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REACTOR & MATERIALS TECHNOLOGY MONTHLY REPORT
FEBRUARY 1965

F. W. Albaugh

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REACTOR AND MATERIALS TECHNOLOGY DEPARTMENT
MONTHLY REPORT - FEBRUARY 1965

Compiled by Members of the
Reactor and Materials Technology Department
PACIFIC NORTHWEST LABORATORY

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REACTOR AND MATERIALS TECHNOLOGY DEPARTMENT
MONTHLY REPORT - FEBRUARY 1965

TECHNICAL ACTIVITIES

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A. FISSIONABLE MATERIALS - 02 PROGRAM

1. Metallic Fuel Development

Alternate Uranium Composition. The development of improved metallic uranium fuel involves in- and ex-reactor studies of a number of additive and alloy compositions.

Four uranium compositions, fabricated to N-single tube geometry, were obtained for heat treatment and ex-reactor property evaluation. The compositions are: (1) standard 150 Fe-100 Si, (2) 350 Fe-800 Al, (3) 250 Fe-250 Si-250 Al, and (4) 250 Fe-350 Si. Fuel element lengths were beta and gamma heat treated; both treatments being the salt-salt process. Samples are being processed for microstructure examination, hot hardness and mechanical property testing, and recrystallization behavior. Specimens for recrystallization study were cold worked 22% by swaging and are being annealed. Hardness tests at 300 C were completed. Alloys (2) and (4) are hardest and essentially the same, while the standard composition (1) is the softest. Gamma treated material in each case is slightly harder.

Three Zr-2 clad rod coextrusions were made with uranium shot containing (1) 60 ppm carbon, (2) 550 ppm carbon, and (3) 500 ppm carbon and 1130 ppm phosphorus. Sections of this material were beta heat treated and the microstructure examined to characterize the type of dispersion of UC or UP present. No agglomeration of the compounds was noted by optical microscopy. Tensile properties were determined to 600 C in both the as-extruded and beta heat treated conditions. No marked difference in strength was found due to either the UC or UC+ UP dispersion (approximately 1.5 and 3.5 vol% dispersed phase, respectively). The tensile properties of these materials were comparable to normal uranium compositions in the cast, wrought, and beta heat treated condition except that the ductility at 500-600 C was reduced.

Studies are also in progress to evaluate uranium base alloys in terms of composition, fuel fabrication history, corrosion behavior, and irradiation performance. Alloys being studied include uranium with 2 wt% Zr, Mo, or Cb and up to 800 ppm Al or Si as well as uranium with Fe, Si, Al, P, or C additions of up to 800 ppm. Assembly of 40 irradiation test capsules, containing three fuel samples each, and five temperature monitoring capsules with one fuel sample each has been completed. The finished assemblies were shipped to B-Reactor and were charged on March 1. These fuel samples will be irradiated to approximately 3000 Mwd/ton at a volume average uranium temperature of 550 C.

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Comparative Swelling of Uranium Alloy Fuels. Samples of irradiated fuels from KSE-5 fuel elements with 0.31 at% B.U. (2700 Mwd/ton) have been postirradiation annealed and studied to obtain further information on the influence of alloy additives on swelling behavior. One sample contained 150 ppm Fe and 100 ppm Si (N-Reactor fuel), and another contained 400 ppm Fe and 800 ppm Al. Volume changes during annealing were as follows:

Fuel	Percent Volume Increase			
	As Irrad.	100 Hrs @ 625 C	100 Hrs @ 700 C	Total
With Fe & Si	2.2	1.1	1.7	4.9
With Fe & Al	1.2	0.7	-0.3	1.6

Postirradiation metallography showed both grain boundary tearing and "aligned porosity" in the Fe and Si fuel, while the Fe and Al fuel showed only slight grain boundary tearing and no aligned porosity. After the 625 C anneal, a coalescence of both types of porosity was evident in the Fe-Si additive fuel, but little significant change occurred in the Fe and Al fuel. After the second anneal (at 700 C), spherical porosity was observed in both the grains and grain boundaries of the Fe-Si fuel. This porosity is believed to result from coalescence of fission gases. In the Fe and Al fuel there was a small amount of porosity developed at grain boundaries. Both the metallographic observations and the density data indicate that fission gas mobility is inhibited to a greater extent in the Fe-Al fuel than in the Fe-Si additive fuel.

Evaluation of KER Irradiated Co-Product Drivers. A review of KER-3 operating data indicates that from the standpoint of stable prototype operating conditions the co-producer test was one of the most successful. The loop outlet temperature was maintained nearly constant at 270 C with the ΔT maintained between 59 and 63 C. An average specific power of 105.8 kw/ft of uranium was maintained within less than 7%. Based upon "weasel" data for the charge, the maximum fuel element exposure was 2220 Mwd/ton, and the maximum specific power was 184.8 kw/ft of uranium. In the four central elements of the test, in which fuel swelling up to 3.3 vol% was observed, maximum fuel operating temperatures ranged from 500 C to 555 C.

Length measurements made on two of the co-producer element indicate that no significant length changes occurred during irradiation.

Target Element Development. A special study was started during February to determine the effects of a high temperature reactor excursion on Zr-2 clad Al-Li alloy target elements.

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Examination of the three irradiated targets which were subjected to heating tests is under way. All three elements failed during heating, and the molten target alloy was released into the surrounding annulus. Although the examination is not complete, all evidence indicates that the elements failed due to the high internal pressures generated by the helium and tritium released from the target alloy during heating and melting.

A 1/4 to 3/8-inch-long split in the cladding of one element has been examined, and high ductility in the Zr-2 cladding at the point of failure is evident. It is estimated that at least one-half of the molten core was discharged through the split at the time of failure. Metallographic examination of sections taken at the cladding split and near the center of the element shows a compound layer of Zr and Al on the inside surface of the Zr-2 cladding. However, from 23 to 30 mils of the original 30-mil-thick cladding remained unreacted.

Zr-2 tubes, 0.565-inch diameter by 0.020-inch wall thickness, were pressure tested at 800 C and 930 C to determine the rupture strength of the Zr-2 target element cladding at high temperature. After reaching the test temperatures on two specimens, the pressure was increased until failure occurred. In three tests, the pressure was applied rapidly. All tubes showed extensive elongation of the Zr-2 at the point of failure, and all failures occurred in a 2-inch length between the end caps and supports. Failure occurred at 700 to 1000 psi for tests at 800 C and near 125 psi at 930 C. The low rupture pressures observed in these tests are well below the pressures which could be generated within the irradiated target elements during heating.

Tritium Evolution from Aluminum-Clad Target Elements. A pair of irradiated, aluminum-clad target elements -- one anodized and one nonanodized -- were heated in a vacuum furnace at 300 and 350 C to (a) determine tritium species adsorbed on the target surface, (b) measure the rate of tritium evolution through the cladding at 350 C, and (c) see if the anodized coating retarded tritium evolution. The target elements, irradiated in KER loops, contained an aluminum-0.8 wt% enriched lithium (40 wt% Li⁶) alloy. The alloy contained 15 volumes gas per volume metal. Irradiation time was 61.2 days (26.2 days at 40 C and 35 days at 280 C). Total tritium in the elements (corrected for tritium decay) was 145 cc, STP. No tritiated water appeared in either outgassing run (1 hr at 300 C). A trace of HT (0.04 mole%) appeared in the 300 C run on the anodized element, but was barely above the detection threshold (0.02%). Tritium evolution at 350 C appeared predominantly as HT. Differences in average tritium evolution rate from the two claddings were not significant.

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Protective Coatings for Zirconium. Flame sprayed coatings on Zircaloy-2 are being studied for their possible effect in protecting the alloy from attack by high temperature molten aluminum. Nearly all available materials which can be flame sprayed have been used in the first rapid survey of coatings. Some materials, though unlikely to be protective in themselves, may form intermetallic compounds with either the zirconium or the aluminum which may offer some degree of protection. The coatings thus far applied have been: Al_2O_3 , ZrO_2 , tungsten carbide, an aluminum-iron bronze, molybdenum, tantalum, aluminum, iron, copper, and nickel. SiO_2 and niobium will be applied if forms can be found suitable for flame spraying.

Refractory Target Materials. Small quantities of pure Li_2SiO_3 were prepared by reacting stoichiometric ratios of Li_2CO_3 and SiO_2 powders at 1100 and 1250 C. Respective densities were 83 and 98% theoretical.

Pressed and sintered pellets of LiAlO_2 (75% theoretical density) were crudely crushed and loaded into cylindrical tubes by vibrational compaction to 62.5% theoretical density.

Be-Zr Braze Wire for N-Fuels. Zr - 5 wt% Be brazing wire was successfully extruded to 0.025 and 0.035-inch diameter. The fabrication involved successive extrusions of Be-Zr alloy rods clad with stainless steel and vacuum sealed in mild steel billets. Final extrusions at 800 C resulted in 49 wires/rod of uniform diameter. The extrusion constants were 22 tons/in² for ingot breakdown and 28-32 tons/in² for reducing the composite billets.

N-Reactor Fuel Support Development. The mechanical test series on co-producer and tube-tube fuel inner supports continued. Fatigue data are being developed for 1/4-, 3/16-, and 1/8-inch-wide inner supports. Target fuel support assembly tests specimens are being fabricated.

2. Corrosion and Water Quality Studies

Boridizing Steel Chain. It has been suggested that mild steel chain, suitably treated to make it "black" to neutrons, be dropped into existing reactors for reactivity control during down-time.

In an effort to produce mild-steel chain black to neutrons, chain segments were pack-boridized both in commercial Ferroboration and B_4C powder. The best results were obtained with Ferroboration, using a nine-hour exposure at 1160 C. Chain so treated exhibited 90 to 100% of the neutron blackness of 1% boron-steel balls in reactor tests. The chain exhibited no high-boron phase on the surface of the steel which could be abraded off and lost in the reactor.

Improved Inhibitors for Reactor Coolant - IPD. Sodium nitrite-silicate mixtures are being evaluated as an alternative corrosion inhibitor to sodium dichromate in once-through cooled reactors. This

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inhibitor system is more economical and contains no chromium which can produce Cr^{51} activity and Cr^{VI} compounds in the reactor effluent.

An in-reactor test in the 1706 KE single-pass facilities using 1 ppm NaNO_2 and 1 ppm Na_2SiO_3 at typical temperatures and flow rates was completed. After five months of testing, the coupons and fuel elements showed corrosion rates for aluminum similar to those in water inhibited by dichromate, while corrosion rates for carbon steel were higher. The aluminum coupons corroded in both types of water at a rate of $\sim 1 \times 10^{-3}$ inches/year; the corrosion rates of the aluminum jackets on the fuel elements were unmeasurable, indicating a good protective oxide film. The corrosion rate of carbon steel in the nitrite-silicate water was $\sim 3.0 \times 10^{-3}$ inches/year as compared to 0.4×10^{-3} inches/year in the dichromate-inhibited coolant.

Effect of O_2 in N-Reactor Graphite Cooling System. A one-month ex-reactor test to determine the corrosion effects of high oxygen concentrations (40 ppm) in water at N-Reactor graphite cooling system conditions has been completed. Severe pitting was found on the carbon steel and light pitting on some of the 400 series stainless steels. There was no stress-corrosion cracking of any of the alloys, including 17-4PH in the H900 condition stressed to yield. This alloy was found to be very susceptible to cracking in similar-quality coolant at 550 F, however.

Decontamination of N-Reactor Steam Generator Tubing. The N-Reactor primary system steam generators are scheduled to be decontaminated in April 1965, using the proposed two-step APSUL procedure (alkaline permanganate followed by inhibited sulfamic acid). Tests were performed during the month to evaluate potentially less-corrosive (HV) or simpler (one-step) procedures.

The HV procedure utilizes a solution of ammoniated hydrazine and versene (EDTA) solution. In the laboratory, a sample of the steam generator tubing was immersed in the HV solution for 22 hours at 95 C. The decontamination factor was so low (~ 2.0) that this reagent was eliminated from further consideration.

A one-step proprietary reagent (Wyandotte 5231-1) was evaluated using steam generator tubing. Decontamination factors were very good (~ 500), but the solution formed a precipitate after about 20 hours at the use temperature (100 C). This precipitate may limit its value for the steam generator decontamination.

Corrosion of Stressed Zircaloy-2 in Ammoniated Coolant. Examination of a failed Zr-2 sample holder irradiated in the MTR (KAPL 3029) showed both excessive hydriding near the fracture areas and localized subsurface oxidation. The cause of the hydriding has not been defined, but important parameters appear to be exposure temperature (< 475 F) and time, a possible effect of NH_4OH , surface damage from handling,

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and environmental factors such as neutron flux or stray currents present in the loop.

An in-reactor test was performed in the K-1 Loop in K-Reactor to determine if coolant conditions employed at N-Reactor would similarly attack cold-worked Zr-2. Samples with various combinations of peening, shearing, and drilling and in both etched and unetched conditions were exposed in- and out-of-flux for two months in pH 10 ammoniated coolant at temperatures below 475 F. Weight measurements and metallographic examination of the specimens failed to reveal any abnormal oxidation or hydriding.

Corrosion of Copper Alloys by Ammoniated Water. The ammoniated coolant adopted as standard for the N-Reactor primary system appears satisfactory from a general corrosion standpoint, but escaping ammoniated water or vapors may accelerate corrosion of copper alloys located internally or externally to the system. A series of tests is being run to evaluate conditions which might lead to corrosion in N-Reactor components.

Two valves were exposed in a loop, operating with pH 10 ammoniated water at 530 F and 1100 psi, for five and eight months, respectively. The packings were loosened to allow leaking ammoniated steam to impinge on the ASTM B132 and B138 manganese bronze bushings and flanges. Fluorescent penetrant and microscopic examination following the test did not reveal any ammonia stress-corrosion cracking. The Teflon valve packing was in excellent condition. The silver plating on the retaining rings was completely removed at all locations in contact with the ammoniated water.

Stressed, corrosion samples of seven additional brass alloys were placed in N-Reactor piping chambers to monitor ammonia stress-cracking corrosion. Although several ASTM B147 samples cracked during earlier tests, the nonrecurrence of this cracking under the present test indicated improved control of ammoniated water leakage from the primary system.

3. Gas-Atmosphere Studies

N-Reactor Graphite Burnout Monitoring. The first set of burnout monitors at N-Reactor has been examined. This set consisted of alternating samples of TSX and TSGBF graphite placed end-to-end down the entire length of the reactor core.

The monitoring facility, Process Tube Channel 1648, was uncooled and unvented. The only gas reaching the burnout monitors diffused through the process tube block from the 2 by 3-inch gas passages. Burnout of the moderator by O₂ oxidation would not be detected because the O₂ would be depleted before it could diffuse through the process tube

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block. After the first set of monitors was discharged, this condition was corrected by drilling several 1-inch diameter holes at each end of the monitoring facility; these holes were located in the front and rear gas plenum chambers and will permit a flow of reactor gas through the monitoring channel in future irradiations.

A profile of the burnout rates from the first set of monitors showed a dome-shaped curve with a peak of approximately 0.2% per 1000 operating days located three feet upstream from the reactor-core centerline. It was found that the TSGBF material was approximately 1.5 times more reactive than TSX. The reactor was not operating at full power during the entire monitoring period from July 9, 1964 to January 10, 1965, and the temperature of the monitors was in the range from 500 to 625 C.

4. Process Tube Development

Postirradiation Examination of a C-Overbore Tube. A Zircaloy-2, C-overbore process tube was removed from C-Reactor after two years use. General Electric Company, IPD, requested that this tube be examined for irradiation effects. Two small pieces from the rear-face end of the tube and one three-foot piece from the center of the tube were sent to Radiometallurgy for this purpose. Borescope examinations indicated only superficial scratches on the interior of the tube. No effects of reactor exposure on structure or grain size were observed. Some small areas of localized hydriding were seen on the interior surface of the tube, but this could not be attributed to irradiation. Ring tensile tests indicated an increase of approximately 14% in ultimate strength due to irradiation. A BNWC report on the results of the postirradiation examination is being prepared.

Fracture Studies. Acoustical methods of detecting crack initiation and propagation are being investigated as a possible tool in controlling the fatigue growth of cracks in test specimens of pressure tubing. Initial work in selecting proper transducers and developing electronic circuitry is being conducted on simplified flat plate specimens to conserve the more scarce and expensive tube sections. The two most promising transducers to date appear to be a solid-state strain gage and an accelerometer. One of each of these was applied to a Zircaloy-2 flat plate specimen and an incrementally growing crack induced by stressing the specimen in a tensile machine. The strain gage consistently produced a stronger acoustical signal than did the accelerometer. The signals produced were quite clean cut sine waves with a frequency of approximately 10,000 cycles per second. It is not yet certain whether this frequency is a typical signature of the fracture of this particular material or whether it is strongly influenced by the geometry of the specimen.

5. Thermal Hydraulic Studies

Predictions of Pressures in a K-Reactor Downcomer. An analysis was started to determine the pressures in the K-Reactor downcomer during a temperature increase of the reactor coolant. Such an event could occur during an unscheduled power surge and possibly cause damage due to overpressurization of the downcomer with boiling of the bulk coolant.

Four cases were considered to determine the rate of water level rise and the time at which boiling occurs. These are as follows:

Case	Temperature Rise	Initial Conditions		
		Near Riser °C	Far Riser °C	Flow gpm
1	0.5 C/sec for 20 sec	97	93	210,000
2	"	93	97	"
3	2 C/sec for 3 sec	97	93	"
4	"	93	97	"

For cases 1 and 3 (near side riser hot), a large amount of overflow occurs before surface boiling occurs, especially for the fast transient. This would cause a possible siphon effect with pressure oscillations of unknown magnitude. Even if the overflow did not cause a siphon effect, the hot flow from the near side stream would begin to flash and overflow part of its stream as flashing, two-phase water. This part of the transient is believed to have the largest unknown effect.

For cases 2 and 4 (far side riser hot), no boiling occurs in the approach section until saturated water from the far side stream arrives. Boiling also approximately coincides with the beginning of overflow. A significant siphon effect would not be expected for these cases. It is difficult to determine maximum pressure, since a two-phase mixture would be discharged through the overflow and possibly part of the downcomer elbow.

It is planned to continue the analysis to improve pressure estimates during boiling and to test further the assumptions used.

Hydraulic Tests for the Present Production Reactors. A series of tests were performed to determine the hydraulic characteristics of various modifications contemplated for the existing reactors.

Pressure drop tests were performed to determine the number of solid aluminum dummy pieces required to throttle tube flow for arch-rail fuel charges to that for a bridge-rail charge. Data were also

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gathered to determine the effects on Panellit pressure and tube flow when the solid elements were placed in various positions in the downstream dummy pattern. The results of the tests were reported in BNCC-16, "Pressure Drop Characteristics of Solid Aluminum Dummy Patterns in K-Reactor, Zircaloy-2 Process Tube."

The hydraulic characteristics of new overbore fuel elements in the C-overbore process tube were determined. These elements, identified as CVIAN I&E's, are hot die sized to 1.975-inch OD and 0.350-inch ID--a 10-mil increase in annular and hole dimensions over the standard CVIN elements. Results of the tests are given in BNCC-8, "Hydraulic Characteristics of CVIAN I&E Fuel in the C-Overbore Process Tube."

Flow tests were conducted in the laboratory to find the pressure differential and flow associated with two "mixed" charges of natural uranium, bismuth, and flux monitors in the C-overbore tube. The results are reported in BNCC-13.

6. Graphite Studies

K-Reactor Hot-Test-Hole Temperatures. Five thermocouple boats of two designs were placed in the hot test holes of the K Reactors. One design provides information on the temperature gradient across a typical boat loading and across a reactor-size bar. Four boats of the second design give temperatures at the center of a boat loading for the respective boat positions in the reactor.

Complete analysis of the thermocouple data has not been made; however, some generalizations may be made at this time.

- (1) All graphite samples are presently being irradiated in the temperature range 500 C to 700 C.
- (2) Each boat position of each test hole has a range of ± 50 C unless the reactor loading is changed appreciably.
- (3) All samples within a boat lie in a range of ± 7 C.
- (4) The temperature variation in a reactor-size bar will be in a range of ± 10 C.

N-Reactor Flux Monitoring. Samples for dimensional monitoring experiments in Rod Channel 74 at N-Reactor have been fabricated, assembled, and will be charged in the next two or three months.

Two, 24-inch long flux boats were charged into the rod channel. The flux-monitors consist of Fe, Ni, and Co-Al wires at nine positions in each boat. They will remain in the reactor for two or three months.

N-Reactor Irradiations. Irradiations of samples of N-Reactor graphite in the GETR continue to progress satisfactorily. Two, fifth-generation capsules, H-4-5 and H-5-5, have successfully completed four and two cycles of operation, respectively. The maximum neutron exposure on the long-term samples is estimated to be approximately 7.5×10^{21} nvt, $E > 0.18$ Mev. Construction of the final, fifth-generation capsule, H-6-5, is completed, and the capsule will be installed in the GETR in early March.

During the GETR shutdown the first week in February, a test run was made of a revised core loading called the "checkerboard" arrangement. The test, consisting of 8-hour operation at 30 MW, depressed the H-4-5 and H-5-5 sample temperatures to 125 C below operating conditions. Consequently, the core arrangement was returned to the regular pattern for the startup of the present cycle. A fuel element was installed adjacent to the capsules, since the physics calculations showed a decrease in flux with the pressurized water loop removed. Upon startup, however, the H-4-5 and H-5-5 sample temperatures were on the maximum operating limits, restricting reactor power levels to less than 30 MW. Consequently, the reactor was shut down after 35 hours of operation, and the fuel element replaced with an isotope capsule and filler piece. Since subsequent startup, both sample temperatures and capsule operation are satisfactory.

B. WEAPONS - 03 PROGRAM

Research and development in the field of plutonium metallurgy continued in support of the Hanford 234-5 Building operations and weapons development programs of the University of California Lawrence Radiation Laboratory (Project Whitney). Details of these activities are reported separately via distribution lists appropriate to weapons development work.

C. REACTOR DEVELOPMENT - 04 PROGRAM

1. Plutonium Recycle Program

Fuels Development

PRTR High Power Density Core Elements. The maximum burnup of the four UO_2 -2 wt% PuO_2 high-power-density elements in PRTR is 2010 Mwd/ton_U. This burnup of the vibrationally-compacted elements was accumulated at a tube power of 1350 Kw (equivalent to a rod power of 19 Kw/ft), which produced maximum fuel temperatures of approximately 2600 C.

Flux peaking in the ends of these elements caused high fuel temperatures near the end caps. Localized fuel melting occurred near the bottom ends of rods of the first high-power-density elements (FE-6000) irradiated in PRTR at a tube power of 1350 Kw. High-power-density elements are now loaded with graded enrichment in the ends of the rods to avoid high fuel temperatures adjacent to the end caps. The top end is loaded with 1-cm of natural UO_2 , followed by 1-cm of UO_2 -1 wt% PuO_2 , followed by the UO_2 -2 wt% PuO_2 fuel material. The bottom end, which has more severe flux peaking, is loaded with 1-cm of natural UO_2 , followed by 3-cm of UO_2 -1 wt% PuO_2 , followed by the UO_2 -2 wt% PuO_2 fuel material. This loading scheme should cause maximum fuel temperatures to be generated in the midplane of the elements. Four short clusters containing graded enrichment were fabricated during the month.

Fuel Cycle Analysis. A flow sheet was developed for a one-ton per day plant which produces mixed-oxide fuel elements. When the plant design is complete, this information will be incorporated into the Fuel Element Fabrication Code to calculate unit fabrication costs. These costs will be used as a basis for the fuel cycle analysis of several different plutonium enrichment schemes.

Fuel Element Cost Studies. An internal accounting system was established to accumulate labor costs for preparing plutonium fuel elements. The system will be used to generate primary unit data for fuel fabrication cost analyses, incremental cost analyses, forecasting, estimating, planning, and scheduling.

Specification for Nuclear Uranium Dioxide Powder, Pneumatic Impaction Grade. Specifications were published (BNWC-17) to define the chemical and physical requirements of UO_2 powder for densification by pneumatic impaction. The high density material is intended for use in packed-particle nuclear fuel elements made by vibrational compaction, swaging, or similar processes.

Nondestructive Testing. Infrared testing of swaged and vibrationally compacted fuel elements is being evaluated for quality control and prediction of in-reactor performance. The technique, being developed

by the Physical Measurements Unit, involves analysis of the infrared radiation emitted by fuel elements when heated by various methods. The effects of variations in density, thermal conductivity, and continuity (i.e., cracks in cladding or fuel) on thermal diffusivity are indicated by a pulsed heat wave.

Vibrational Compaction Facilities. A small pneumatic vibrator was modified for possible use in compacting small fuel elements having simple configurations (e.g., rods and tubes). Preliminary strain-gage measurements showed lower stresses in EBWR cladding tubes, both empty and filled, than those induced by either the variable impact or the electrodynamic vibrators.

Determination of Bulk Densities of Crushed Impacted Fuel Materials. An instrument was developed for reproducibly determining pour and tap densities of irregularly-shaped particles usually produced by crushing pneumatically-impacted mixed oxides. The instrument, designed and built locally, provides fuel particle data useful for studying vibrational compaction behavior of this type of fuel material.

Test Element with Replaceable Rods - GEH-4-107. Metallographic examination of both a connecting end bolt and end cap of the replaceable-rod test element showed no fretting or chemical corrosion after 15.8 days of operation in the MTR. Photomicrographs of transverse cross-sections showed Zr-hydride concentration to be less than 100 ppm. One of the four rods of the element was remotely replaced, demonstrating the feasibility of this end cap design. A test cluster will be irradiated in PRTR.

Wire Enrichment. Fuel elements containing sintered UO_2 pellets enriched with 85% plutonium-zirconium wire are being prepared for irradiation in the PRTR. The wire and enough cored, UO_2 pellets to assemble the first full-length PRTR fuel rod were fabricated.

Fuel Element Rejuvenation. Mechanical problems caused an unsuccessful attempt to rejuvenate a fuel element with Pu alloy wire. The element, irradiated for one cycle in MTR, was returned to Radiomet for rejuvenation. A carbide bit broke while drilling a pellet in one rod, and galling occurred while parting the end cap in a second rod. A second element will be returned to Hanford for rejuvenation next month.

Heterogeneously Enriched Fuel Element. Failure of the heterogeneously enriched fuel element (GEH-4-108) was due to eccentric loading of the UO_2 -8.4 wt% PuO_2 in the annulus between the UO_2 pellets and the cladding. Examination of this rod indicated that failure after an exposure of 6×10^{18} fissions/cc was of the burnout type. The only fuel washout experienced was a small amount of loose powder exposed by the defect.

PRTR Salt Cycle Fuel Elements. Carbon tetrachloride extraction of "salt cycle" fuel disclosed an organic contaminant identified as Unichrome, a polyvinyl chloride latex used as a corrosion-inhibiting coating. Decomposition of this material during irradiation could cause internal hydriding of the Zircaloy-2 cladding. Equipment causing the contamination was discarded, and fuel rods scheduled for PRTR irradiation were recharged with fresh, salt-cycle fuel.

A single clad capsule containing salt cycle UO_2 -2 wt% PuO_2 was scheduled for irradiation in the MTR core. Maximum power generation of this capsule will be 2.2 Kw/inch and will operate with a peak surface heat flux of 192 watts/cm². End spacers were attached to a bottom section of a PRTR salt cycle rod to insure positioning in a standard S-basket.

PRTR High-Power-Density Core

Design Analysis Studies. Design data developed for the PRTR "chemical shim" soluble poison shim control were updated in accordance with recent reactor physics calculations and were summarized for criteria consideration.

For the proposed eight-month "batch cycle" operation of the PRTR, the desired linear decrease in boron-10 concentration over the eight-month period will require boron addition during the first six months and removal during the last two. Because of the extremely low flow rates required for continuous boron concentration adjustment (maximum rate of 0.09 gal/hr addition, 0.2 gal/hr removal), stepwise rather than continuous adjustment was recommended.

Use of the chemical shim system for scram recovery will require boron removal rates equivalent to about 10 mk/hr reactivity increase. Flow rates in the removal system will vary with boron concentration from about 2-18 gpm. To compensate for subsequent xenon burnout during startup of the reactor, boron injection flow rates up to 50 gpm may be required, actual rates varying with the time the reactor has been subcritical. These values are in substantial agreement with previous calculations.

A review of potential boron migration from the moderator to other reactor systems indicated a low likelihood of such migration except possibly for the reflector system. It was recommended that existing interties between the moderator and reflector systems be eliminated or modified to provide more positive isolation; that the fluid collected in tanks TK-A-1, -3, -4, and -6 should be routinely monitored for boron; that all borated D_2O be stored in unmistakably marked containers; and that investigation be made of the feasibility of slightly pressurizing the reactor reflector vessel to reduce possible in-reactor leakage from the moderator to the reflector.

Thermal Hydraulics Studies. Consideration was given to the potential hazards caused by a jacket failure of a PRTR fuel element operating at a power density where the core would be molten. Hazards arise from large volumes of steam formed should hot (approx. 6000 F), molten $\text{PuO}_2\text{-UO}_2$ ceramic contact the coolant. Local pressures from rapid steam formation might rupture the pressure tube or molten ceramic might contact the pressure tube wall and cause failure.

It was concluded that more information is needed to evaluate the problem. Specific information is needed on the nature of molten ceramic release into the coolant, the heat transfer coefficient between the ceramic and the coolant, the extent and heat of chemical reaction between the ceramic and water, and the heat of fusion of the ceramic fuel.

Corrosion and Water Quality Studies

PRTR Boron Shim Pilot Plant Corrosion Tests. Two tests were conducted in the Boron Shim Pilot Plant. The first test was conducted at 25-50 ppm boron at 140 F for two weeks. The test section coolant was in a static condition (no-flow) for almost the entire test period. Examination of the aluminum sleeve force-fitted against the inconel shim rod control section revealed severe pitting (up to 10 mils deep) on the surface contacting the shim rod. Although most pitting occurred at areas where the sleeve was scratched during the charging operation, some pitting was observed on other areas of the tube. Coupons from the loop indicated no excessive attack.

The second test was conducted under conditions similar to those described above except that a 10 ppm boron solution was used and the shim rod was mounted in a 6061 aluminum well similar to PRTR conditions. The well was bent slightly to insure contact with the shim rod. After 10 days of operation there was no apparent pitting; however, there were several areas where pitting attack had started on the aluminum webbing on the shim rod. Coupons from the loop were similar to those for the first test. The test is being continued under the same conditions for another two-week period.

Studies to determine equilibrium boron deposition values on 6063 aluminum and 304 stainless steel specimens exposed to various boric acid concentrations at 75 F and 140 F under dynamic conditions were completed. The maximum value obtained was 4×10^{-6} gm boron/cm² for 6063 aluminum exposed at 140 F to a 98 ppm boron solution. This value is not significant in terms of a permanent reactivity poisoning effect (~ 1 mk) at PRTR. Maximum deposition on specimens exposed under anticipated Phase I test conditions (140 F and 10 ppm B) was only 2×10^{-6} gm boron/cm². There was no appreciable effect of temperature, surface conditions, or specimen materials during these tests. Equilibrium deposits formed within two days during these tests.

Desorption tests were conducted in deionized water to measure release of boron from specimens exposed in the tests described above. Results indicated that $<1 \times 10^{-6}$ gm boron/cm² was released after four days of exposure to 140 F coolant. It was concluded that the boron deposits essentially irreversibly under these conditions.

Crevice Corrosion Tests on Simulated PRTR High-Power-Density Fuel Elements. An initial test to study potential crevice corrosion on PRTR fuel elements using Zircaloy-2-clad, cartridge heating units was terminated by cartridge failure after 70 days. The test was conducted in pH 10, lithiated water at 293 C and 1350 psig. Microscopic examination of the heating unit surface showed gray-white oxide deposits up to 0.005 inch thick in crevices associated with the "cluster" band. Surface heat flux of the test assembly was 550,000 BTU/hr-ft², which was high enough to initiate local (surface) boiling. Oxide formation is attributed to lithium concentration in the crevices during boiling. This initial test indicates that PRTR High-Power-Density fuel elements should be designed to avoid the concentration of additives by local boiling. Tests are continuing.

PRTR Decontamination Studies. The IRP Loop was decontaminated using the APSUL procedure recommended for decontaminating the PRTR and used successfully to decontaminate an ETR Loop. However, difficulties were encountered during the decontamination of the IRP Loop. Radioactive material, precipitated during the procedure, entrapped the activity. Since this precipitate collected at the filter used to simulate deadlegs and low flow areas, the decontamination factors were very low (<2).

The principal reason for the precipitation and low efficiency appears to be the decomposition on storage of one of the reagents, the proprietary inhibited sulfamic acid. Subsequent to the IRP decontamination, the reagent was tested in the laboratory and was found to be only partially soluble under the conditions used. Apparently some degradation had occurred during the storage (2 years) of this material. The IRP decontamination indicates that it is necessary to determine the effectiveness of decontamination chemicals immediately prior to use.

Corrosion of Aluminum in Boric Acid. The study of aluminum corrosion in dilute boric acid solutions has been required by the proposed use of these solutions in the PRTR moderator system.

The corrosion of type 6061 and 6063 aluminum alloys by dilute boric acid solutions in high-purity water and deuterium oxide at 50 C and 120 C was studied in small static systems. The results indicated a mild inhibiting effect of boric acid (up to 500 ppm B) in the early stages of corrosion. This inhibition disappeared after 800 hours for solutions up to 100 ppm boron in H₂O but was still apparent for 166 ppm B

in D₂O. Weight gains in the 250 ppm B solution in H₂O approached the pure H₂O curve after 4000 hours at 50 C.

Specimens of 6061 aluminum alloy were galvanically coupled to Inconel, Haynes 25, 304 stainless steel, Zircaloy-2 and 6061 aluminum by bolting together 1 x 2-inch coupons at one end. These couples were tested in various boric acid solutions at 50 C. The outer surfaces of the aluminum coupons developed uniform corrosion films of expected appearance. The surface of the aluminum coupons forming the crevice developed nonuniform films in the tighter portions of the crevice, and some of the aluminum specimens pitted substantially. Specimens exposed to a 100 ppm B static solution (air not excluded) coupled to Inconel and stainless steel pitted most rapidly. No pitting was observed on similar specimens exposed to a 100 ppm B refreshed solution (argon blanketed) in 2000 hours.

PRTR Pressure Tubes

Mechanical Testing. Two stress rupture tests, two burst tests, and one crack propagation test were completed. The results indicated an initial increase in stress rupture life with short exposure followed by some decrease in stress rupture life with prolonged exposure to PRTR conditions. The relationship of bursting strength to fast neutron exposure for cold worked material tested at 550 F and for annealed material tested at room temperature was extended by the two burst tests. The crack propagation stress in a PRTR tube was 48,800 psi which, because of the one-inch long crack in this specimen, lies very close to the curve expressed by the equation:

$$\text{Stress (crack length)}^{-0.625} = 49,200.$$

This is the twelfth test confirming the above relation that has been obtained using irradiated and unirradiated specimens.

Pressure Tube Monitoring. During the tube inspection run in January 1965, the lower end of the inspection probe was overheated, resulting in some charring of the illuminating lamp assembly and smoke blackening of the objective and relay lenses in the objective section of the borescope. These lenses were removed and cleaned and the lamp assembly repaired and the equipment restored to operating condition. Little or no radiation darkening of the lens elements (Corning 7940 purified fused silica and dense flint glasses) has occurred from service over the past nine months. For this period the accumulated gamma radiation dosage exceeds about 3×10^8 R.

Zr-Nb Pressure Tubing. About 75 feet of Zr-2 Nb alloy tubing was received and inspected on site by means of ultrasonic, Zyglo, and white light borescoping. This material was produced with either 30% or 60% cold work. About 40% of the tubing containing 30% cold work

exhibited inside surface cracking. The vendor will attempt to provide an additional 20 feet of tubing in this cold-worked temper.

Stress Analysis. A numerical method for analyzing thermal stresses in cylinders formed by a grid of triangular and rectangular rings has been programmed, and a rough draft report describing the computer program has been written. Latest additions to the program include a new method for representing a plane strain end condition and the representation of the rectangular rings in terms of four triangular rings where the temperatures and material properties of each triangle ring are specified. The representation of the rectangular rings in this form allows for a finer definition of material and temperature conditions without increasing the work of numerical evaluation. The program for the rectangular rings was checked for cases of uniform internal pressure, twisting, and uniform axial tension.

All the modifications to the stiffness coefficient due to boundaries other than free surfaces are done in one section of the program. Thus, boundary conditions such as plane strain, net axial forces, or radial surface forces can be made by simply changing this section of the program. The program will be applicable for fuel element end caps or transition joints between tubes.

Reactor Components Development

Ex-Reactor Studies. To confirm fretting corrosion results reported last month, the test using a UO_2 fuel element with 1/4-inch-wide by 1/2-inch-long centering feet was repeated. Examination of the liner of the vertical test section of TF-7 after exposure to externally excited vibration confirmed that fretting corrosion associated with a 500-hour test at 530 F is negligible. Penetration of the three abraded areas was less than 0.0005-inch which, on the basis of past experience, is considered insignificant depth. These results establish that fretting attack might be reduced to a negligible level by minimizing radial pad clearance and increasing pad area.

PRTR Tests. The sub-test of PRTR Test 101 dealing with the time history of the zirconium concentration in the coolant was performed on February 15, 1965, and the first preliminary data were received. Several samples will be re-analyzed to verify and to establish the concentration where reported to be greater than 10 ppb.

Vibration data were not acquired during this test; however, a 21% increase in vibratory motion was probable from an increase in steam pressure from 335 to 365 psig, based on past studies. The vibration monitoring system on selected inlet jumpers was inoperative during the month because of amplifier malfunction. The above test will be repeated after the vibration monitoring system is restored to service.

PRTR Mechanical Shim Rods. In a laboratory investigation of a Mark-II, first-generation shim rod, rod sticking was initiated by thorough degreasing and subsequent operation (reported previously). The sticking was traced to jamming in the ball nut assembly apparently caused by the presence of gray, flaky, foreign material. Spectrographic analysis and metallographic examination of the Haynes alloy 25 balls and ball vanes substantiated that this foreign material was corrosion product from the 6063 aluminum lead screw. Metallographic examinations at 1000X indicated the balls, ball vanes, and guide vanes to be free of any foreign or corrosive material or signs of corrosive attack.

Design Analysis

Fuel Re-use. All the economics cases were re-run with the revised QUICK code. Losses are correctly handled for both U-235 enriched and re-use cases. Sufficiently high "fuel service charges" (up to \$300/kg) were applied to the re-use cases to preclude these being limitations on the "indifference value" analyses. Although some extrapolations are still required, it now appears quite certain that the indifference value (of the fuel service charge) is insensitive to the fuel use charge.

A proposed outline of an experimental program for initial in-reactor demonstration of the fuel re-use concept (consecutive irradiation of fuel element in different reactor regimes) was prepared. The experiment is intended to yield data on enrichment in depleted UO_2 fuel elements from irradiation in fast reactor radial blanket. Since this irradiation will cause nonuniform plutonium buildup and distribution, information on the effect of succeeding irradiation of such non-uniformly enriched fuel element in a thermal reactor will be provided. These data will enable the estimating of the amount of fuel element segmenting or rod rearrangement desired in the cycle.

Plutonium Utilization. An investigation of plutonium burning in large thermal power reactors was initiated. The study is designed to reveal limitations related to the substitution of plutonium enrichment for uranium in thermal power reactors. The Yankee pressurized water reactor, Core I, was selected for a first case, and a burnup analysis was set up for the ZODIAK 2 multigroup, multiregion code.

PRTR Advanced Testing Study. Conversion of the PRTR to a two-region core is one of several methods of providing a versatile, multipurpose facility. Preliminary study of this concept was directed toward defining the central region core dimensions and operation conditions. Core power levels in the 40 Mw to 60 Mw range are under consideration. Using current PRTR, 19-rod cluster elements as driver fuel, the number of clusters, average tube power, and fuel rod surface temperatures were calculated as a function of core diameter with fuel-to-flow ratio as a parameter. Required coolant flow rates were determined for coolant outlet temperatures near 150 F.

2. Plutonium Ceramics Research

UO₂-PuO₂ Mixed Fuel Microscopy. Examination of a specimen of impacted UO₂-20 wt% PuO₂ fuel was conducted by replica electron microscopy. The fuel, previously examined by electron microprobe, consisted of PWR grade UO₂ plus PuO₂ (prepared from the oxalate), ball-milled 64 hours and impacted.

Microscopy of replicas of the polished surface revealed comparatively rough areas (~4 microns diameter) distributed randomly over the specimen. A similarly rough-appearing, continuous phase interconnected these areas, surrounding most of the particles (~3 microns) of the remaining smoother phase.

The appearance, distribution, and proportions of the smooth and rough phases suggest that the PuO₂ is present as ~1/4 micron-diameter particles that form both the 4-micron agglomerates and the continuous phase surrounding each UO₂ particle. This interpretation is consistent with the 20-micron diameter electron microprobe scan, which showed occasionally high Pu concentrations superimposed on a more uniformly fluctuating Pu background. Detailed study of additional specimens is needed to confirm this interpretation.

Phase Studies. Calibration of the tensimetric apparatus with CeO₂ was completed. Several isothermal equilibrations at fixed oxygen pressures showed good agreement for the composition of CeO_x with data recently published in the literature. Results from 11 experiments yielded values of x agreeing to within 0.003 of the literature data (over the region 1.781 < x < 1.983) with one extreme deviation of 0.005. The cryostat was used successfully in several of these experiments at temperatures between 0 and -25 C. Plots of 1/2 log P_{O₂} against 1/T for constant compositions were in agreement with the literature cited within 0.03 log units. Equilibrium was not achieved during heating at 75 C/hr, and it appears that extremely slow heating rates or isothermal conditions are necessary to produce the equilibrium state in the Ce-O system.

Preparation of Pu-B Compounds. The difficulty of arc melting -325 mesh boron powder was circumvented by first "dynapaking" the powder in stainless steel cans. Arc melting of alpha plutonium and boron in chunk form was accomplished at the Plutonium Metallurgy Facility (231-Z) for compositions containing up to 96 at% B. Loss of boron was minimal. A series of heat treatment, x-ray analysis, metallographic, and electrical property studies of these compositions will be undertaken.

Plutonium Ceramics Metallography Facilities. Design of a special glove box facility for taking macrographs of plutonium-bearing particulate fuels is under way. Programs planned for this facility include (a) characterization of UO₂-PuO₂ fuels to determine the particle size

distribution in various particle size fractions and to subsequently correlate it with fuel rod fabrication and performance, and (b) characterization of large grained, arc fused PuO_2 . This facility is necessary to prevent gross contamination of the metallographic hood and to allow efficient handling of unmounted powder specimens. The importance of maintaining the metallographic facilities as contamination-free as possible was emphasized recently by the specimen decontamination required to permit off-site microprobe analysis of Pu-bearing and Pu-contaminated specimens in "cold" facilities.

High Temperature Dilatometer. The high temperature dilatometer was calibrated to 1500 C with a ThO_2 sample holder. It appears that the ThO_2 sample holders must be heat treated to stabilize them before use in the dilatometer. Therefore, the maximum useful temperature for this material has not yet been determined. A new incremental heating and cooling cycle was developed which allows thermal equilibrium to be attained every 100 C. This cycle increases the accuracy and stability of the measurements. The system was operated routinely, unattended, on a 24-hour cycle. However, an inadvertent loss of cooling water resulted in a system burnout. The dilatometer is now being installed in a glove box, and adequate safeguards are being incorporated to prevent a reoccurrence of the heating element burnout.

PuO_2 Microspheres. In order to evaluate the physical and irradiation properties of PuO_2 microspheres, a joint program has been initiated with Mound Laboratory. High density PuO_2 particles crushed, sieved, and fully characterized at BNW, are suitable feed material for the hooded plasma torch at Mound Laboratory. Mound will use this material to prepare microspheres in the 100-200 μ diameter range. The spheres will be returned to BNW for physical and irradiation testing.

Stainless Steel- PuO_2 Cermet Extrusions. The first plutonium-bearing cermets were successfully extruded. Two stainless steel-20 vol% PuO_2 cermet billets were extruded to 1/4-inch diameter pins about 30 inches long. Radiographs show the clad thickness to be fairly uniform--between 0.010 and 0.015 inch. Metallographic examination of longitudinal and transverse sections from one of the extrusions show a metallurgical bond between the clad and core.

Cermet Irradiation Test in P-7 Loop. Cermet fuel pins about 0.25 inch in diameter and 12 inches long are being fabricated for an irradiation test in the ETR P-7 loop. The cermets consist of 20 vol% PuO_2 dispersed in 304 stainless steel and are fabricated by extrusion, cold-pressing, or impaction. For comparison, fuel pins containing 20 vol% U-235 dispersed in stainless steel will also be tested.

Cold-pressed cermet pellets (82% TD) containing -65 to +325 mesh PuO_2 in stainless steel were prepared. Extruded fuel pins are also being

fabricated. An attempt is being made to fabricate integrally clad and capped fuel pins by pneumatic impaction. These pins will be about four inches long and can be welded end to end to a desired length.

Stainless Steel-PuO₂ Cermets. Capsule irradiations in the MTR of stainless steel-20 vol% PuO₂ cermets are continuing. Two capsules--one containing high density and the other low density cermets--were discharged from the reactor after an estimated 2% burnup of the plutonium. Calculated centerline temperature of the cermet was 1300 C. These capsules are being returned to BNW for postirradiation examination.

The stainless steel-20 vol% PuO₂ blended powders were pneumatically impacted to obtain high density (98+% TD) cermets. Low density (82% TD) cermets were made by cold-pressing the blended powders at 70 tsi.

Low density cermets heated to 900 C in vacuum for up to 810 hours showed no appreciable change (<0.6%) in density. High density cermets heated to the same temperature for 409 hours decreased 2.18% in density.

3. Ceramic (Uranium) Fuel Research

Electrical Properties of UO₂. The electrical resistivity of a single crystal specimen of UO₂ was measured to 1600 C in a dried argon atmosphere using the four-probe method. A change of slope of $\ln \rho$ plotted against $1/T$ was observed at 1040 C. The activation energy for conduction was calculated to be 0.47 ev between 700 and 1040 C. The activation energy between 1040 and 1600 C was 1.87 ev.

Cathodic Etching Behavior of UO₂. The cathodic etching behavior of both sintered and arc fused UO₂ is being studied. Differences between chemically-etched (9 parts 30% H₂O₂: 1 part H₂O₄) and cathodically-etched (6 hours at 2 kv, radiofrequency, in krypton gas at 10⁻⁴ torr), sintered UO₂ as observed by replication electron microscopy include (a) greater intraparticle detail and sharper intergranular contrast of the chemically-etched specimen, and (b) rounded pits produced in the grains of the cathodically-etched specimen. This latter effect may be the result of the sintered-nature of the UO₂. The effect is currently being sought in arc fused material.

UO₂ Melting Tests. A UO₂ fuel capsule was irradiated to test the hypothesis that once-molten UO₂ can solidify during continued irradiation as the effective thermal conductivity is improved through fuel structure changes. UO₂ pellets (1.36 inches OD) containing tungsten spheres (-100 to +200 mesh) in coaxial holes were irradiated

in a horizontal position in the KW "Snout" facility. After two hours of irradiation at a heat generation rate sufficient to cause initial melting to ~50% of the fuel radius, the capsule was rotated 180°, irradiated an additional four minutes, then discharged. Destructive postirradiation examination to determine the position of the tungsten spheres will reveal whether or not part or all of the molten UO₂ solidified during the two-hour irradiation.

Hot Stage Microscopy. A new hot stage specimen holder was constructed for reflection electron microscopy. The holder will permit use of the full 30° specimen tilt available in the microscope instead of the presently usable 20°. The new device features a boron-nitride sample block which can be interchanged through the vacuum lock without opening the column in a manner similar to specimen changes routinely accomplished in cold stage reflection operation. Tungsten electrical contacts are used to permit operation to well over 2000 C.

UO₂ Tube-in-Tube Element. Examination of a vibrationally-compacted, tubular UO₂ fuel element showed that failure occurred in the inner annulus of the inner tube. The defect (which occurred after a burnup of 5×10^{18} fissions/cc) was ~7-1/2 inches from one end, near the peak flux position of the reactor. The defect appeared to be a small angular crack with some degree of hydriding in the surrounding area.

Purification of Uranium Dioxide. The heat treating of sintered UO₂ wafers at 1750 C for 12 hours in flowing hydrogen lowered the fluoride content from 670 ppm to less than 2 ppm.

Materials and Information Exchange. As part of the exchange program with Argonne National Laboratory, approximately three kilograms of PuO₂ were prepared by the Nupac process with no unusual problems. The density obtained was 97% of theoretical, the highest density that has been achieved on PuO₂ or UO₂ fine-grained powders. Two and one-half kilograms of densified PuO₂ has been prepared for shipment to ANL, and the balance retained here for analysis and study. Characterization of the densified PuO₂ is under way.

Current requestors for single crystals of UO₂ included two sites in Japan and the National Magnet Laboratory at MIT.

The US-Japan Research Newsletter No. 3 was compiled.

4. Basic Swelling Studies

Irradiation Program. A general swelling capsule containing eight, 2.88 at% enriched, high-purity uranium specimens in several metallurgical states continues to operate successfully in-reactor at a controlled temperature of 375 C.

The third controlled pressure-temperature swelling capsule (P-4), scheduled for in-reactor operation at 1000 psi and 625 C, was assembled and shipped to a reactor for irradiation testing. It contains ten, high-purity uranium, U + Fe - Al and U + Fe - Si specimens with several histories of heat treatment.

Postirradiation Examination. Longitudinal sections of three specimens (two, high-purity uranium and one U + Fe - Al) recovered from capsule P-2 controlled at 450 C and 1000 psi were processed for optical and electron microscopy. The burnup in the specimens was 0.13 at%. A few scattered tears and pores were present in the high-purity uranium specimens, both of which were in the as-extruded condition prior to irradiation. The U + Fe - Al specimen, also in the as-extruded state, was completely free of tears and pores. "Banding" in the extrusion direction due to preferential irradiation growth was observed. Examination of unirradiated U + Fe - Al specimens revealed that recrystallization was not complete in the as-extruded condition. This accounts for the banding and for the macro striations which were observed in the U + Fe - Al specimen.

Supporting Activities. A document entitled "Physical Property Changes in Thorium Postirradiation Annealed to 950 C" was written and submitted for publication as a BNWL report. This paper summarizes work done on thorium during the past several years by Physical Metallurgy personnel.

5. Irradiation Damage to Reactor Metals

Alloy Selection

Several nickel-base alloys are being studied to determine the effects of irradiation and environment upon their mechanical properties. During the past month specimens of Incoloy 800, Inconel 600, Inconel 625, Inconel 702, Inconel 718, Hastelloy N, Hastelloy X-280, and Hastelloy R-235 were tested at room temperature and 650 C. These specimens were irradiated at an average temperature of 740 C to an estimated exposure of 2×10^{20} nvt, fast. Data from the tests conducted at 650 C indicate a marked reduction in ductility as compared to the room-temperature tests. Similar specimens irradiated to a higher dosage were discharged from the reactor during the month.

Present and future reactor concepts utilize a liquid metal coolant. Components for a bench model of an irradiation capsule to be used to evaluate the effects of irradiation and a liquid metal coolant upon the mechanical properties of structural metals are being fabricated and assembled. The design of safety control circuits and a temperature control system was completed and reviewed by reactor personnel.

In-Reactor Measurements of Mechanical Properties

An in-reactor creep test on annealed 304 SS at 650 C and 10,000 psi is in progress. After 300 hours at test conditions it shows a creep rate of 6.8×10^{-6} /hr. The ex-reactor test at these conditions has not accumulated enough time to allow a creep rate to be determined.

The metallographic investigation being conducted on the irradiated 304 SS creep specimen is partially completed. Photomicrographs of transverse sections from one buttonhead and from near the fracture show no unusual features when compared to the unirradiated material. Longitudinal sections from the area near the fracture and the buttonheads will be made soon.

Irradiation Effects in Structural Materials

The purpose of this program is to investigate the combined effects of irradiation and reactor environment on the mechanical properties of structural materials. Special attention is being given to the determination of mechanical property changes produced in metals by irradiation at elevated temperatures.

Tensile tests were conducted on BONUS transition joint specimens in the as-fabricated condition and after an exposure of approximately 725 hours in the out-of-reactor loop. These tests were performed at 550 F and at room temperature on specimens formed by both coextrusion and explosive welding processes. The integrity of both types of joints is very good since in the 14 tests conducted to date, all of the specimens have failed in the Zircaloy-2. No changes were noted in either strength or ductility after exposure in the out-of-reactor loop.

Several tensile tests have been conducted on stainless steels at temperatures from 400 to 750 C. The data from these tests were submitted for processing on the computer. Preliminary results indicate a continual decrease in ductility as a function of increasing test temperature up to approximately 600 C. Above this test temperature the ductility is gradually improved up to 750 C. These tests are being performed on stainless steels irradiated at 50, 290, 550, and 750 C. One of the objectives of this study is to determine the combinations of irradiation and test temperatures which produce minimum ductility. This information can be used to establish practical design limits for reactor components.

During the month, a dynamic solution of crack extension in the double cantilever beam (DCB) specimen was developed. This solution is now being used to aid in the proper design of the specimen. Basically, the solution relates the crack velocity to crack length subject to the effect of crack velocity on the energy absorption rate. The problem of developing a relationship between the energy absorption rate and the crack velocity so far has defied solution.

A computer program for processing fracture data from DCB specimens has been initiated and is now in the final stages of development. This program is being styled to fit the larger and more general REM program for inclusion at a later date.

Irradiation Effects in Nickel-Base Alloys

Twenty-four Hastelloy X-280 tensile and stress-to-rupture specimens are being irradiated in the G-7 position of the ETR at 540 F to a goal exposure of approximately 2×10^{20} nvt. These specimens are to test the effect of irradiation upon several experimental pre-irradiation treatments. These treatments induced general precipitation within the matrix, thus reducing the formation of embrittling deposits at the grain boundaries during irradiation. Additional Hastelloy X-280 specimens are being prepared in similar treatments for irradiation at 1300 F.

Ten Inconel X-750 tensile specimens, representing five experimental pre-irradiation treatments, have been fabricated and are being placed in quadrants for irradiation in the G-7 position of the ETR as soon as space becomes available. Other specimens are being prepared in similar treatments for irradiation at 1300 F.

Specimens of Inconel 600, a solution-hardenable alloy with similar chemistry to that of Inconel X-750, with the exception of lesser amounts of aluminum and titanium so that precipitation-hardening does not occur, are being prepared for irradiation at 540 F and 1300 F. These will undergo pre-irradiation treatments similar to those of Inconel X-750. It is felt that a better understanding of the nature of radiation damage in complex high temperature alloys may result from a comparison of the test data for the two alloys.

Damage Mechanisms

The objective of this program is to determine how interactions between irradiation-induced defects and dislocations modify plastic deformation mechanisms in a metal. The role of interstitial impurities in alpha iron is currently being investigated.

During this period several single crystals of Ferrovac "E" iron were grown by a strain anneal method, were indexed, and were polished preparatory to deformation and slip trace analysis. The largest of these crystals was roughly 50 mm x 5 mm x 1 mm.

A hardness recovery curve determined from an isochronal annealing experiment over the range 0 - 900 C showed two major recovery processes in Ferrovac "E" iron. The first was a broad peak over the range 270 C - 470 C which represented 37% of the hardness recovery. The second peak, which was much narrower, was centered at 680 C and represented 59% of the hardness recovery. Similar experiments on irradiated specimens of Ferrovac "E" and BMI zone-refined iron are in progress.

A study of machine effects during strain rate changes was conducted to ascertain the cause of inhomogeneous yielding in specimens of Ferrovac "E" iron tested at room temperature. Yield point effects in Ferrovac "E", which shows a yield point, and BMI which does not, were studied by simultaneously recording load-time and strain-time curves during strain rate changes, affected by the Instron automatic speed changer. These experiments indicated a slight delay in strain-rate transition, but the rate steadily increased to the new rate rather than the decrease and sudden surge observed by Bolling, et al. No differences were noted in the curves for Ferrovac "E" and BMI iron.

Similar experiments on initial yield points, where the specimen is loaded at a constant crosshead rate, showed a large increase in rate following yielding. This is caused by the elastic springback of the tensile machine following yielding at the upper yield point. The rate returns to that imposed by the crosshead after the lower yield point is reached.

ATR Gas Loop Studies

Model Loop Studies. An extensive test run was made with the model gas loop to determine the characteristics of foil insulation that contained one prototypical ATR joint, and one open joint with a 1/16-inch longitudinal gap between the stacks of foil. No attempt was made to prevent gas flow through the open joint. A preliminary analysis of the data produced a conductivity of 0.65 BTU/hr-ft-F at a mass flow per unit cross sectional area of pipe of 22,900 lg/hr-ft² and a mean temperature of 1200 F. This mass flow compares closely with prototypical flow of 5,000 lb/hr in a 6-inch pipe which is 24,500 lb/hr-ft². The tests showed a definite dependence on flow, which is in direct contrast with the previous runs which indicated no affect of flow. After the next series of tests an attempt will be made to modify the open joint in order to determine if this joint is affecting the conductivity of the insulation three feet downstream. The open joint created a hot spot on the test section that was several hundred degrees hotter than areas covered by the insulation.

During the test run the loop operated with a small bypass cleanup system that consisted of a charcoal and a molecular sieve cold trap. Several days of operation were completed with less than 1 ppm of water, and with hydrogen being the only other major impurity. Gas temperatures out of the heater ranged up to 1700 F during this period. A catalytic copper oxide bed is being installed in the bypass cleanup system to aid in the removal of the hydrogen.

The test section is being modified to incorporate a cooling water jacket. This modification will permit testing of the foil insulation with high temperature differentials and will allow testing under ATR conditions. The gas-bearing compressor has completed 270 hours of

operation over a wide range in pressure, temperature, and speed. During this operation the compressor has been started about 80 times. No problems have developed with the compressor.

Model Loop Heater Design. Detail drawings of the second generation helium gas heater for the model gas loop were completed. Orders were placed for the electrical control system and 60% of the material for major components of the heater. An analysis is being made to determine what piping changes will be necessary in the loop to replace the existing heater with the new model.

ATR Loop Corrosion Studies. Microbalance studies of niobium exposed to oxygen at low pressures was started. As-received niobium was oxidized in 10 to 12 μ replenished oxygen atmosphere. Linear rate constants calculated for these tests are: 1.96×10^{-4} mg/cm²sec (50 to 1200 min) at 1000 C, 2.44×10^{-4} mg/cm²sec (initial rate), 1.68×10^{-4} mg/cm²sec (175 to 1100 min) at 900 C, and 2.38×10^{-4} mg/cm²sec (initial rate), 7.73×10^{-5} mg/cm²sec (50 to 800 min) at 800 C. The rate of oxidation is relatively fast, the initial rate apparently controlled by the diffusion of oxygen to the sample. The sample sits in a static leg removed from the point of pressure measurement, oxygen leak, and system pumps in that order. The actual oxygen pressure around the sample is not known. System modifications will be required for a quantitative determination of the surface reaction rate as a function of oxygen pressure.

Dynamic Materials Testing Apparatus. A 10-day run with a niobium alloy was completed on February 1, 1965. Shortly before completion of the run, the guard heater failed. Upon inspection of the guard heater, it was found that grain growth of Pt-1% Rh heating wire had taken place and the wire had failed. This winding had about 800 hours of service at an operating temperature of approximately 2050 F. Wire temperature probably is several hundred degrees F above this temperature. The guard heater has been replaced, and installation of the new recorder-controller has been started.

The Cb-1 Zr sample was exposed to untreated Grade A helium in the loop for 285 hours at 2050 F and 350 psi. Gas velocities are 525 fps. It was found that the sample formed a tenacious oxide film, with no evidence of the spallation found during oxidation of Co or Ni-base superalloys. The sample showed a weight gain of 2 mg/cm², and an average increase in the diameter (normally 0.25 inch) of 4 mils.

Outgassing of Ceramics. Specimens of Coors AD-94 (94% Al₂O₃), AP-100 (100% Al₂O₃), and AHP-99 (99% Al₂O₃, machinable) were supplied by Materials Engineering for outgassing studies. These materials are candidates for model gas loop heater insulation. The AP-100 material was not supplied in samples massive enough for a meaningful test. The AD-94 and AHP-99 material was broken into pie-shaped pieces

weighing about 100 gm each. These pieces were stacked three deep, placed on a grate, and outgassed in vacuo for two hours at 400 C and 600 C. The AHP-99 material lost an average of 0.013 wt% during the 400 C test, and 0.020 wt% during the 600 C test. The AD-94 material lost 0.004 wt% during each test. The weight lost during either test, from either material, was gained back rapidly during exposure to lab air (~80% in 30 min), presumably by adsorption.

Helium Diffusion Cell. The quartz helium diffusion cell was tested as a source of ultra-pure helium. This helium will be used as gas chromatograph carrier gas and ATR reference helium for zeroing the instrument. Helium delivered from the cell was invariably contaminated with 10-20 ppm H_2 . It appears the source of the H_2 is internal contamination. Discussions with another user of this same device at Bell Telephone Labs indicated the cell is capable of producing ultra-pure helium, suggesting our unit is defective. The cell was returned for repair or replacement.

6. Nuclear Graphite Studies

Gamma Irradiation Facility. An additional 86,000 curies of cobalt-60 were added to the gamma irradiation facility to bring the total to 185,000 curies. Ceric sulfate dosimetry measurements were completed in a 2-inch diameter tube which extended from the source to three feet above the surface of the water. With all of the cobalt positioned around a single, 2-inch diameter tube, the dose rate was 1.65×10^7 r/hr. The variation in dose rate over an 8-inch length was less than 5%. When the cobalt was placed around two 2-inch diameter tubes, the dose rate was 1.23×10^7 r/hr.

One tube is currently used for irradiations near room temperature, and irradiations at temperatures up to 500 C are conducted in the second tube. This facility is quite versatile and could be easily modified for irradiations at other conditions.

EGCR Graphite Irradiation. The report, "The H-3 Irradiation Experiment: Irradiation of EGCR Graphite--Interim Report No. 2," HW-71500B, was issued. This report presents information from the first seven capsules in the series of EGCR graphite irradiations.

The ninth capsule, H-3-9, in the series successfully completed four cycles of operation in the GETR and was removed from the reactor on February 1. Maximum neutron exposure is estimated to be approximately 1.35×10^{22} nvt, $E > 0.18$ Mev. Upon disassembly of the capsule, it was found that the four samples in Position 1 were frozen into the central graphite collar and cooling ring, and during removal the ends were accidentally broken off. Consequently, the hole-to-hole length measurements will be used to determine the length changes for these samples. All other samples were recovered in excellent condition.

Construction of H-3-10 is under way concurrently. The four samples from H-3-9 position will be modified by rounding off the broken ends and remeasuring. Since these samples provide data at approximately 325 C, they will be used in H-3-10. The central graphite collar will also be revised by splitting it into sections to prevent expansion from freezing the collar around the samples. A new special sample holder will hold the samples in place. The H-3-10 capsule will include 19 samples from H-3-9, three from H-3-8, and two from H-3-7, with four samples having been in all nine previous capsules. H-3-10 will be installed in the GETR the first week in March.

During the past GETR shutdown and just prior to the removal of H-3-9, a flux run was made with the capsule located in a new position, E8. The purpose of the run was to (1) measure fast fluxes by means of nickel and aluminum wires in several reactor positions and correlate these with physics calculations, and (2) determine sample temperatures in H-3-9 at several power levels to enable extrapolations to higher power levels contemplated for the future. Flux wires are still being measured; however, sample temperatures indicate that reactor operation at approximately 45 MW would be possible with the capsule temperatures remaining at the present operating levels. Consequently, additional determinations are being made by the GETR physics and engineering groups to determine in detail the feasibility of increasing reactor power levels.

High-Temperature Graphite Irradiations. The second capsule, GEH-13-11, of the high-temperature graphite irradiation series is performing satisfactorily in the K7 position of the ETR. The capsule contains 29 samples being re-irradiated plus five new samples. Maximum sample temperatures are approximately 1150 C, and all thermocouples are functioning properly.

Irradiation of Experimental Graphites. Samples from the Speer contract (DDR-164) were discharged after an initial exposure of 1650 Mwd/At_K. The samples were irradiated in the 3C test hole at KE Reactor at a temperature of 600 ± 100 C.

Two series of extruded samples were tested. The first series was prepared from one formulation of raw materials and the dimensional change during processing was controlled by varying the rate of temperature rise during graphitization. The second series, prepared using a graphitized filler and a pitch binder, were heat-treated to various final temperatures ranging from 700 to 2900 C.

An effect of graphitization rate and binder graphitization temperature was seen for both transverse and longitudinal orientations. At low-binder temperatures, the transverse orientation experienced the larger contraction. However, higher exposures were required to determine long-term effects.

The graphitization rate appears to have no effect on the radiation-induced changes to the coefficient of thermal expansion (CTE) for either orientation. Irradiation increased the CTE about 45% in transverse samples and about 24% in parallel samples.

For transverse samples in the binder series, the CTE's increased 56 and 44% for binder temperatures of 700 and 2900 C, respectively. For parallel samples, the changes were +35 and -5% for the same binder temperatures.

Annealing of Irradiated Carbon. Four carbon samples containing equal amounts of lampblack filler and pitch binder and heat treated to 1400 C were irradiated at 600 C to 2.5×10^{20} nvt and subsequently annealed. The neutron exposure caused the carbons to shrink 0.9% and the bulk CTE to increase 8.7%, but the CTE of the crystallite c spacing and the value of the c spacing remained the same. These results are most easily explained if the material between the crystallites had densified. The driving force for densification could have come from the internal stresses developed during cooling from the 1400 C heat-treatment temperature.

Subsequent annealing to 1100 C made the carbons shrink an additional 0.02% without any noticeable change in the other measured properties. Further heating to 1375 C caused the carbons to expand 0.02% with a corresponding decrease in the bulk CTE to a value 3.8% greater than the unirradiated carbons. During this further heating, the c spacing decreased 0.3%.

These observations are explainable if the small microcracks along the c planes between the subgrains had grown. Microcrack generation would be expected as a result of the decrease in c spacing. Moreover, the generation of microcracks which could absorb the crystallite thermal expansion would explain the decrease in bulk CTE.

Dosimetry for ETR Boronated Graphite Irradiations. A mockup of the shielded boronated-graphite capsule, GEH-23-8, was irradiated at low power in the ETR M6 position to measure the cobalt-cadmium ratio for the shielded facility and to determine axial and azimuthal flux distribution.

Three sets of Co, Cd-covered Co, and Fe foils were spaced 120° around the periphery of the capsule. These wires extended the full 24-inch length of the capsule. A set of foils was also placed in the center of the capsule at reactor centerline.

Analysis of the foil activities (A) gave a Cd ratio ($\frac{A_{\text{bared}}}{A_{\text{Cd-covered}}}$) of 1.71 for the sample position and a spread of 1.68 to 1.88 for the peripheral positions.

The effect of these measurements is to decrease the thermal-neutron exposure to 40% of the original estimates for the previously reported, shielded, boronated-graphite irradiations.

7. Metallic Fuel Development

Irradiation of Thorium-Uranium Fuel Elements. The irradiation of three, tubular, Zircaloy-2 clad, thorium-2.5 wt% uranium-1.0 wt% zirconium, fuel elements continued successfully in the ETR-P7 loop. The fuel elements are currently operating at a maximum temperature of only 385 C due to a decreased neutron flux in the area of the ETR-P7 loop. The surface heat flux is 30 cal/sec-cm² (4.0×10^5 BTU/hr-ft²), and the integrated exposure is currently 3.7×10^{20} fissions/cm³ (10,800 Mwd/ton). No swelling beyond that expected from solid state fission products was observed.

Nb-1 Zr Clad, Th-2.5 U-10 Zr Alloy. Nb-1 wt% Zr alloy has potential application for high temperature service in liquid alkali metal environments due to its corrosion resistance and high temperature strength. The high temperature performance of metallic thorium base fuel makes it potentially useful for liquid-metal-cooled reactor fuel applications. This material combination has not been employed previously, and it was felt desirable to determine the fabrication and bonding characteristics. One rod coextrusion was made producing a 0.565-inch diameter rod with 0.026-inch Nb-1 Zr cladding over a Th-2.5 U-10 Zr core alloy. Extrusion conditions were 800 C - 2-1/4-hour billet preheat, Cu-10 Ni extrusion can with Aquadag lubrication, at a reduction of 13.3:1 in 500 C container and die. K_{max} was 23.7 tsi. Sections of the coextrusion showed excellent clad thickness uniformity (± 0.0005 inch) and complete metallurgical bonding. The Cu-10 Ni extrusion cladding was easily removed with HNO₃-H₂O without attack of either the Nb-1 Zr or thorium alloy core material.

Additional sections were heated in vacuum for two hours at 900, 1000, and 1100 C to check diffusion, bond failure due to thermal cycling, and recrystallization of the cladding. Additional bond zone growth was observed with no bond failure. Components of the diffusion zone are being determined utilizing the electron beam microprobe. The Nb-1 Zr cladding appears partially recrystallized to a fine-grained, equiaxed structure after heating at 1100 C. These results indicate that fabrication of other geometries (tubes or honeycomb shapes) is feasible.

8. Critical Flow of Steam-Water Mixtures

A report entitled "The Critical Discharge of Steam-Water Mixtures from Pipe Elbows and Tees" was submitted for publication. The report covers the study of the critical discharge of two-phase, steam-water mixtures from a piping system when the system is opened to the atmosphere adjacent to a tee or elbow. Two phenomena uniquely determine the maximum

rate at which a two-phase mixture can be discharged from such a system: (1) the critical flow phenomenon and (2) the two-phase, pressure-drop phenomenon. Experiments were performed at moderate temperatures and pressures on 1/2-inch commercial piping and welding fittings to study the effect of the presence of the fitting on these phenomena. In these experiments the range of steam-water total enthalpies considered was between 500 and 1000 Btu/lb. Flow rates ranged from 200 to 800 lb/sec-ft², and measured critical pressures were between 23 and 87 psia. The results indicated that the elbow at the discharge of the flow passage did not significantly influence the critical flow rate. The tee in this location reduced the critical discharge rates from 10% at enthalpies of 900 and 1000 Btu/lb up to 24% at 500 Btu/lb. This indicates that the tee immediately upstream of the location of the flow-choking acts to increase the degree of homogenization of the two-phase mixture. The data gathered in this investigation were correlated by two methods. In the first method the critical flow phenomenon and the two-phase pressure drop across the fitting were considered as one effect, and the critical discharge rate was related to the flow enthalpy and a "pseudo-critical" pressure upstream of the fitting.

In the second correlation the critical flow model of Fauske was combined with the pressure loss information obtained in this investigation. These combined data were referred to the momentum exchange pressure drop model of Levy and the empirical relation of Thom to obtain a general pressure loss correlation for pipe elbows and tees during choked flow. A comparison between the resulting pressure loss correlation with one proposed by Fitzsimmons for normal (nonchoked) flow in similar fittings demonstrated a surprising consistency.

9. Upstream Boiling Burnout

A survey was made of the results obtained to date in the investigation of conditions which can cause boiling burnout to occur at positions upstream of the exit of electrically heated flow channels. The following conclusions were made:

1. In tests with a 12-foot, 0.44-inch ID test section at 1000 and 1500 psig, upstream burnout was found to occur as a reproducible phenomenon with inlet enthalpies near or above saturation and at mass velocities of 5×10^6 lb/(hr)(ft²) and higher.
2. Upstream burnout was also encountered in low-pressure tests (90 to 150 psi). Fifteen burnout points, all upstream, were found with a test section composed of 0.1-inch bands of high heat flux separated by 0.9-inch bands of lower heat flux. Upstream boiling burnout was also encountered with a uniformly heated test section, but the burnout point was usually

accompanied by large pressure oscillations. When the flow apparatus was modified to decrease the pressure oscillations, the burnout points always occurred downstream.

3. Both the high-pressure and the low-pressure data indicate that the burnout heat flux must be a function of some quantity in addition to enthalpy which varies along the length of the test section. The chief significance of the data obtained to date lies in this indication that burnout depends on some factor besides those usually taken into account.

10. Advanced Reactor Concept Studies

Nitride Fuel Cycle Studies. Additional multigroup calculations were completed for the 1000 Mwe nitride fueled fast reactor study. Two cases were investigated to determine what changes in neutron energy group structure and cross sections are desirable before refining the core design further. The 16-group YOM cross section set, used initially, lacks low energy detail and does not contain the more recent high energy plutonium data. A comparison of the neutron spectrum for the carbide fueled 1000 MWe Combustion Engineering study obtained with an improved 18-group set shows a substantially softer spectrum than that predicted by the YOM set. Further, the sodium coefficient calculated for the nitride core appears to be strongly negative, a questionable result for a core of this size. It is concluded that the 16-group YOM set is not adequate to continue this study and that alterations in both group structure and nuclear data are necessary.

Study of the processes for preparation of nitride fuels revealed that they have been prepared to date only by small-scale laboratory methods involving reaction of the metal (uranium or plutonium) or its hydride with nitrogen. Alternate methods which appear feasible but require development include a carbon-nitrogen reduction of the oxide, or reacting the chlorides with nitrogen or ammonia. As one phase of the nitride fuel cycle study, the feasibility of potential nitride conversion methods will be analyzed and conversion costs estimated. The establishment of a reasonable conversion cost for nitride fuels may well be the key element in establishing their economic position relative to the carbides and oxides.

Plutonium and U-233 Fueling of a Fast Compact Reactor. Survey calculations on Pu- and U-233-fueled, compact reactors, including the effect of tantalum on core size, were completed. A linear extrapolation of size based on volume fraction of Ta appears satisfactory. Three cases, corresponding to the three fuels at 0.95 total pressure ratio, were selected for core zoning. They were zoned using the SWISS CHEESE computer code and are being checked for criticality.

Definition of the minimum possible size core based upon fuel temperature limitations and reduction of tungsten content was not possible. The calculated thermal conductivity of 30 vol% tungsten is still high enough at the design point to preclude fuel temperatures of more than 200 F higher than the tube wall.

The core zoning program, SWISS CHEESE II, was completed and checked against the base design, and an outline of the final report was drafted.

Fast Flux Test Reactor Studies. Information on testing capabilities of the 200 Mwt core was developed and is presented below:

	<u>Open Loops</u>	<u>Closed Loops</u>	<u>Reflector Test Holes</u>
Coolant	Na	Na	Optional
No. of Test Facilities	8 (33)	4 (12)	6 to 20
Power per Loop, Mwt	5-10	5-20	($\phi=2 \times 10^{15}$)
Inlet Temperature °F	450 (750)*	600-1000	Optional
Outlet Temperature °F	800 (1100)*	900-1400	Optional
Net Velocity ft/sec	25-60	20-50	--
Test Specimen "Diameter," In.	3.3 (6)**	3.3 (6)**	1 to 6
Fueled Length, Inches	35	35	--
Total Length, Inches	70	70	70++
(including blankets & plenum)			

*Nuclear preheat; multipass or series flow.

**1 or 2 peripheral positions.

A fast reactor core design-analysis program is being written jointly with Systems Research for use on the Beckman Ease 2133 analog computer. The program is intended to permit rapid parametric surveys of a wide range of design variables. The initial scope of the program will include coolant temperatures, flow rate, and pressure drop; geometry of fuel pin, fuel element, and flow tube; critical mass and flux as functions of core size and composition; temperatures and temperature gradients in film, clad, and fuel; and burnup limits as functions of fuel temperatures and power distributions.

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Although aimed directly at optimizing the FFTR core, the program will be generally adaptable to other fast reactors.

Computer studies to determine the decay heat generation in candidate driver and test fuels for the fast test reactor are under way. The information will be important to the design of fuel handling and fuel examination facilities.

11. Nuclear Rocket Fuels Studies

Studies conducted for AEC-SNPO on tungsten-UO₂ fuel element development and on fueled graphite problems are reported via a separate distribution list.

D. DIVISION OF RESEARCH - 05 PROGRAM

1. Radiation Effects on Metals

This program is directed toward establishing the combined effect of impurities and neutron irradiation on the properties and structure of specific metals, and deducing from thermally activated recovery processes how the damage state can be altered. Present studies involve single and polycrystalline specimens of molybdenum, nickel, and rhenium.

Radiation damage in rhenium, a cph metal, is being studied in a manner analogous to that of molybdenum, a bcc metal. Rhenium foils have been irradiated to an exposure of 7×10^{18} nvt and examined by transmission electron microscopy. Defect structures which could be attributed to neutron irradiation were not observed. This is in marked contrast to observations made on fcc and bcc metals where damage in the form of spots and loops is observed after equivalent exposures. The absence of observable clusters in irradiated rhenium may be explained in several ways. Clusters may exist but may be too small to be resolved by the electron microscope. It is also possible that clustering has not occurred because point defects are not mobile at the irradiation temperature. The irradiated foils are currently being annealed in an effort to promote growth of clusters.

Single crystal specimens of molybdenum which have been used for length-change-lattice parameter measurements have been cut into shorter sections on which crystal faces have been ground and polished. These faces are parallel to (110) and (100) planes. The intensity of diffracted x-rays from these faces will, it is hoped, provide an indirect means for observing the movement of isolated defects during low-temperature annealing. Measurements currently are being made on samples annealed four hours at 200 C, and differences in intensity have been observed.

Studies on the changes in the length of irradiated molybdenum due to annealing have been extended to 220 C. The results, however, were anomalous. An initial decrease in length followed by an abrupt increase was observed. The possibility exists that the oil bath used for annealing may be responsible for contamination of the molybdenum. In subsequent experiments the crystal rods will be sealed in glass ampules before immersion in the oil bath.

A paper, "Line Broadening in Irradiated Molybdenum," was prepared for presentation at a meeting of the X-ray Diffraction Group of the Fuel Element Design Committee convening in Richland in February.

The equations for the two-beam dynamical theories of electron diffraction through a foil were programmed and evaluated for foils of various thickness and with various values for the elastic scattering coefficients. Parametric studies have been made for various foil thicknesses with the electron intensity transmitted through the foil plotted as a function of the deviation from the Bragg reflection condition. Several attempts have been made to correlate experimental and theoretical intensities in order to determine the foil thickness. Kikuchi lines have been used to measure the deviation from the Bragg reflection condition. Difficulty was encountered in obtaining representative two-beam diffraction patterns and verifying predicted foil thicknesses.

Tests on the rate- and temperature-dependence of the flow stress in irradiated polycrystalline molybdenum continue. The drop in load occurring upon initial yielding has been correlated with exposure and with postirradiation annealing treatment based on current rate theory.

2. Plutonium Physical Metallurgy

The objective of this program is to derive fundamental information relative to (1) the kinetics and mechanics of the phase transformation in plutonium, and (2) the mechanisms by which monoclinic plutonium deforms.

The influence of plastic deformation of a stable plutonium phase on subsequent phase transformation has been investigated and related to the mechanism of the phase transformation. Previously it was reported that plastic deformation of the beta phase retards the beta to alpha transformation and that plastic deformation of the gamma phase accelerates the gamma to beta transformation. Currently it was shown that plastic deformation of the alpha phase by compression, e.g., 30% reduction in thickness, accelerates the alpha to beta transformation and that plastic deformation of the beta phase accelerates the beta to gamma transformation. The accelerating effect of plastic deformation

of the stable phase on its subsequent transformation is a characteristic of diffusional transformations. The retarding effect of plastic deformation of the stable phase on its subsequent transformation is a characteristic of diffusionless transformations.

Studies on the effect of applied uniaxial tensile stresses on the low-temperature, allotropic transformations of plutonium were performed and the data were compared to similar compression studies. The results were related to the phase transformations of other metal and alloy systems. Applied stresses during beta to alpha transformation produce a crystallographic texture and a microstructural shape texture. The alpha grains are elongated in a direction perpendicular to the applied tensile stresses. They are parallel to the applied compressive stresses. The elongated grains are aligned in bands which are perpendicular to the direction of applied compressive stresses and parallel to the direction of the applied tensile stresses. The (010) plane is parallel to the direction of the applied tensile stress and perpendicular to the direction of the applied compressive stress. The data also indicate that texture can develop as a result of alpha to beta, beta to gamma, and gamma to beta transformations under either applied uniaxial tensile and compressive stresses.

Some phase transformation creep occurs during the transformations when subjected to an external stress. Phase transformation creep is the strain during transformation that cannot be attributed to either normal creep or anisotropic volumetric changes. More alpha to beta transformation creep occurs under a tensile stress than under an equal compressional stress. Little or no beta to alpha transformation creep occurs under compression, but some beta to alpha transformation creep does occur under tension.

The effect of applied tensile stresses above about 4000 psi on the alpha to beta transformation is to cause all of the transformation volume changes to occur in a direction perpendicular to the applied stress.

The temperatures for the start of the beta to alpha transformation under applied uniaxial compressive and tensile stresses were determined and related to the mechanism of transformation. Both applied uniaxial tensile and compressive stresses raise the temperature for the beginning of the beta to alpha transformation 3C/1000 psi at a cooling rate of 2 C/hr. This can be explained qualitatively on the basis of the transformation being a shear-type transformation. A similar effect has been observed in shear transformations in other alloys. Shear and normal compressive components favor the beta to alpha transformation, but a normal tensile component opposes the transformation. Accordingly, the shear stress in the beta to alpha transformation in plutonium aids the transformation more effectively than the normal stress opposes it. For this explanation to be valid, the shear displacement would have to be much larger than the dilational displacements.

It was previously reported that the cycling of a textured alpha plutonium sample into the beta phase does not restore the alpha to a random condition. This was borne out in recent investigations with added indications of a definite crystallographic relationship existing between alpha and beta. For example, it was noted that in alpha samples, oriented by beta to alpha transformation under compressive stress, an excursion into the beta region results in a 90° shift in the [010] direction. While this was not found in every specimen examined, it was repeated a sufficient number of times to indicate that certain temperature and stress conditions arising during the beta to alpha transformation can result in this rather unusual crystallographic phenomena. The fact that the [010] direction appears to be so sensitive to the direction of applied stress during the formation of alpha from beta plutonium suggests that maximum change occurs in the [010] direction.

A study of slip lines and directions was undertaken on textured alpha plutonium samples. Alpha pressing resulted in the formation of slip lines which are evident in photomicrographs. These have not, as yet, been completely analyzed.

E. CUSTOMER WORK

1. Radiometallurgy Laboratory

Examinations. The results of routine examinations and measurements are or will be reported as part of the sponsoring research and development programs. During the reporting period, examination and testing included the following:

Photomicrography	- - - - -	46
Photomacrography	- - - - -	68
Photomosaics	- - - - -	7
Autoradiography	- - - - -	18
Replication	- - - - -	7
Fission Gas	- - - - -	12
Hydrogen Determination	- - - - -	15
Density	- - - - -	11
Dissolution	- - - - -	31
Microdrilling	- - - - -	55
Microhardness	- - - - -	5
Macrohardness	- - - - -	122
Tensile Testing (Rm Temp)	- - - - -	28
Tensile Testing (Hi Temp)	- - - - -	29
X-ray	- - - - -	3

Project CAH-136, Service Addition - 327 Building. The project bid package was completed. Bids are due in March.

"E" Cell Metallographic Facilities. Operational tests were completed on the hand-operated fine grinder. Metal removal rates were good and remote operation of the paper rolls was satisfactory. Two additional grinders were fabricated and installation of these and the belt sander is in progress.

Molding of Spline Seals. A mold was designed and fabricated and two prototype spline seals were molded from polyvinyl-chloride. The seals will be tested by IPD Plant Engineering personnel to determine whether the new seals will solve the spline-seal leaking problem.

Strain Gage Spot Welder. Strain gages were successfully attached to cold Zr-2 process tubing using the remote spot welder in a mockup. In-cell testing of the spot welder is in progress.

"B" Cell Modification. Preliminary drawings were completed for a cell tray, heater-stirrers and hood for the south end of "B" cell.

2. Metallography Laboratories

During the report month 398 samples were processed, a total of 732 micrographs and macrographs taken, 1949 negatives printed, and 7032 prints processed.

3. High Temperature Lattice Test Reactor (HTLTR)

Fabrication and installation of another set of graphite heating elements was completed--two of the four heating elements presently installed are of a new "hourglass" design, while the remaining two are of the rectangular design used in Run No. 1. Inadvertent fabrication errors including (1) slippage of a jig holding an element, (2) breaking an element by bumping, and (3) cutting an end taper backwards, resulted in the loss of three heating elements. Electrical resistances of the finished elements as measured with a Wheatstone bridge indicated the individual resistances varied $\pm 1\%$ from an arithmetic average of the measured values. When installed, the individual heating element circuits matched within $\pm 5\%$ of an arithmetic average.

The molecular sieve-type gas cleanup system was designed and installation is 70% complete.

The following startup items were completed prior to start of evacuation:

1. Vent line relocated.
2. Tygon cooling water hoses replaced.
3. Heating elements replaced.
4. Ground bus isolated from mockup shell.
5. New thermocouples installed.

6. Sized three orifices and installed.
7. Safety and control rods installed.
8. All recorders calibrated.
9. Gas analysis equipment adjusted.
10. Recording-controlling pyrometer calibrated.
11. Blower bypass installed.
12. Graphite sample system installed on west door.
13. Install doors.

The safety rod assembly was installed on the mockup preparatory to testing. Piping and wiring of the rod was completed, and cold testing (on the mockup) was started. The horizontal control element was disassembled and all components weighed prior to installation in the large mockup.

The examination of TD Nickel cladding from fuel and poison rod specimens from the HTLTR environmental test described last month was completed. There was no evidence of reaction between the cladding and UO_2 or Al_2O_3 - Gd_2O_3 mixture.

Metallic specimens which were exposed in the initial run of the HTLTR mockup in the 314 Building were found to have been affected by an oxidizing atmosphere. In this environment the chromium-containing alloys were the least affected because of their oxidation resistance, whereas the alloys, TD Nickel, Hastelloy B, and Nickel 200 were severely corroded. Surveillance of materials will continue in future runs of the 314 mockup. The chemical nature of the atmosphere in this test chamber is expected to change as the water content is lowered with continued operation.

4. EBWR Fuel Elements

EBWR Plutonium Recycle Fuel. The next shipment of plutonium fuel rods to Argonne is scheduled for March 15, and will complete the plutonium fuel required for the EBWR core in support of the Plutonium Recycle Demonstration Experiment. More than 700 depleted UO_2 -1.5 PuO_2 fuel rods and 20 special rods (Table I) will make up the shipment. Most of the 700 rods were returned from Argonne National Laboratory and repaired by replacing the top end cap.

TABLE I

SPECIAL RODS

<u>Quantity</u>	<u>Core Composition</u>	<u>wt% Pu-240</u>
4	Al-3.35 Pu-2 Ni	8
4	Al-3.35 Pu-2 Ni	26
4	UO_2 (Normal)	--
4	UO_2 -1.5 PuO_2	20
4	UO_2 -1.5 PuO_2	26

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Fabrication of 150 spare fuel rods will begin after the above shipment is made.

Irradiation-Testing of EBWR Prototypic Fuel Rods. Maximum exposures of the in-reactor (MTR) and discharged, short EBWR specimens are 4.1×10^{20} fissions/cm³ (15,000 Mwd/ton of fuel) and 4.8×10^{20} fissions/cm³ (17,000 Mwd/ton of fuel), respectively. Capsule irradiation tests are continuing and indicate that the EBWR fuel will perform satisfactorily. Seven capsules with short sections of EBWR fuel rods are still being irradiated and 25 have been discharged from the MTR.

A 21-rod bundle (5 x 5 array with corner rods missing) with full-size replaceable EBWR fuel rods is being irradiated in the PRTR rupture loop. The bundle is operating with a heat output of 450 Kw, which is equivalent to an average rod surface heat flux of 51 w/cm². The maximum operating conditions to date have been: heat output - 520 Kw, rod heat flux (average) - 59 w/cm², and rod heat flux (maximum) - 65 w/cm² (60 w/cm² is maximum value for EBWR). The bundle has operated for 552 hours in the loop. Upon completion of the one cycle in the loop, it is planned to position the bundle in the PRTR core and continue the irradiation test. Tentative plans call for rod replacements after the following exposures: one rod at 0.4×10^{20} fissions/cm³ (1500 Mwd/ton of fuel), two rods at 1.4×10^{20} fissions/cm³ (5000 Mwd/ton of fuel), and two rods at 2.7×10^{20} fissions/cm³ (10,000 Mwd/ton of fuel).

Twelve, 3-rod clusters of full-size EBWR rods commenced irradiation. Proposed goal exposures for the four-cluster groups are: 0.4×10^{20} fissions/cm³, 1.4×10^{20} fissions/cm³, and 6.8×10^{20} fissions/cm³ (25,000 Mwd/ton of fuel). Rods in all clusters are of the repaired type.

EBWR Cladding Stress Studies. Tests are continuing to determine the nature and magnitude of stresses developed in EBWR cladding during vibrational fuel loading using either electro-dynamic or pneumatic variable impact vibration (VIV) shaker units.

Many resonance frequencies were found when using the electro-dynamic system for vibrating EBWR tubes. The resonance frequencies induce two general types of waves in the Zircaloy tubes: (1) horizontal standing waves with maximum deflections normal to the tube axis and (2) vertical waves which are superimposed on the sinusoidal wave generated by the shaker system. Cracking from fatigue is easily produced from the horizontal motion of the horizontal standing waves but sustained shaking with only vertical deflections will not induce cracking. Stress-coat analysis of cladding strains during shaking has helped delineate areas of high stress, while subsequent strain gauge measurements in these areas show stresses of 20,000-40,000 psi.

It was further observed that stress values in the tubing near the clamp assembly and resonance frequencies changed during UO_2 loading of the tube. Particularly bad stresses ($\sim 20,000$ psi) were recorded in the cladding near the clamp at 350-400 cps for full fuel rods. Specific clamp arrangements, designed to minimize stress concentration, showed some promise of success. It also appears that solid lubricants such as MoS_2 are effective in reducing cracking, probably because of reduced fretting action between the stainless steel clamp and the Zircaloy tubing.

Studies with the pneumatic VIV shaker show different stress patterns. Momentarily high stresses ($\sim 20,000$ psi) are obtained during impact, but they dampen out within 0.1 sec. Impact pulses are produced every 0.25 sec and decrease in intensity as the rods are filled with fuel. Initial studies indicate that the integrated fatigue during a normal loading operation is much less for the VIV system than for the electrodynamic system.

EBWR End Cap Cracking. Metallographic examination is being performed on selected rods exposed to thermal cycling studies. Four of these rods showed ultrasonic defect indications in the end cap region prior to cycling. Of these end cap indications, two remained relatively unchanged after thermal cycling and two propagated extensively. Weld-bubble voids were found in nonpropagating defects, and cracking was found in end caps with propagating defects.

Subtle differences appear to exist in ultrasonic trace records made on these rods. Efforts to determine if cracks can be distinguished from voids by analysis of ultrasonic signals are being made.

One rod, originally free of defects prior to fabrication, demonstrated defects 2-3 mils deep in the cladding. Preliminary examination of the cladding "defects" indicated that they are surface roughness caused by oxide fuel impinging on the interior surface while loading.

5. Other Customer Work

Saxton Fuel Element Fabrication. Forty-eight kilograms of UO_2 -6.6 wt% PuO_2 were blended and sent through the various steps of the Nupac process. Eighty-four individual rod loads were prepared from this material and are now on hand.

Six fuel rods, loaded by a special technique, resulted in contamination of the counterbore areas. This problem did not occur in loading three additional rods by the standard technique, which uses a sleeve insert to protect the counterbore. X-rays of these three rods showed that a 1/8-inch alumina spacer, which is inserted in the fuel tube prior to loading, had shifted in position. Fine Nupac powder had worked its

way around the spacers and moved them 1/16 to 3/16-inch up from the bottom of the tube. Permission to eliminate these bottom spacers is being sought from Westinghouse.

Ultrasonic examination of these rods showed no crack indications in either the weld or clamp areas.

Weld samples were sent for approval to Westinghouse and were rejected on the basis of not having 100% penetration. Examination showed that all welds did have 100% penetration, but that the thickness from the throat of the weld to the outside surface of the end cap was less than 100% of the counterbored tube wall thickness in a number of samples.

A second series of weld specimens was prepared, changing the weld parameters to eliminate this problem. Photomacrographs at 5X showed the weld throat to be equal to the wall thickness. These samples are now being examined by Westinghouse. An attempt is being made to develop nondestructive test to ultrasonically measure the cross section of the weld metal.

Approximately one-half of the 72 kilograms of Nupac material, which was originally rejected for high gas content and low density, has been given a 1500 C hydrogen furnace treatment as the first step of recycling. In general, the gas content is being lowered in the range of 0.03 to 0.07 cc/g, although most of the results are above the 0.06 cc/g specified for Saxton.

Manager
Reactor and Materials Technology

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