chemistry Research. – Four organonitrogen (primary, secondary, and tertiary amines and a quaternary ammonium compound) and four neutral organophosphorus [alkyl phosphate, phosphonates (2), and phosphine oxide] reagents were shown to be capable of separating polonium from bismuth by solvent extraction from strong chloride solutions. Extraction of polonium by the amines was high at all chloride levels tested (1–10 M); extraction by the phosphorus reagents was high at high HCl levels (6–10 M). Continued study of sulfate transfer in the uranyl sulfate–didecylamine sulfate–benzene extraction system (ORNL-3491) showed that sulfate exchange between the uranyl-amine-sulfate complex and the uncomplexed amine sulfate in the organic phase is surprisingly slower than the sulfate transfer between phases. The sulfate transfer conforms to first-order kinetics. Both sulfate transfer rates and associated estimates of the mole ratio of sulfate to uranium transferred confirm that both anion exchange and neutral-species transfer contribute to the uranium extraction.

Tributyl phosphate dissolves water in approximately a 1:1 mole ratio, and there is controversy as to whether or not this reflects the existence of an association species, TBP·H_2O. Recent measurements of the dielectric constant ϵ vs mole fraction of water X_2 at 25° showed a nearly linear increase from $\epsilon = 8.13$ at $X_2 = 0$, to $\epsilon = 10.57$ at $X_2 = 0.51$ (saturation), in contrast to $\epsilon = 78.54$ at $X_2 = 1$, suggesting that the slope might change at $X_2 = 0.5$. Since more water dissolves in TBP at lower temperatures, the measurements were repeated at 2°C. The entire system was placed in a

thermostatically controlled cold room (2 ± 0.5°C) to avoid difficulties from temperature gradients and condensation of moisture. The results, $\epsilon = 8.76$ at $X_2 = 0$, $\epsilon = 11.18$ at $X_2 = 0.5$, and $\epsilon = 11.46$ at $X_2 = 0.53$, confirmed a definite change at $X_2 = 0.5$ and thus indicate that a real (although probably very weakly bound) association species $TBP \cdot H_2O$ does exist.

Volatility Studies: Sorption of PuF_6 and UF_6 by Metal Fluorides. — An apparatus for comparatively evaluating a large number of solids as possible sorbents for PuF_6 sorption and desorption has been assembled and tested with UF_6. The data may be useful in designing systems for the gas-phase separation of PuF_6 from UF_6 and from impurities.

In a test with UF_6 and 23 different sorbents, the sorbents fell into groups of relatively high, intermediate, and low capacities for sorbing UF_6. The high group, in order of capacity, included NaF, CsF, RbF, and KF (0.21 to 0.07 g of uranium per gram of sorbent). The intermediate group contained AlF_3, CaF_2, InF_3, CdF_2, CuF_2, FeF_3, TlF, and ZrF_4 (0.013 to 0.007). The low group contained SnF_4, ThF_4, CoF_3, CeF_4, CrF_3, NiF_2, PbF_2, AgF_2, ZnF_2, BaF_2, and MgF_2 (range below 0.002).

High-Temperature Aqueous Solution Chemistry. — From the electrical conductance measurements of dilute, aqueous sulfuric acid solutions at temperatures up to 800°C and pressures to 4000 atm, both the first dissociation constant of H_2SO_4 (between 400 and 800°C) and the second dissociation constant (up to 300°C) were determined as a function of density (at 0.4 to 0.85 g/cm^3 for K_1 and 0.8 to 1.0 g/cm^3 for K_2). For some of these results, estimates were made of the standard heat of ionization, $\Delta H°$, for the first ionization process between 400 and 800°C at pressures of 1000, 2000, 3000, and 4000 atm.

By using equivalent conductances from both the present conductance studies and the literature, and some transference numbers for low temperature from the literature, limiting ionic conductances of the potassium, sodium, hydrogen, chloride, bisulfate, and sulfate ions in water were estimated to 400°C. These were 455, 440, 945, 520, 415, and 780 cm^2 ohm^{-1} eq^{-1} respectively, at 400°C. The limiting conductances of lithium, ammonium, and hydroxide ions were estimated to 300°C, being 562, 579, and 821 cm^2 ohm^{-1} eq^{-1} respectively, at the highest temperature.

The second dissociation constant of H_2SO_4 was calculated at temperatures from 25 to 350°C through the use of previously determined and new values for the solubility of $CaSO_4$ and its hydrates both in aqueous sulfuric acid solutions and NaCl solutions at temperatures from 25 to 350°C. The constant decreased from 0.0103 at 25°C to 5.0×10^{-7} at 350°C, and compared reasonably well with some literature values to 225°C and with those obtained by the conductance method.

TRANSURANIUM-ELEMENT PROCESSING

Plutonium-Target Fabrication. — Fabrication of the first 31 dummy HFIR target elements to be used by the Reactor Division in flow tests is almost complete. These elements were loaded with aluminum slugs instead

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of the normal PuO_2-Al pellets. In all other respects, these elements were made and inspected by procedures that will be used in making the target elements.

One of the problems we encountered in making the 18 $^{242}PuO_2$-containing elements recently completed was distortion of the target rods during the hydrostatic-collapse fabrication step. It was not uncommon for a rod to exhibit a total indicated readout (TIR) of 1/2 to 1 in. after collapse. They were then straightened manually, an extremely tedious and time-consuming operation. But, in the TRU facility, manual straightening probably cannot be easily done by remote manipulation. The obvious solution appears to lie in the use of commercial tube straighteners. However, the fins on the rod preclude this method.

In an attempt to obtain a more satisfactory solution to this problem, we recently tried to straighten the rods by pulling them. In the experiments, performed on a tensile machine, dummy rods were straightened to within 0.030 in. TIR, with an elongation of about 2% (about 1/2 in. in the 26 in. in which elongation occurs). Dimensional data on three rods straightened this way, obtained from radiographs, showed that the elongation occurred uniformly throughout the pellet column. Currently, a device for pulling the rods is being designed. If experiments indicate that pull-straightening be adopted, it will be necessary to adjust the target-rod hardware to account for the elongation.

Curium Recovery Facility. – Six Martin slugs in which ^{242}Cm had been produced by irradiating ^{241}Am were dissolved during this report period. After the solution was adjusted to meet Clanex feed specifications, it was processed to provide about 3 g of ^{242}Cm. Satisfactory recovery was obtained during the run (CL-4) and exceeded 99% of that contained in the feed. Decontamination factors for ^{95}Zr and ^{103}Ru averaged 1000 and 10 respectively.

The Clanex product, practically free of aluminum, was then converted to meet Tramex feed specifications and processed in run TR-4. It was noted throughout this run that hydrochloric acid was being removed from feed solution at a rate of 0.7 meq/whr (G value $\cong$19). Since the power density of the feed was 14 w/liter, the entire acidity of the feed could be consumed in 24 hr. A temporary expedient – adding acid every 8 hr – permitted processing but increased the run time and created uncertainties about the actual acid concentration of the feed. The continuous gamma-ray scanner indicated the trivalent rare-earth decontamination factor to be in excess of 1800, and 19 for ^{103}Ru. However, the removal of ^{144}Ce from the feed was insignificant, due evidently to oxidation caused by the high power densities. More than 90% of the curium in the feed was recovered. The product solution met all specifications for ionic impurities, but additional processing must be done to remove the cerium.

Cell 3 Operations. – A total of 17.4 g of ^{244}Cm has been purified by LiCl anion exchange, separated from ^{243}Am, and transferred to the Source Fabrication Facility of the Isotopes Division; an additional 1.9 g each of ^{244}Cm and ^{243}Am has been purified. The americium content in the curium product amounted to 0.8%, and radioactive contaminants were reduced to less than 20% of the curium gamma radioactivity. The separated ^{243}Am (17.7 g) was transferred to Building 3508 for further purification and conversion to the oxide.

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Several interesting observations were made during this work. Purified ^{244}Cm solution was stored for as long as six weeks in a glass container prior to shipment. The solution was 2 M in HNO_3, and the radioactivity level was equivalent to about 50 w/liter. During the storage period, about 100 cc of silica accumulated due to alpha-particle attack on the glass. It was possible to filter it out. Also, nitric acid solutions of ^{244}Cm did not evolve noticeable amounts of radiolytic gas; however, gas evolution from hydrochloric acid was copious. Methanol, which does not appear to be effective in suppressing radiolytic gas formation when ionic impurities are present, effectively reduced gas formation in hydrochloric acid that contained pure ^{244}Cm.

METALLURGY AND MATERIALS

Physical Property Studies. — We have extended our thermal conductivity measurements on tungsten having a density 99.8% of theoretical. The conductivity decreased sharply with increasing temperature from 50 to 600°C, then nearly linearly up to 1000°C. The lattice contribution K_L can be described by

$$1/K_L = 2.612 + 2.77 \times 10^{-3}\ T - 1.185 \times 10^{4}\ T^{-2}\ \text{cm (°K) w}^{-1}\ ;$$

this equation becomes nearly linear above 600°C. The coefficient of T gives a value of 318°K for the Debye temperature, in good agreement with values obtained by other means. We have extended our electrical resistivity measurements on tungsten to 1400°C.

To enable us to extend our thermal conductivity measurements to 1400°C, we have completed intercomparison of several thermometers through several thermal cycles to 1400°C. Compared with an optical pyrometer a W–5% Re vs W–26% Re thermocouple showed negligible change, but Pt–10% Rh vs Pt and W vs W–26% Re thermocouples changed 7° at 1400°C. Resistance thermometers of tungsten and W–26% Re were stable after initial 1% decreases in resistivity due to annealing. The resistivity of the W–26% Re alloy approximately obeyed Matthiessen's rule, with a value about 25 microhm-cm larger than that of tungsten.

Sintering Studies. — As we have reported previously (ORNL-3771, p. 29), the creep deformation of thoria is accompanied by the creation of voids. These voids are located only at grain boundaries and are aligned with the direction of the axial compressive stress. A recent analysis of the slip systems in a variety of lattice structures by Groves and Kelly [Phil. Mag. 8, 877 (1963)] has shown that the usual slip system in thoria, $\{100\}\ \langle\bar{1}00\rangle$, yields only three independent slip systems, but at least five are required by the von Mises–Taylor criterion for general shape change of a polycrystalline body without the creation of voids. Because the individual grains of the polycrystal cannot sustain a general shape change when deforming, grain boundary sliding occurs first and then voids open at some of boundaries. We demonstrated the existence of sliding in thoria that is deforming by creep by the separation of the two halves of a scratch drawn across a grain boundary before the creep test.

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Similar results had been reported with UO_2, which, like thoria, has the fluorite lattice structure. These observations indicate that the creep of thoria involves deformation by slip within the grains, in combination with grain boundary sliding. Voids are created at the grain boundaries because sufficient independent slip systems do not exist within the lattice to permit a general shape change of the grains.

CONTROLLED THERMONUCLEAR RESEARCH

The DCX-1 Injection-Accumulation Experiment. — Most of this report period has been spent gathering and assimilating data needed to clarify and expand information for three papers being prepared for publication about the DCX-1. This newly obtained data, while mainly a refinement of old results, has yielded new insights and several new results.

The effect of microinstabilities in a plasma created by gas dissociation has been studied in considerable detail. The density thresholds at which the azimuthal instability appears has been measured to range from 5 to 10×10^5 protons/cm^3 for the fundamental frequency and nearly the same for the next two harmonics. For long confinement times ($\tau > 1$ sec), direct radial loss of protons in time coincidence with the formation of azimuthal charge waves has been observed. At their worst, these instability losses are four times greater than the charge exchange losses. By varying the H_2^+ beam and gas pressure, we have found the density limit for the plasma created in this way to be about 8×10^7 protons/cm^3. This limit seems to be somewhat lower than that found with Lorentz dissociation, and this difference might possibly be due to the difference in the density gradients. The main points to be recognized are that the density limit in both cases is similar and that the plasma exhibits the same loss mechanisms and instabilities. In a low-density plasma we obtained a confinement time of $2\frac{1}{2}$ min, three times longer than the best previously reported. This is mainly due to the attainment of a pressure of 8×10^{-10} torr.

With gas dissociation the instability has been suppressed to the point of removing the instability losses. In the manner mentioned in the last report, we injected so that there were initially no protons on the equilibrium orbit; this disordering method of stabilization allowed the plasma density to be eased upward to 2×10^8 protons/cm^3, the expected density for loss solely by charge exchange. We shall continue to explore this avenue of stabilization with Lorentz dissociation, for then the density permitted in the absence of instability losses is 2×10^9 protons/cm^3, a result of the much higher proton injection rates available with this trapping technique.

For the first time, protons have been trapped by Lorentz dissociation of H_3^+. The trapping fraction for the 400-kev beam injected into the DCX was 2×10^{-5}, a factor of 20 above the gas trapping rate under those same conditions. The plasma created in this way is especially useful for stabilization studies, for it is initially introduced with a large amount of disorder.

No runs were made during the last three weeks of this report period because of a succession of difficulties with the 600-kv power supply.

Properties of Plasmas Created by Electron Cyclotron Heating. — The location of the neutral beam injection into the electron cyclotron plasma

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(ECP) in the EPA facility has been changed during the period of this report. The beam now passes 4 in. from the axis of the mirror field. This change allows particles to be trapped with their guiding centers on the machine axis. The density of trapped particles has increased by more than a factor of 5 with the ECP on, relative to the trapped density with no ECP. The decay time of the trapped particles has also increased by a factor of about 5. This increase in decay time is the result of ionization of the neutral gas. The absence of a larger increase may be due to the presence of fast "Franck-Condon" neutral atoms in the interior of the ECP which cannot be ionized by the plasma. The product of detector current and decay time, which should be proportional to the observed density, is a strong function of the magnetic field. This may either be due to changes in the orbits of the fast ions, or to cold plasma changes due to the resulting shifts in heating zones.

The injection experiment in the EPA facility has temporarily stopped for modifications. The lead wall which surrounds the facility is being modified to provide more convenient access and more room on the west end so that an arc can be run axially through the cavity. Second, a beam pulser is being added so that the 20-kev neutral beam can be pulsed repeatedly to increase the data acquisition rate. Third, new mirror coils will be added soon so as to improve the orbits of the captured 20-kev protons. New cavity end sections are being made to fit the new coils. An rf probe has been added which will be used to attempt to detect ion-cyclotron fundamental and harmonic radiation in the presence of the high-power microwave radiation. End plates are being made which will allow a gas arc to run through the cavity. This will permit studies of the stabilizing effect of the microwave field on the arc flutes. It will also allow the arc to be used to ionize the 20-kev neutrals and to be used as a plasma feed for the electron cyclotron plasma.

An instability which can be induced in the hot-electron plasma in the PTF is being studied. Silicon solar cells have been found to be quite sensitive to energetic electron bombardment and to respond in about 1 μsec when operated in a short-circuit condition. These cells have been used to measure the flux of hot electrons that leave the plasma during an instability. The flux is simultaneously measured at points outside the mirror throats on different field lines. The results indicate that the instability dumps hot electrons out of the plasma along field lines and that the flux is more or less uniform over the plasma. The duration of the instability is around 10 μsec, but there are variations in electron flux during the instability. There is good correlation in time between these variations on the different detectors and also on the output of an x-ray detector. Near the time of peaks in the flux, there are bursts of rf radiation from the plasma with a frequency of around 10 Mc/sec. This frequency is several times higher than ion-cyclotron frequency but is near the precessional frequency of 50-kev electrons in the PTF.

Properties of Plasmas Created by Beam-Plasma Interaction. — Burnout V has operated smoothly during this period, and three observations have been made. (1) Pinhole images of the plasma formed by charge-exchange neutrals from the midplane show that ion orbits pass through the axis as they do in Burnout IV. (2) 92% of the power in the 50-kw reflex electron beam has been accounted for by the temperature rise in the liner

and end-plate cooling water. About 45% of the power appears to be dissipated in the end plates, 38% in the liner end sections, and 9% in the midplane section of the liner. (3) Two bands of radiation are observed and are assumed to be at the ion plasma and electron plasma frequencies. The plasma density calculated from each of the two observations is in agreement and was found to be about 5×10^{12} cm^{-3}.

The mode II electron heating experiment has been installed in the Beam Plasma Facility with the purpose of creating a hot electron blanket. With the discharge electrodes mounted in the mirror coils, 8 in. off axis, a plasma annulus (hydrogen discharge) has been observed with x-ray energy maximums greater than 150 kv. Preliminary results show that the plasma annulus originates at the plasma column and drifts azimuthally in the direction of electron drift in the magnetic field gradient. The annulus extends axially from mirror to mirror, having a maximum diameter of 24 in. in the median plane and a visual thickness of approximately $1\frac{1}{2}$ in.

Ion Source and Ion Beam Technology. — Development of an ion source for intense ion beams is continuing with the 100-kv test stand. A total hydrogen ion beam of 900 ma at 70 kv has been obtained. However, only 50% of this beam could be focused through a $1\frac{1}{4}$-in. aperture located 26 in. below the solenoid lens. Earlier studies have indicated that a source of the type being used should produce about 80% H^+. A study was made of the ion extraction region to determine what geometry would yield a beam from which the solenoid lens could focus all of the H^+ ions through the $1\frac{1}{4}$-in. aperture. The result of this work has been a more concentrated, less divergent beam at less total current. For example, 80% (all of the expected H^+ component) of a 400-ma 60-kv beam and 75% of a 500-ma beam can be focused through the $1\frac{1}{4}$-in. aperture. The ion source uses an auxiliary magnetic field located around an expanded anode "cup" on a duoplasmatron ion source.

Theoretical Plasma Physics. — We have found that, for practical purposes, plasma confinement by magnetic wells not only avoids flute instability, as originally intended, but also radial "pumpout." We have shown previously that diffusion by high-frequency microinstabilities (greater than ion gyrofrequency) will be tolerable in a device of reactor size. The latter can cause "stochastic" diffusion analogous to collisional diffusion but anomalously large because bunched charges scatter collectively rather than two at the time. The rate is therefore primarily a matter of bunch size, or equivalently, electric-field magnitude. We showed that, in a large plasma close to thermal equilibrium, the available energy to drive field growth is tolerably small. However, even with weak fields, there remained the possibility of pumpout, that is, rapid diffusion by $E \times B$ transport, if there exist low-frequency instabilities with large radial wavelength. We have now shown thermodynamically that the radial wavelength is limited to about an ion gyroradius, ρ_i, in a magnetic well of sufficient depth, ΔB, relative to the central field value, B. The requirement is $\Delta B/B > T_e/T_i$, where T_e, T_i are electron and ion temperatures. If the magnetic gradient is positive (a well) only on the average, there is an additional requirement that $L/R < (T_i/T_e)^{1/2}$, where L is the length along $\vec{B}$ over which the gradient changes sign, and R is the plasma radius. Under the above conditions, the radial diffusion coefficient for

low-frequency E × B transport should satisfy the inequality

$$D_{\perp} < (cT_i/eB)\ (\rho_i/R)\ (T_e/T_i + m_e/m_i)^{1/2} ,$$

where $m_{e,i}$ are masses. Diffusion is tolerable, $D_{\perp} < 0.001(cT_i/eB)$, for $R \leq 1$ to 2 m and ratios T_e/T_i typical with mirror confinement. We have assumed low but finite β. If the above is correct, the major remaining threat to mirror confinement is end loss by high-frequency anomalous scattering. The latter has been observed in the DCX-1.

BIOLOGY AND MEDICINE

SOMATIC EFFECTS OF RADIATION

Statistical Services and Research: Mortality from Secondary Disease. — Preliminary results are available from the fourth in a series of experiments designed to determine the possible interaction of variables known or suspected in the past to have an influence on mortality from secondary disease. The levels of bone-marrow dose, day of injection, age of donor, and sex used in this series result in a noticeable reduction in the proportion of mice dying from secondary disease after having had homologous bone-marrow transplant. In fact, the mortality percentage at 90 days for mice having had homologous transplants is equivalent to the response at 90 days for isologous transplants. Another remarkable result is that not a single mouse treated under the conditions specified in this design died of radiation death, that is, within ten days after exposure to 950 r of x rays. This experiment is being replicated so that the present results might be substantiated and strengthened.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — A large body of information exists pertaining to the kinetics of antibody formation. This information mainly concerns the immunologic variables in terms of circulating antibodies, whereas relatively little is known about the cells or cellular events involved in an immune response. Histologic study has shown the presence of large blast cells with very basophilic cytoplasm to be a constant feature of the early stages of the reaction to foreign erythrocytes. Cytoplasmic basophilia observed in the light microscope does not enable one to distinguish the fine structure of cytoplasm. High-resolution electron microscopy allows more precise definition of cellular components, and basophilic cytoplasm can be observed to vary in ultrastructural organization.

An electron microscopic study of the large blast cells seen in the white pulp of the spleen during the first 48 hr of the response to foreign erythrocytes indicates several differences in their fine structure. Many

blast cells observed 24 hr after antigen injection are characterized by their preponderance of ribosomes which do not appear to be highly organized into clusters (polysomes). These cells also have large, swollen-appearing mitochondria with poorly developed cristae and a small Golgi complex. Nucleoli are hypertrophied in these large cells. Blast cells with cytoplasm containing an extensive organization of ribosomes into clusters or polysomes are numerous 48 hr after stimulation, although they are also present at earlier intervals of the response. Variations in the amount and distribution of rough-surfaced endoplasmic reticulum are encountered in these large blast cells. At present, it is not known whether these differences reflect different cell types responding to antigen, different pathways of differentiation, or merely stages in the development of a single family of cells culminating in a specialized antibody-producing cell. The type of morphologic information presented must in any event be integrated with evidence obtained by other methods in order to form the basis for a clear understanding of an immune response.

MOLECULAR- AND CELLULAR-LEVEL STUDIES

Biophysics: Free-Radical Measurements in Seeds. — As an introduction to possible correlations between free radicals vs chromosome aberrations, germination, and seedling height, preliminary electron spin resonance (ESR) measurements have been made of free radicals, both naturally present and induced by ionizing radiation, in the embryonic root tips of *Vicia faba* and in the whole seeds of wheat and the New York and Grand Rapids varieties of lettuce. In the unirradiated state of all these materials, a low-amplitude, broad (470 gauss), six-peak resonance system is found which is probably from Mn^{2+}. Narrow signals at $g \cong 2.004$ are found in the three whole seeds. The dark Grand Rapids lettuce seed has a strong, narrow (6-gauss) resonance. Exposure to, say, 200 kr of x or gamma rays produces strong, narrow resonances in *Vicia* and both lettuce species. Recently developed ESR difference techniques will be essential to refinement of these studies.

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