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PROGRESS RELATING TO CIVILIAN APPLICATIONS
DURING NOVEMBER, 1958

by

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REPORTS RELATING TO CIVILIAN APPLICATIONS
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- BMI-1291 "Shielding-Research Area at Battelle", by Walter R. Morgan, Harold M. Epstein, James N. Anno, Jr., and Joel W. Chastain, Jr.
- BMI-1297 "Coating of Uranium Dioxide Powders With Metallic Tungsten Films", by Joseph H. Oxley, James F. Hannah, John M. Blocher, Jr., and Ivor E. Campbell.
- BMI-1298 "Constitution, Metallurgy, and Oxidation Resistance of Iron-Chromium-Aluminum Alloys", by Sam Alfant, Arthur A. Bauer, Walston Chubb, Ronald F. Dickerson, Edward J. Jablonowski, and Fred R. Shober.
- BMI-1301 "Progress Relating to Civilian Applications During October, 1958", by Russell W. Dayton and Clyde R. Tipton, Jr.

A-1

A. DEVELOPMENTS FOR ZIRCONIUM-CLAD FUEL ELEMENTS

F. R. Shober

The effect of irradiation on the thermal conductivity and electrical resistivity of uranium and UO_2 is being investigated. A shipment of unirradiated unclad uranium specimens has been received. The thermal-conductivity measurements of unirradiated UO_2 and modifications to the apparatus to permit measurement of irradiated specimens are being continued.

The creep properties of 15 per cent cold-worked Zircaloy-2 are being investigated in the 290 to 400 C temperature range for times exceeding 10,000 hr. Additional specimens have been prepared for an investigation of the effect of a periodic temperature cycle from 290 C to room temperature to 290 C on the creep properties of the cold-worked Zircaloy-2.

The density distribution of crushed graphite is being investigated by the sink-float method. The density distribution appears to be a function of the graphite particle size. A separation of highly crystalline particles of nearly theoretical density from the extremely-low-density particles may be possible by this method.

Thermal Conductivity of Uranium and UO_2

C. F. Lucks and H. W. Deem

Uranium

Unclad unirradiated uranium specimens have been received for thermal-conductivity measurements, but experimental work has not been started. Some changes in apparatus are necessary because of a change in specimen size. Measurements on these specimens will start next month.

Uranium Oxide

During the past month, work continued on adapting the thermal-conductivity apparatus for measurements on irradiated specimens. The apparatus was modified so that the thermocouples could be positioned by the manipulators at the hot cell. This modification makes it possible to withdraw the thermocouples far enough to change the specimens. A calibration run was made on a titanium-alloy specimen to check out the thermocouple modification. The calibration checked with the former run made on the same specimen. UO_2 Specimen 1000 (unirradiated) is being measured, but the first points have not yet been calculated.

Work will continue on Specimen 1000 during the next month. A medium-density UO_2 specimen will then be measured.

A-2

Modifying the thermocouples for remote operation completes almost half of the work necessary to adapt the apparatus for the hot cell. Provisions for guiding the specimen into place will be added to the bottom of the apparatus. Hooking up the auxiliary equipment should complete the assembly.

Receipt of irradiated UO_2 specimens is expected in the next couple of months.

Mechanical Properties of Zirconium Alloys

F. R. Shober and J. A. VanEcho

A program is in progress in an effort to determine and evaluate the stress-rupture and creep properties of 15 per cent cold-worked Zircaloy-2 in vacuum in the 290 to 400 C temperature range. Most of the 12 creep units presently in use involve tests which have been in progress approximately 9000 hr. It is planned to continue these tests to failure or to total times exceeding 10,000 hr.

Additional annealed and cold-worked Zircaloy-2 creep specimens have been prepared for testing in the same temperature range. Some will be tested under cyclic temperature conditions similar to those expected during service. Of the remaining specimens, some will be tested at 290 and some at 345 C for comparison with the 15 per cent cold-worked material.

Physical Distortion of Graphite

J. Koretzky, W. C. Riley, and W. H. Duckworth

Research to develop a method of sink-float density measurement to identify factors affecting irradiation-induced volume changes in graphite was continued.

Sink-float density distributions were obtained for three different samples of minus 40 plus 60-mesh TS-GBF graphite. Results indicate good reproducibility in the experimental method. In addition, a nearly identical density distribution was obtained from a sample of minus 100 plus 140-mesh TS-GBF graphite.

The density distribution of a minus 325-mesh sample obtained by the standard method is recorded in Table A-1.

The reproducibility of the method for the finer particle sizes will be determined. The density distributions of the intermediate size ranges will be obtained. Several size ranges will be combined to reduce the number of runs for each bar sample.

A-3 and A-4

TABLE A-1. DENSITY DISTRIBUTION OF MINUS 325-MESH GRAPHITE

Specific Gravity of Benzene-Bromoform Solution	Amount Floated, g	Amount Floated, w/o	Cumulative Amount Floated, w/o
1.533	0.0035	0.67	0.67
1.618	0.0136	2.62	3.29
1.763	0.0071	1.37	4.66
1.838	0.0052	1.00	5.66
1.899	0.0239	4.61	10.27
1.964	0.0192	3.70	13.97
2.015	0.0344	6.63	20.60
2.100	0.0532	10.26	30.86
2.174	0.1061	20.46	51.32
2.232	0.0929	17.91	69.23
2.246	0.1509	29.11	98.34
2.260	0.0086	1.66	100.00

Preparation of Molybdenum Single Crystals

J. A. DeMastry, F. R. Shober, and R. F. Dickerson

A modified Andrade furnace is being used to grow single crystals of high-purity molybdenum for use in irradiation studies. Single crystals are grown by establishing a temperature gradient along the length of the specimen. This gradient is obtained by resistance heating 1/8-in. -diameter molybdenum rods to about 2200 C and water cooling the ends.

Preliminary investigations indicated that if a power input of 240 amp at 2.5 to 3.0 v is maintained for 5 hr, a single crystal approximately 2 in. long can be produced about 50 per cent of the time. Metallographic examinations of cross sections of finished rods established the existence of single crystals. Absence of grain boundaries in the cross section after etching was considered proof of a single crystal. Six acceptable single crystals have been grown to date. The failure of some rods to produce single crystals using the above technique indicates that purity and postfabrication history of the molybdenum influence single-crystal growth. An attempt will be made to grow six more crystals when a supply of suitable molybdenum rods on order is received.

When 12 molybdenum single crystals have been prepared, an attempt will be made to prepare single crystals of alpha zirconium. Two methods utilizing the Andrade furnace are proposed. A zirconium rod will be heated into the high-beta region and a beta-zirconium single crystal grown. It is hoped that this beta-zirconium crystal can be transformed into an alpha-zirconium crystal by reducing the specimen temperature to the high-alpha region and holding for some specific length of time. The second method will involve holding an 1/8-in. -diameter zirconium rod in the high-alpha range for several hours. A vacuum of less than 10^{-5} mm of mercury absolute pressure will be required for this study.

B-1

B. DEVELOPMENTS FOR ALUMINUM-CLAD FUEL ELEMENTS

R. J. Carlson and N. E. Daniel

Centrifugal-casting techniques for the production of aluminum-35 w/o uranium castings in the form of hollow cylinders are being investigated. A device for rotating the molds on a roller bed has been devised in an effort to eliminate the eccentricity encountered in previous castings. It has been noted that structural changes occur during fabrication which appear to favor the formation of UAl_4 in the aluminum-35 w/o uranium alloys containing ternary additions. X-ray diffraction studies now in progress should substantiate this observation.

Preparation of Aluminum-Uranium Alloys

N. E. Daniel, E. L. Foster, and R. F. Dickerson

Among the first systems to be investigated for potential reactor-fuel alloys was the binary aluminum-uranium system. Among the reasons for this interest was the low thermal-neutron cross section of aluminum and the low cost for aluminum of relatively high purity. Alloys from this system containing up to 20 w/o uranium have since proved themselves as suitable fuels for low-temperature water-cooled water-moderated reactors. If the alloys of this system are to find more general application than they now enjoy, it is necessary that casting and fabricating techniques be developed for alloys containing greater amounts of uranium. A greater uranium content would permit increased fuel loadings, thereby decreasing fuel-handling costs and would also make the alloys more attractive for use in export reactors where the enrichment of the fuel is severely limited by law.

The two major problems encountered when casting aluminum-uranium alloys are segregation and porosity. At the present time, the only acceptable method of casting reasonably sound material involves the use of a hot top of considerable size in order to feed the solidifying casting. This necessitates recycling a considerable quantity of material. One method of casting which might overcome this difficulty is centrifugal casting. Among the advantages inherent in this method of casting is the substitution of centrifugal force for the feeder head now used.

Efforts to this time utilizing centrifugal-casting techniques to produce aluminum-35 w/o uranium alloy castings up to 18 in. long have appeared promising. However, as the castings are increased in length, evaluation becomes difficult because of the irregular surface of the interior wall. These irregularities are due to dross and to some extent the eccentric rotation of the centrifugal mold. In an effort to obtain more uniform castings, a device consisting of a steel cylindrical mold on a roller bed has been designed and made. This device is capable of handling molds up to 36 in. long without introducing any appreciable eccentricity into the castings. It is believed that the improved method of supporting the mold and the contemplated changes in the pouring technique will improve the quality of the castings. A series of castings now being prepared utilizing bottom-pour and tilt-pour techniques should clearly delineate the applicability of the centrifugal-casting technique.

Further studies are in progress on ternary aluminum-uranium alloys. Of the original 13 ternary additions, five (copper, gadolinium, nickel, sodium, and titanium) have been eliminated from further considerations by virtue of their unsatisfactory behavior during casting and/or fabrication. Of the series of alloys containing 3 w/o additions to the aluminum-35 w/o uranium, seven, containing germanium, lead, magnesium, niobium, palladium, silicon, or zirconium, have been submitted for X-ray diffraction analysis. It was noted by metallographic techniques that structural changes occurred in these alloys during fabrication. These changes suggest that fabrication favors the formation of UAl_4 , thereby decreasing the amount of aluminum matrix present in the alloys. X-ray diffraction should indicate the amount and presence of UAl_4 .

Future work will be concerned with preparing and evaluating 24- and 36-in. -long centrifugal castings. It is also planned to determine the effects of selected ternary additions on the castability of the aluminum-35 w/o uranium alloys. Further fabrication studies will be conducted on the alloys now being evaluated as well as on other alloys which appear to offer promise.

C-1

C. RADIOISOTOPE AND RADIATION APPLICATIONS

P. Schall

In the study of radioisotopes for quality control, research on the radiometric determination of magnesium and the activation analysis of a number of constituents of cement is continuing. The effort on the use of isotope exchange for calcium analysis has been discontinued because the slow exchange rate makes this approach impractical.

Irradiation and evaluation of chemical products formed from urea inclusion compounds is continuing. The major effort is being devoted to the characterization of urea-hydrocarbon systems.

A program on the investigation of applications of intrinsic radioisotopes in process streams was initiated during the past month. The effort thus far has been limited to a survey of specific systems to select promising areas for experimental evaluation. The survey effort will be continued and progress reported next month.

Development of Radioactive-Tracer Quality-Control System

J. E. Howes, T. S. Elleman, C. T. Brown,
D. N. Sunderman, and M. Pobereskin

The study of radioactive-tracer quality-control techniques applicable to the cement industry is still in progress.

Several experiments have been conducted to develop the neutron-activation procedure for the elemental analysis of cement. National Bureau of Standards cement samples were irradiated in the BRR along with weighed quantities of aluminum, calcium, manganese, potassium, silicon, and sodium compounds. The gamma spectra of the cement and the elemental standards were observed at various time intervals after the irradiation. From these data, the optimum conditions for the analysis of the cement were obtained. Quantitative estimation of each element was made by comparing the area under its most prominent gamma peak in the irradiated cement with its counterpart in the irradiated elemental standard. Present results indicate that quantitative determination of aluminum, calcium, manganese, and sodium may be possible. Silicon determination is less promising while potassium cannot be identified at all. Work is continuing to evaluate this technique.

The development of the radiometric procedure for the determination of magnesium in cement is continuing. It appears that conditions of the analysis, such as pH, excess phosphate concentration, and temperature, must be controlled closely to obtain reproducible results. Effort is being directed, therefore, to studying the effect of these factors on the accuracy of the analysis.

To reduce the time required for sample preparation for a radiometric magnesium analysis, an investigation was initiated to develop a method for the rapid separation of magnesium from the other elements by the use of ion-exchange techniques. In these

experiments, the cement sample was dissolved in dilute hydrochloric acid and passed through Dowex 50 cation resin. In initial runs, partial separation of the magnesium was accomplished. However, sufficient data are not available to determine if quantitative separation is possible.

Preliminary experiments on the feasibility of determining calcium in cement by isotopic exchange have been completed. Results indicate that development of such a procedure is not possible since the exchange between radiocalcium in solution and stable calcium in the solid phase takes place too slowly. In a period of 24 hr less than 1 w/o of the calcium exchanged.

During the past month, in discussions with Portland Cement Association Research Laboratory chemists concerning research on cement analysis and other problem areas in the quality control of cement, it was pointed out that there is a definite need for a method for the rapid determination of magnesium in cement and cement raw materials. The need arises from the fact that raw materials used in many cement plants throughout the country contain near the maximum permissible quantity of magnesium. Consequently, magnesium analyses must be run at several steps in the production of the cement to assure that its concentration does not exceed ASTM standards. Under present procedures, such analyses are quite time consuming.

Several other quality- and process-control problems were also discussed. It would be desirable to have analytical methods for calcium and magnesium which could be used in the field at the quarry or other source of raw material. An analytical system which could be applied in a continuous manner to a moving stream is also of interest. A number of mixing problems could be studied by tracer techniques and eventually reduced to a process-control system. Complexometric titrations for calcium might be improved by tracer techniques and become of greater value in quality control.

These problems and possible tracer applications will be considered and the potential for radiotracer improvement of present techniques will be evaluated. If the applications are promising, experimental studies will be initiated.

Radiation Chemistry of Inclusion Compounds

M. J. Oestmann, E. J. Kahler, and W. S. Diethorn

The chemical analysis of irradiated organic compounds and their urea complexes was continued in November.

Difficulties in the analysis of the stearic acid complex have not been resolved. Analysis of this complex has been terminated so that the entire effort can be devoted to the analysis of irradiated octane, dodecane, decane, and cetane, and their complexes. Amide formation observed in the irradiated cetane complex has been detected in the octane and decane complexes also. The formation of amide is believed to be a characteristic product of the radiolysis of the urea-hydrocarbon complexes. Further work will be required to determine the yields of amide and the nature of other radiolysis products.

C-3 and C-4

The entire effort in December will be restricted to the analysis of the four hydrocarbon complexes listed above. Preliminary results obtained to date will be verified and better values for the radiation yields of the major products of radiolysis will be determined.

D-1

D. PROCESSING OF FEED MATERIALS

E. L. Foster

National Lead of Ohio as operators of the Feed Materials Production Center has a continuing interest in the quality of fabricated uranium fuel slugs. Since one step in the production process is the vacuum-remelt and -casting operation, the physical factors involved in the solidification of the casting produced are of considerable interest. These factors are being studied with the objectives of determining their relative importance and to help resolve problems that may arise out of future casting needs.

Solidification of Uranium

E. L. Foster, C. K. Franklin, B. L. Fletcher,
and R. F. Dickerson

A study of the processes involved in the solidification of uranium castings in graphite molds is being made. The influence of pouring temperature, ingot configuration, and other casting variables on the physical soundness of unalloyed uranium is of primary interest. Since the freezing pattern of the casting and its subsequent soundness are dependent upon heat transfer, a mathematical model has been formulated to describe the heat flow in the metal and the mold and to indicate the rate at which the ingot solidifies. In addition to a theoretical treatment of the problem, temperature histories of the mold and ingot are being obtained experimentally. The thermal data gathered from the casting experiments will be used to refine and check the results calculated by means of the mathematical model.

A 76-cell mathematical model which represents the mold and the casting is being used. As it is the aim of the theoretical treatment to determine the transient heat flow in the metal-mold system, it is necessary that the thermal properties of the mold and of solid and liquid uranium be known. The thermal conductivity of solid uranium was measured in the temperature range from 100 to 950 C. The measurements were taken on the material in both the transverse and longitudinal directions with respect to the long axis of an unalloyed uranium casting. The thermal conductivity was found to increase as the temperature increased in each case. In the longitudinal direction of the ingot the conductivity of uranium increased from 0.27 w/(cm)(C) at 100 C to 0.50 w/(cm)(C) at 950 C. In the transverse direction, it increased from 0.26 to 0.43 w/(cm)(C). Hemispherical total-emissivity measurements were taken on oxidized uranium. A value of 0.53 was found to exist at 864 C and values increased with temperature to about 0.54 at 1000 C. Currently, hemispherical total-emissivity measurements are being taken on graphite and graphite coated with MgO mold wash. Emissivity values of graphite were found to be about 0.80 at 900 C and decreased with increasing temperature to 0.71 at 1350 C. MgO·ZrO₂-coated graphite gave emissivity values of 0.71 and 0.46 at 800 C and 1350 C, respectively. Again a decrease in the emissivity of the specimen surface with an increase in temperature was observed.

Specific-heat measurements of solid and liquid uranium will be obtained. Also, the thermal conductivity of liquid uranium and the thermal resistance of a layer of mold wash will be determined.

Temperature histories are being obtained using 100-lb uranium melts. Three melts have been poured into bare graphite molds using vacuum-induction melting techniques. The melts were poured at 1316 C and under a partial pressure of 100 μ . Thermocouples were placed in the mold to measure temperatures along the central axis of the metal, at the mold-metal interface, and along the mold surface. In addition, thermocouples were placed on the inner wall of the furnace shell and directly above the top of the ingot to measure environmental temperatures.

A temperature of 21 C was found to exist at the furnace jacket during the casting runs. Temperatures above the top of the casting reached a maximum of 249 C a few minutes after the pour. Along the outside mold surfaces, temperatures increased to 466 C, and inner mold-surface temperatures as high as 527 C were obtained. The thermocouples at the center line of the casting recorded temperatures of 1149 C in areas in the top portion of the metal.

Arrangements are being made to obtain temperature histories during casting of 100-lb melts at 1400 C using vacuum-melting techniques.

E-1 and E-2

E. DEVELOPMENTS FOR LMFRE

J. McCallum, R. W. Hardy, and C. L. Faust

Work continued on electrolytic oxide and electroless oxide coatings on Croloy 2-1/4. Samples of each coating were submitted for testing in liquid bismuth.

Relatively thick (0.002 in.) coatings of molybdenum metal were also prepared on Croloy panels by alternately electrodepositing molybdenum oxide and then reducing the deposit in hydrogen at 1050 F. The adherence of such deposits must be improved before they will be useful.

Electroless coatings on Croloy could not be reduced in hydrogen at 1050 F and were not affected by immersion in liquid bismuth (515 C) for 2-1/2 hr. Thermal-cycling tests (25 cycles between 1000 and 2000 F and 100 cycles between 750 and 1000 F) did not cause any physical changes in the deposits. Analysis of the electroless coatings has been started.

F-1

F. RESEARCH FOR AEC REACTOR DEVELOPMENT DIVISION PROGRAM

S. J. Paprocki and R. F. Dickerson

REACTOR MATERIALS AND COMPONENTS

R. F. Dickerson

Experimental work has been continued on the study of the effects of additions on the phase stability of oxides. New materials have been prepared and are currently being evaluated. Preliminary testing of the new high-temperature high-pressure die has been initiated and, although no attempt has been made to achieve extreme conditions, pressures of 50,000 atm were achieved. A new uranium oxide phase has been produced by heating U_3O_8 to 440 C at a 30,000-atm pressure.

Structure and pressure-composition-temperature studies of the zirconium-25 w/o uranium-hydrogen system show that the eutectoid temperature lies between 601 and 627 C. An isochore treatment of the data will be used to locate the eutectoid temperature with precision. Capsule irradiations are progressing according to plan.

The instrumented capsule containing Type 347 stainless steel specimens which indicated thermocouple failure after two ETR cycles has been opened and examined. It was obvious from an examination of the temperature monitors (lead) that a temperature of at least 630 F (melting point of lead) was attained. Examination of some of the aluminum inserts around the larger specimens indicated that aluminum had melted. On this basis it can be assumed that the thermocouple readings during operation were correct. The malfunctioning couples may have been caused by a water leak in the capsule. It is now postulated that the reason for the high temperatures attained may have been a change in the power density in the vicinity of the test hole. A nonlead capsule will be loaded into the same position, irradiated for one cycle, and returned to the hot cell for examination.

Valence Effects of Oxide Additions to Uranium Dioxide

W. B. Wilson and C. M. Schwartz

Experimental work was continued to determine the effect of additive oxides on the oxidation characteristics and phase stability of uranium oxide. The current objectives are:

- (1) To reduce the amount of additive required to achieve stabilization
- (2) To use stabilized oxide as an additive to ThO_2 in order to prevent the fragmentation that occurs with high uranium content

- (3) To obtain a more detailed knowledge of the role of massive additions to uranium oxide.

Previous work toward each of these objectives has been described. New materials have been prepared and are currently being evaluated.

High-Pressure High-Temperature Solid-State Studies

W. B. Wilson and C. M. Schwartz

Preliminary testing of a new die design has been initiated. This unit was devised to extend the pressure and temperature range beyond that of the right-circular-cylinder-type die equipment previously used. No attempt has been made as yet to achieve extreme pressures or pressure and temperature simultaneously with the unit. However, during calibration, pressures of 50,000 atm were reached without damage to any parts. The die has excellent characteristics with regard to insulation and ease of use.

Direct confirmation of the new uranium oxide phase has been obtained by heating U_3O_8 to 440 C in the Bridgman anvil pressure unit at a 30,000-atm pressure. The phase produced was identical to that produced in the right-circular-cylinder die under the same conditions during attempts to react U_3O_8 and BeO. The new phase did not react with the BeO and volatilized from the body when fired at 1650 C in air.

The new phase tentatively identified as "X" phase has been found at 30,000 atm as a single phase, as UO_2 plus X phase, and as U_3O_8 plus X phase when heated at various temperatures. Both the alpha and beta modifications of U_3O_8 have been found when U_3O_8 was held at 30,000 atm of pressure at ambient temperature. At temperatures from ambient to around 500 C, X phase has been found with either UO_2 or U_3O_8 . At higher temperatures, around 1000 C, it appears that temperature offsets the effects of pressure and only U_3O_8 has been found. The uranium-oxygen phase diagram would appear to be quite complex, with pressure introduced as a new degree of freedom, and the appearance of UO_2 obtained from U_3O_8 under pressure indicates that pressure can reduce U_3O_8 to form the more dense UO_2 structure in accord with the le Chatelier principle. A similar occurrence was found by Bridgman for SnO_2 , which transforms to SnO under pressure. The Bridgman-anvil type pressure unit offers a convenient method to determine pressure effects on the uranium-oxygen system.

Work will continue to calibrate the new die design and to determine its ultimate pressure capabilities after which it will be employed for synthesis purposes.

Fueled Zirconium Hydride Moderator

H. E. Bigony, A. K. Hopkins, and H. H. Krause

The fueled-moderator study has continued with the determination of additional hydrogen-absorption isotherms for the zirconium-25 w/o uranium alloy, and

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high-temperature X-ray diffraction patterns of hydrides of the 1 and 50 w/o alloys. Capsules containing the specimens of the 2 w/o alloy are undergoing radiation in the MTR at present.

Structure and Pressure-Composition-Temperature Studies

Isotherms for the zirconium-25 w/o uranium alloy were obtained at 601, 627, and 690 C. Sieverts plots of the data were used to define the boundary limits of single-phase and two-phase regions. The results are given in Table F-1, with the phase boundaries designated numerically, pending verification by X-ray studies.

TABLE F-1. PHASE BOUNDARIES IN THE ZIRCONIUM-25 w/o URANIUM ALLOY HYDRIDE SYSTEM

Phase Boundary	601 C		627 C		690 C	
	Hydrogen Absorption, cm ³ per g	Pressure, cm of mercury	Hydrogen Absorption, cm ³ per g	Pressure, cm of mercury	Hydrogen Absorption, cm ³ per g	Pressure, cm of mercury
1	30.0	0.059	31.2	0.103	35.0	0.395
2	(a)	(a)	55.9	0.103	58.8	0.398
3	(a)	(a)	65.0	0.122	77.3	0.670
4	123.2	0.059	123.8	0.127	127.9	0.695

(a) Only two phase boundaries found at this temperature.

At 601 C only two phase boundaries were detected while four boundaries were found at all of the other temperatures investigated (627 to 803 C). This result indicates that the eutectoid temperature lies between 601 and 627 C, and interpolation of linear plots shows it to be closer to 601 C. An isochore treatment of the data will be used to locate the eutectoid temperature with precision. Anomalies in the isotherm at 710 C are being checked by additional runs at temperatures which are slightly higher and lower than 710. Isotherms in the vicinity of 575 and 825 C are planned also.

The high-temperature X-ray diffraction patterns have been taken in the range 500 to 800 C for the zirconium-1 w/o uranium and the zirconium-50 w/o uranium alloys. Processing of the data to determine the phases present is under way, and additional X-ray patterns will be run to resolve some discrepancies which have been noted.

Radiation-Damage Studies

Capsules containing zirconium hydride and fueled specimens of zirconium-2 w/o uranium alloy hydride have been inserted in the MTR. The temperature of Capsule BMI-20-1 has been somewhat lower than the design temperature of 1500 F, and measures will be taken to increase the temperature by a change of position for the next reactor cycle. Capsule BMI-20-2 has been at temperatures between 1200 and 1300 F, which is slightly higher than was planned. The irradiation will be continued for eight to nine cycles.

Irradiation Surveillance Program on Type 347 Stainless Steel

F. R. Shober, A. W. Hare, F. A. Rough,
and R. F. Dickerson

The irradiation of AISI Type 347 stainless steel at ETR process-water temperature, about 120 F, and at 600 F and subsequent determination of irradiation damage are being done in support of the KAPL-33 loop to be installed at the ETR. The tensile, cyclic-strain fatigue, and impact properties are to be determined after exposures to an integrated fast-neutron flux of approximately 2×10^{14} nvt at 6-month intervals of exposure in the F-10 position for lengths of time up to 3 years. Based on recent fast-flux measurement (neutrons with energies greater than 1 Mev) these times will produce total integrated fast fluxes of approximately 1.2, 2.4, 3.6, 4.8, 6.0, and 7.2×10^{21} nvt. These values are somewhat lower than originally expected since it was thought that the integrated fast flux in the F-10 position would be about 3×10^{15} nvt.

Eight capsules containing either tensile and cyclic-strain fatigue specimens or impact specimens have been under irradiation at about 120 F in the F-10-NE and F-10-SE positions since June, 1958. It is planned to discharge two capsules in early January. The capsules will be returned to the Battelle Hot-Cell Facility, opened, and the specimens tested. The tensile and fatigue specimens are to be tested at room temperature and 600 F. The impact specimens will be tested at room temperature only.

The core filler piece fabricated at Battelle has been inserted in the F-10 position. This piece has slots on each face and a hole in the center for dosimeters. It is thought a detailed flux map for the F-10 position can be determined using all or a portion of these dosimeter positions. It is planned to load the center hole with eight nickel and eight ammonium sulfate dosimeters enclosed in quartz tubes. Eight other nickel dosimeters will be placed in a hole on one face and one nickel dosimeter each will be positioned in the remaining holes in the faces of core filler piece. Evaluations of these dosimeters after one ETR cycle should provide fast-flux information at eight horizontal and two vertical planes of the F-10 position. Determinations of fast flux will be based on threshold energies of 3.2 and 2.5 Mev for nickel and sulfur, respectively. This first set of measurements will provide information on the flux distribution and flux spectrum in the F-10 position and indicate the stability of quartz as a capsule material. It is planned to ship these dosimeters to the ETR early in December, 1958, for loading in late December. It is expected that these measurements will be made just prior to movement of this experiment to another test hole in the ETR. The same measurements will be repeated after the anticipated move has occurred.

The instrumented capsule, BMI-27-17, containing tensile and cyclic-strain fatigue specimens, four Chromel-Alumel thermocouples, and four low-melting metals and alloys was returned to the Battelle Hot-Cell Facility and inspected after two cycles in the ETR. The examination was made after unexplainable high temperatures (1000 to 1200 F) were indicated in the capsule during irradiation. The capsule had been designed to produce temperatures of approximately 600 F by gamma heating. Since the capsule was representative of seven other nonlead capsules in design and construction, it was imperative that the validity of the temperatures indicated be established. The

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appearance of the outside of the capsule was bright except for one small burnished area over the junction of the sheathed thermocouple to the cable. There is no apparent explanation for the indication of the high temperature in this zone. Two flaws and places for possible leaks were observed in a weld which sealed the upper chamber of the capsules. The capsule had two separately evacuated and sealed sections. One section contained the specimens and the other the thermocouple-lead connections. These flaws were in the region of the thermocouple connections. The one flaw appeared to have been the result of welding technique while the other appeared to be a hairline crack at the base of the V-notch prepared for welding. In either case water could have leaked into the section containing the thermocouple leads. Upon opening this lead section of the capsule, the condition of the thermocouple and the interior of the capsule indicated water had been present during irradiation.

Examination of the section of the capsule containing the specimens was made by first puncturing a hole in the bottom of the capsule and obtaining a sample of the released gas for introduction into a helium mass spectrographic leak detector. A strong signal indicated that the gas released was helium, which substantiated other evidence that the lower section of the capsule containing the specimens had remained sound. The capsule was cut apart and the interior examined. The stainless steel sample and the aluminum inserts were discolored slightly. Examination of the temperature-monitor capsule to which powdered lead had been added prior to irradiation showed evidence that melting and solidification of the lead had occurred. This definitely established that a temperature of at least 630 F was obtained in the capsules. Further examination of the aluminum inserts surrounding the stainless steel sample showed evidence that a small portion of the aluminum in contact with the larger specimens had been molten. Since aluminum melts at 1267 F it appears that the temperatures recorded during irradiation are valid. The aluminum in contact with the smaller specimens showed no evidence of having melted, but had oxidized. It now appears that malfunctioning of thermocouples did not occur until after the thermocouples had recorded temperatures of about 1100 F. Erratic temperature measurements occurred after water leaked into the section containing the thermocouple connections.

A thorough investigation of the capsule design has not disclosed any reason for the capsule behavior. The only other explanation is that a change in power density occurred in the vicinity of the F-10 position as a result of xenon oscillation and flux "tilting" in the reactor. This effect has been found in other reactors and has been found to increase power in localized areas by a factor of ten.

It is planned to load a nonlead capsule in the same position and irradiate it for one cycle. It then will be returned to Battelle for examination. The temperature monitors and the aluminum inserts will be examined to see if melting has occurred. If melting has not occurred at either location it will be assumed that this type of high-temperature excursion is not a common occurrence and the remaining six nonlead capsules will be loaded. A third instrumented capsule will be constructed and sent to the ETR for temperature monitoring of the test position.

STUDIES OF ALLOY FUELS

R. F. Dickerson

The results of initial fabrication studies show that the cast structure of the niobium-10 w/o uranium alloy can be successfully broken up by hot rolling at 2400 F and that the resultant sheet is ductile. The nonuniform deformation of the niobium-30 w/o uranium and the niobium-20 w/o uranium alloys indicates that a rolling temperature higher than 2400 F may be required. A higher rolling temperature will be required for the alloys containing 60, 50, and 40 w/o uranium.

Improved metallographic techniques which have been developed for the thorium-uranium alloys show that arc melting produces much better material than does induction melting. The included uranium is much better dispersed and the material contains appreciably less oxide. Corrosion testing in 200 C water is in progress and the lowest corrosion rate to date (39.8 mg per cm² after 168 hr) was exhibited by a thorium-25 w/o uranium-25 w/o zirconium alloy. Niobium additions apparently have very little effect on the corrosion resistance of these alloys. Zirconium additions increase the hardness at temperatures below 400 C and tend to decrease the softening temperature. Quaternary additions of niobium tend to hold the softening temperature to around 550 C.

Development of Niobium-Uranium Alloys

J. A. DeMastry, F. R. Shober, and R. F. Dickerson

Alloys of uranium and niobium are being considered as possible high-temperature reactor fuels. A limited amount of work has been conducted on the alloys of the niobium-rich portion of the uranium-niobium phase diagrams. An evaluation of the available data indicates that the properties of uranium-niobium alloys are sensitive to the purity of both the uranium and niobium used in alloying. The effects of the major impurities, oxygen and zirconium, on the physical and mechanical properties, fabrication behavior, and corrosion life in water, sodium, carbon dioxide, and air are being investigated as part of this program.

The effects of impurities are being determined by employing three grades of niobium as base materials: high-purity niobium metal containing less than 300 ppm oxygen and less than 100 ppm zirconium, a low-zirconium grade of niobium containing 700 ppm oxygen and 0.17 w/o zirconium, and a high-zirconium grade of niobium containing 600 ppm oxygen and 0.7 w/o zirconium.

Evaluation of hot-hardness data suggests that alloys containing more than about 70 w/o niobium may be fabricated in the same way as unalloyed niobium. If the cast structure can be broken down by hot or cold rolling, the alloys may then be cold rolled if ordinary care is taken. Alloys containing less than 70 w/o niobium apparently will have to be fabricated at very high temperatures (2600 F), if at all.

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Alloys containing 70, 80, and 90 w/o niobium prepared from both the high and low zirconium-bearing niobium have been melted twice by consumable-electrode arc melting. The remaining alloys containing 40, 50, and 60 w/o niobium prepared from all three grades of niobium, and the 70, 80, and 90 w/o niobium prepared from high-purity niobium, have been melted once and are being sectioned for remelting.

An evacuated Type 304 stainless steel pack containing buttons of 40, 50, 60, 70, 80, and 90 w/o niobium wrapped in 0.005-in. molybdenum foil was hot rolled at 2400 F. The uranium-90 w/o niobium alloy was successfully reduced 60 per cent, and the resultant sheet appears to be quite ductile. The uranium-70 and -80 w/o niobium alloys deformed in a nonuniform fashion during rolling and appear to be segregated. A higher rolling temperature is indicated. The uranium-40, -50, and -60 w/o niobium alloys broke through the molybdenum foil and alloyed with the stainless steel pack. Evaluations of these results indicate that a canning material such as molybdenum is needed so that higher rolling temperatures may be used.

Individual molybdenum packs containing 1/4-in. circular wafers of each alloy are being prepared. These packs will be rolled at temperatures above 2600 F. At the same time, in order to obtain more information about the fabrication characteristics of uranium-niobium alloys, small test buttons will be cold and warm (1472 F) rolled without any jacketing material.

Development of Thorium-Uranium Alloys

V. W. Storhok, A. A. Bauer, and R. F. Dickerson

Thorium-uranium-base alloys are the subject of an investigation aimed at improving irradiation stability and corrosion resistance by ternary alloying and control of processing techniques. Molybdenum and niobium additions are being made with the intention of stabilizing the gamma-uranium phase, while zirconium is being added to strengthen the thorium matrix.

Metallographic examinations of as-cast binary alloys containing up to 20 w/o uranium indicate a tendency for the uranium to deposit at the grain boundaries in induction-melted alloys. Uranium particles in the arc-melted alloys are more uniformly dispersed throughout the grains. Also, there appears to be more oxide present in the induction-melted alloys. There is a possibility of oxygen and zirconium pickup from the zirconia crucible during melting. Because of these results it seems preferable to prepare thorium-uranium alloys by arc melting, both from the viewpoint of contamination and resultant structure. With respect to the latter point, when uranium is precipitated in the grain boundaries it will act as the matrix phase; the resultant alloy can consequently be expected to exhibit the undesirable irradiation-growth characteristics of uranium. These alloys are being further studied to determine the effects of heat treatment on microstructure and also to determine recrystallization temperatures.

Alloys containing zirconium additions are being corrosion tested in 200 C water. The lowest corrosion rate observed to date was for a water-quenched thorium-25 w/o

uranium-25 w/o zirconium alloy which has a weight loss of 39.8 mg per cm² after 168 hr of exposure. Other alloys exhibited weight losses at least three times as great. Water-quenched alloys generally have lower corrosion rates than similar furnace-cooled specimens. There is a possibility that this may be the result of some beta thorium being retained by quenching. The microstructures of these alloys are being investigated, and X-ray diffraction patterns will be obtained when indicated. Niobium additions apparently have little effect on the corrosion resistance of these alloys.

Hot-hardness data have been obtained for several ternary and quaternary alloys. Zirconium additions increase hardness at temperatures below 400 C and lower the softening temperature from 500 C for a thorium-10 w/o uranium-10 w/o zirconium alloy to 400 C for a thorium-25 w/o uranium-25 w/o zirconium alloy. Similar alloys containing quaternary additions of niobium all exhibit softening temperatures around 550 C.

In addition to the studies now being conducted, thermal-analysis data and the transformation kinetics of some of the alloys are to be investigated.

GENERAL FUEL-ELEMENT DEVELOPMENT

S. J. Paprocki

Cermet fuel materials consisting of from 60 to 90 volume per cent of UO₂, UN, or UC dispersed in stainless steel are being investigated. These materials are promising fuels, as they combine some of the advantages of both the ceramic and dispersion fuels. The materials have been prepared by swaging, hot press forging, and pressure bonding. Densities exceeding 90 per cent of theoretical have been obtained by hot press forging and pressure bonding.

The gas-pressure-bonding process is being investigated for the cladding and bonding of niobium and molybdenum-base fuel elements and subassemblies. Optimum bonding has been achieved at 2100 to 2200 F for molybdenum and 2100 to 2300 F for niobium. As the conditions for the self-bonding of these materials are being established, studies have been initiated to bond niobium- and molybdenum-base cermet fuel materials prepared under the program "Fabrication of Cermet Fuel Elements".

The gas-pressure-bonding process occurs in several stages. In the initial stage, deformation occurs to bring the surfaces into intimate contact. At this point, surface phenomena and diffusion govern completion of the bonding cycle. Studies are in progress to obtain a better understanding of these processes in order that the optimum bonding conditions for any material can be related to its physical and mechanical properties.

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Fabrication of Cermet Fuel Elements

S. J. Paprocki, D. L. Keller, G. W. Cunningham,
and D. E. Kizer

Fabrication techniques for producing dense cores containing 60 to 90 volume per cent ceramic fuel dispersed in a metal matrix are being evaluated. These techniques include hot pressing and pressure bonding and in some cases intermediate swaging. Evaluations are being made on the basis of microstructures and physical and mechanical properties.

A 1-in. -square specimen of 80 volume per cent UO_2 dispersed in niobium has been hot press forged to a density of 93 per cent of theoretical at 2100 F. Also, six 1-in. -diameter cores of 80 volume per cent UO_2 dispersed in molybdenum have been hot press forged at 2100 F to densities ranging from 91 to 95 per cent of theoretical. Transverse and longitudinal metallographic sections of the UO_2 -molybdenum and UO_2 -niobium cores indicate a continuous metal skeleton. Transverse sections (parallel with the direction of pressing) show an elongation of the UO_2 particles in a direction perpendicular to the direction of pressing while the longitudinal section shows the UO_2 randomly distributed.

In addition, six hot-press-forged cores of 80 volume per cent UO_2 -Type 302B stainless steel have been fabricated at 1900 F and machined to 1 by 1 by 0.090-in. specimens having a density of 96 per cent of theoretical. These cores are being assembled in a stainless steel frame for pressure-bonding studies. Also, six green 80 volume per cent UO_2 -stainless steel cermets are being assembled in an identical frame for pressure bonding.

Three 1/2-in. -diameter cores of 100 per cent UO_2 , 90 volume per cent UO_2 , and 60 volume per cent UO_2 in stainless steel having respective green densities of 77, 75, and 72 per cent of theoretical have been pressure bonded in sealed stainless steel tubes under a pressure of 10,000 psi for 3 hr at a temperature of 2100 F. Core densities after pressure bonding were respectively 81, 82, and 91 per cent of theoretical. The pressure-bonded cores were free of cracks in all cases. The 60 volume per cent UO_2 core was sound and intact after removal of the cladding by machining. The outside diameter of the tubes over the core area after pressure bonding was uniform and varied only 0.003 in. on the 60 volume per cent UO_2 core.

In addition, a 1/2-in. -diameter core of 80 volume per cent UO_2 dispersed in nickel has been sealed in a stainless steel tube and swaged at 1800 F. After a 33 per cent reduction in area the density of the core had increased from 73 to 82 per cent of theoretical. No difficulty was encountered during swaging. In addition, transverse and longitudinal electrical-resistivity measurements are being made on hot-press-forged cermets. Also, high-density cermet cores are being prepared for determination of thermal conductivities.

Gas-Pressure Bonding of Molybdenum- and Niobium-Clad Fuel Elements

S. J. Paprocki, E. S. Hodge, C. B. Boyer, and R. W. Getz

The gas-pressure-bonding process is being investigated as a method of fabricating fuel elements and assemblies clad with molybdenum and niobium. Both cladding materials retain their strength at elevated temperatures and possess favorable nuclear properties. Oxidation-resistant cladding is required if molybdenum and niobium are used in an oxidizing atmosphere; however, in an inert gas-cooled or some liquid-metal-cooled reactors oxidation-resistant coatings on molybdenum or niobium are not required. There are, however, no available conventional fabrication techniques which are satisfactory for producing molybdenum- or niobium-clad fuel elements or assemblies. During conventional fabrication, the molybdenum and niobium generally alloy with the protective container materials, and distortion of the element and rupture of the protective container results from the difference in fabrication characteristics of the two materials before sufficient conditions can be achieved to affect a metallurgical bond. In gas-pressure bonding, only a minimum amount of deformation is incurred to achieve a solid-state bond; consequently, problems with fuel-element distortion and protective container ruptures are eliminated.

In the initial phase of this investigation, the bonding of molybdenum and niobium to itself is being studied. Flat-plate specimens of these materials were subjected to various surface preparations and bonded over a range of times and temperatures to determine preliminary bonding conditions. This study was conducted with materials of relatively low purity that were readily available. With these specimens, it was possible to determine preliminary bonding conditions necessary to achieve self-bonding of molybdenum and niobium. These conditions are being verified with high-purity material now available for this study.

Metallurgical bonds with molybdenum have been achieved by bonding at 2100 to 2200 F for 3 hr at 10,000 psi. The molybdenum bonds the most consistently at these temperatures if it is surface conditioned prior to bonding by pickling in a solution of 95 parts H_2SO_4 , 4.5 parts HNO_3 , 0.5 part HF , and 18.8 g per liter of Cr_2O_3 prior to bonding. This solution has to be held at 140 to 160 F to produce a clean pickling of the molybdenum.

Niobium specimens that were surface conditioned with a 20 per cent solution of HF prior to bonding yielded the most consistent bonds for niobium. Bonding parameters of 3 to 4 hr at 2100 to 2300 F at 10,000 psi produced the best niobium bonds.

Additional specimens are being prepared from high-purity double electron-beam melted niobium and high-purity arc-cast molybdenum. These specimens are being subjected to the most favorable bonding conditions determined for molybdenum during the preliminary study with the material of relatively low purity. A series of molybdenum and niobium specimens has also been prepared from annealed, 50 per cent cold-reduced, and 90 per cent cold-reduced material that has surface roughnesses of 40, 80, 125, and 250 rms to determine if conditions of prior fabrication and roughness have a real effect on bonding of these high-purity materials.

F-11

Specimens for producing molybdenum and niobium-clad high-loaded cermets and pure uranium dioxide are being assembled for bonding. These specimens contain one or two cores and are to be bonded by the bonding conditions determined in the preliminary studies of self-bonding.

Basic Studies of Pressure Bonding

S. J. Paprocki, E. S. Hodge, S. D. Beck,
M. A. Gedwill, and D. H. Fisher

This program is concerned with analyzing the process of bonding under gas pressure through a consideration of the deformations required to bring the mating surfaces into intimate contact. Due to the relatively low rate of deformation under moderate hydrostatic pressure, this aspect of the pressure-bonding problem has a critical significance. A necessary condition for bonding is that the surfaces be sufficiently close for the metallurgical phenomena of bonding to occur.

It has been postulated that the deformation of gas-pressure bonding may be represented by second-stage creep behavior under a combined-stress configuration. In principle, the creep behavior of a system may be expressed in terms of two creep parameters which depend only on the temperature and the material. Due to the intrinsic complexities associated with the analysis, however, only the most elementary geometries have known solutions. Specifically, for creep under hydrostatic boundary conditions only the cases for the thick-walled sphere and circular cylinder are known. Due to the more or less arbitrary shape of the surface irregularities, it is felt that using one of these shapes for the model of the surface geometry may be inadequate. The elliptic cylinder may be a more satisfactory choice since it represents more precisely the surface irregularities.

Attempts to solve the analytical problem of creep for the case of the elliptic cylinder have, thus far, been unsuccessful. If a solution cannot be found soon, the comparison of the analytical creep behavior with the experimental results to conclude this program will be based upon either the cylindrical or spherical model of the surface irregularities.

Values of the creep parameters for 2S aluminum at 500 and 700 F have been obtained from a series of torsion creep tests. These values have yet to be rechecked. These creep parameters will be used to predict the deformations of a thick-walled circular cylinder and the predicted values will be compared with previously obtained experimental data.

Factors Affecting Pressure Bonding

G. W. Cunningham and J. W. Spretnak

An investigation is being made of the process whereby solid-phase bonding of metals occurs under the application of heat and pressure. In the initial work,

copper-to-copper bonds and zirconium-to-zirconium bonds are being studied. Major emphasis is being placed on the mechanism which is in effect at intermediate and final stages rather than the initial rapid plastic deformation. Presently developed theories of creep, sintering, and hot pressing will be considered in establishing a theoretical concept of the bonding mechanism.

A hot-press die and a large number of 1/2-in. copper squares have been completed. The hot-press equipment and the vacuum system have been checked, and procedures for pressure bonding have been established. Other investigations have indicated that at high temperature and low stress, creep of copper wires and densification of copper powder during hot pressing can be described by the Nabarro-Heering creep mechanism which predicts material transfer by vacancy diffusion as a function of pressure or applied stress. However, at lower temperatures, hot-pressed compacts appear to densify more rapidly than predicted by this mechanism. The actual densification rate can be expressed as a power function of the applied pressure. By the use of specimens in which the initial surface area and grain size are approximately a constant value, it may be possible to establish whether both these theories apply to pressure bonding and if so at which temperature and pressure the self-diffusion becomes important enough for the Nabarro-Heering theory to become dominant. Porosity in poorly bonded specimens can be measured under the light microscope, and, although additional work is necessary, it may be possible to locate and measure very small pores with the aid of electron microscopy. Accurate density and electrical-conductivity measurements will also be investigated as a means of detecting or measuring porosity.

Additional work is also being conducted with bonded Zircaloy specimens which were previously used in a study to establish optimum bonding conditions. It has not been clearly shown whether areas which appear as voids in electron photomicrographs are voids or were formerly filled with impurities. Sections of a specimen previously evaluated as having a bond interface with 5 per cent complete grain growth with no contamination, 45 per cent grain growth with contamination, and 50 per cent bond with no grain growth across the interface have been cold rolled to 40, 50, 77, and 85 per cent reductions in thickness. Metallographic evaluation of the cold-rolled structures is not conclusive, but the manner in which the void or contaminated interface is elongated and closed tends to support the conclusion that voids are present in the poorly bonded specimens. Sections of these specimens are also being annealed at 1400 F and will be examined under the light microscope. Depending on the results obtained, the bond interfaces may be studied by electron microscopy.

G-1 and G-2

G. FATIGUE STUDIES OF INCONEL AND INOR-8

R. G. Carlson

Fatigue Studies of Inconel

This program has the objectives of obtaining basic fatigue information on Inconel and of establishing quantitative relationships among the variables of temperature, stress, strain, time, and cyclic frequency for this material. The current phase of the program is concerned with measuring and recording strain associated with the cyclic portion of a combined load.

During November stress-cycling tests were continued on Inconel specimens at 1500 F cycled at 1 cps. A plotter was constructed to develop hysteresis curves from the continuous stress-strain traces recorded for each test. The stress-strain traces and the hysteresis curves are being analyzed to determine the relationships between significant variables.

During December the investigation of the 1500 F 1-cps condition will be completed. The next condition to be studied will be 1300 F and 1 cps.

Fatigue Studies of INOR-8

This program is concerned with the investigation of the dependence of fatigue properties of INOR-8 alloy on temperature and frequency.

During November machining of the fatigue specimens was completed. Tests at 1500 F using the new material were begun at 100 and 10,000 rpm.

During December the accumulation of fatigue data will be continued for these conditions.

H-1

H. PHYSICAL RESEARCH

F. A. Rough

Two projects of physical research are reported this month: a study of uranium compounds and a study of the mechanism of migration of hydrogen in zirconium under a thermal gradient. Both projects are supported by the AEC Division of Research.

In the study of uranium compounds, sintered uranium monocarbide is being studied preparatory to irradiation and subsequent heating to evaluate gas-retention characteristics.

In the study of hydrogen migration in zirconium, the process is presently being considered from the viewpoint of the thermodynamics of irreversible processes. Experiments are in progress to determine the necessary diffusion coefficients needed to evaluate subsequent studies of hydrogen migration.

Preparation and Properties of
Refractory Uranium Compounds

A. B. Tripler, M. J. Snyder, and W. H. Duckworth

Work is under way on the preparation of sintered compacts of uranium monocarbide in which bulk density, porosity, and grain size are varied by varying initial particle size, compacting pressure, temperature of sintering, and time at temperature. The sintered compacts will be exposed to short-time low-temperature low-level irradiation in the BRR. A correlation will be sought between the physical properties listed above and fission-product release caused by laboratory heat treatment.

The initial particle size was varied in the following manner. One kilogram of arc-melted uranium monocarbide was crushed, passed through a 100-mesh sieve, and divided into two equal portions. Portion A was temporarily set aside, and Portion B was milled to an average particle size of 6.2μ . The $6.2\text{-}\mu$ material pressed satisfactorily at 100,000 psi in a hydrostatic press.

The relatively coarse fraction from the original crushing, Portion A, did not compact satisfactorily, and so was milled for short periods with compacting trials following each period of milling. Even when the material had been reduced to an average particle size of 14.7μ , it still did not compact satisfactorily, although some improvement was noted. Two possible courses of action were indicated here. The first was to use the two fractions at their present state of reduction, and to use a small amount of binder material in compacting. The second was to reduce the particle sizes of both fractions so that the one with the larger particle size would compact without a binder. A disadvantage in this procedure is the fact that, as the particle size becomes smaller, the milling efficiency decreases, requiring comparatively long milling periods, and excessive contamination of the carbide by the grinding media takes place. The first alternative was therefore chosen.

Furnace schedules have been established for the vacuum sintering of uranium monocarbide. Formerly, in bringing specimens up to temperature, the pressure was never permitted to go above 25 μ . (The rise in pressure is due to outgassing.) This required periods up to 4 days to bring a specimen up to temperature. In the present procedure the pressure is permitted to go as high as 100 μ , enabling specimens to be sintered in 1 day. The heating curves are fairly reproducible. Reproducibility of heating rate is necessary so as not to introduce an additional variable in the preparation of the sintered compacts.

The work on uranium nitride will commence during the next period with the preparation of material and determination of pressing characteristics.

Thermal Migration of Hydrogen in Zirconium

J. W. Droege, W. M. Albrecht, W. D. Goode, and H. H. Krause

Progress has continued on the theoretical analysis of the thermal-diffusion problem, and an apparatus for experimental work has been designed. Measurement of diffusion coefficients for hydrogen in beta zirconium is under way.

Thermal Diffusion

The equations for thermal diffusion, based on the thermodynamics of irreversible processes, have been modified to express the gradients in terms of temperature and hydrogen decomposition pressure. An apparatus has been designed for measuring the hydrogen migration in zirconium hydride under the influence of a thermal gradient. The plan is to measure the hydrogen pressures at two parallel faces of a zirconium hydride specimen, the two faces being maintained at different temperatures. The advantages of this method over one based on concentration measurements stem from the fact that the specimen will not be destroyed in the course of each measurement. Thus it will be convenient to use a thin specimen which will come rapidly to the steady state; the approach to the steady state may be observed, and the temperature and concentration may readily be varied. Since the equilibrium hydrogen pressure is known as a function of concentration and temperature, the results may be interpreted in terms of either the activity of hydrogen or its concentration.

Diffusion Coefficients

Studies of the rates of diffusion of hydrogen in beta zirconium and delta zirconium hydride are being made. Diffusion coefficients for hydrogen in the hydride have been determined and reported.

It was indicated in preliminary experiments that the rate of hydrogen diffusion in beta zirconium is very rapid. A diffusion coefficient of the order of 10^{-4} cm² per sec was found at 700 C. In view of the rapidity of diffusion at this temperature, it was concluded that diffusion coefficients for the temperature range 600 to 900 C could best be

H-3 and H-4

determined by a technique based on permeation measurements. The proposed technique is based on the time-lag method developed by Daynes* and Barrer**. Essentially, this method involves measuring the time lag, L , in establishing a steady-state flow of gas through a permeation membrane. The diffusion coefficient, D , is determined from the equation

$$L = \frac{a^2}{6D},$$

where a is the thickness of the membrane.

An apparatus required for permeation studies has been constructed and calibrated. The apparatus consists of three principal sections; a source of pure hydrogen, a permeation cell, and a calibrated collection volume. Pure hydrogen obtained from the thermal decomposition of UH_3 can be supplied at constant pressure to the upstream side of the permeation cell. The permeation cell consists of a zirconium membrane clamped in a machined heavy-walled stainless steel holder.

The downstream side of the permeation cell is connected through a manostat to a diffusion pump so that any desired pressure may be maintained on this side of the zirconium membrane. The diffusion pump is connected in turn to the calibrated collection volume, which is equipped with a McLeod gage for measuring the amount of gas collected with time.

Since the hydrogen pressure on both sides of the zirconium membrane may be controlled, it is possible to determine diffusion coefficients over small concentration gradients. By varying the pressures of succeeding experiments, it is possible to determine the effect, if any, of concentration on the diffusion coefficient.

A few experiments have been made to establish the techniques of operation of the apparatus. Permeation data have been obtained at 700 and 800 C with pressure differences of 3 to 5 mm of mercury across 0.06 to 0.08-cm zirconium membranes. The diffusion coefficients determined from these experiments were in the order of 10^{-6} to 10^{-5} cm^2 per sec. These values are lower than would be expected from consideration of the adsorption rates. Therefore, samples of the zirconium membranes used in the experiments are being analyzed to see if there is oxygen picked up during the experiments. Also, the experimental techniques are being modified to eliminate possible contaminants from the system more effectively.

* Daynes, H., Proc. Royl Soc., 97A, 286 (1920).

** Barrer, R. M., Trans. Faraday Soc., 35, 628 (1939); Phil. Mag., 28, 148 (1939); Diffusion in and Through Solids, Cambridge University Press (1951).

I-1

I. SOLID HOMOGENEOUS FUELED REACTORS

W. H. Goldthwaite

Fuel elements for a proposed pebble-bed reactor are being investigated in a radiation-effects program at Battelle. Several types of 1-1/2-in.-diameter fueled graphite spheres containing 10 w/o of fully enriched UO_2 are being evaluated before and after irradiation in the BRR.

Encapsulation and Irradiation

J. H. Stang and G. E. Raines

The irradiation of a capsule containing six fueled graphite spheres of three types continued during November in a core position of the Battelle Research Reactor with temperature levels substantially the same as reported in BMI-1302 (1000-1200 F). The decline of temperatures for the initial 40 or 50 hr that was noted during the first reactor cycle was repeated during the second and third reactor cycles. This is probably attributable to a combination of: (1) fission-poison buildup in the reactor, and (2) fission-poison buildup in the capsule itself. While a general reduction of flux throughout the reactor as poisoning is incurred is prevented by withdrawing the shim rods, none of these rods are in close proximity to the space where the irradiation took place. Thus, it is possible that in the local area shimming did not prevent a lowered flux. The poison buildup in the capsule is believed to have been an equally important factor. This is corroborated by the fact that the decline continued for only 40 to 50 hr, which approaches the length of time required for equilibrium buildup of xenon-135, an isotope having a relatively short half-life. The 2-day shutdown between cycles was sufficiently long for decay which accounts for the fact that temperatures immediately after the cycle startups were substantially the same.

After the initial decline, a very gradual temperature increase (25 to 50 F for 200 hr) continued until the end of each cycle. This increase was probably due to the gradual withdrawal of a regulating rod in a position adjacent to the capsule to compensate for burnup.

The irradiation will continue until December 1, 1958, and examination of the various specimens in the hot cell is scheduled to follow immediately. The predicted burnup (1.5 per cent of the uranium-235 atoms initially present) was erroneously reported in BMI-1301 as being based on an irradiation of 40 hr; the time should have been 540 hr.

Postirradiation Examination

R. J. Burian and J. E. Gates

Equipment is being readied for the postirradiation examination of six spherical specimens composed of UO_2 dispersed in graphite. The examination is to include visual inspection, photographs, measurements of dimensions and dry weights, and abrasion, impact, and compression tests. Dosimeters from the capsule will be analyzed to obtain flux data. Samples for fuel-distribution analyses will be drilled from selected specimens.

J-1

J. CORROSION PROBLEMS ASSOCIATED WITH THE RECOVERY OF SPENT REACTOR FUEL ELEMENTS

C. L. Peterson, P. D. Miller, O. M. Stewart, W. C. Baytos,
J. D. Jackson, T. E. Snoddy, and F. W. Fink

Materials of construction for use in the various proposed processes for the recovery of spent reactor fuel elements are being evaluated.

Some localized attack has been observed after prolonged exposure of titanium steam tubes to Initial and Beginning Darex solutions. The extent and cause of this have not yet been determined.

Heat treating stabilized Ni-o-nel, prior to welding, has improved the resistance of weldments to Initial Thorex solution. Other heat treatments will be studied.

Localized attack at the vapor-liquid interface occurs with a HF flow of 10 g per hr in the equimolar NaF-ZrF₄ salt. Very low corrosion rates were found with a possible final concentration of this salt when 2.4 w/o hydrogen was added to the HF sparge.

The Darex Process

Uranium can be recovered from fuel elements containing stainless steel as a diluent, or as a cladding, by means of the Darex process. The elements are first dissolved in dilute aqua regia. The chlorides are stripped from the solution with concentrated nitric acid, and, following suitable adjustment, the dissolved uranium is recovered by conventional methods of solvent extraction.

Dissolver Studies With Titanium

Inspection after 660 hr of the titanium steam tube, exposed to Initial Darex solution with a steam temperature of 135 C, showed no attack except for some fissuring around the interface area on one leg of the hairpin-shaped tube. Metallographic sections of this tube have been taken to determine if this has been caused by segregation of impurities in the metal.

Some general surface roughening was found on the submerged portion of a titanium steam tube exposed to Beginning Darex solution for 1040 hr. Rather deep crevices were noted in the heat-affected areas in and around the weldment. Possibly these crevices are associated with weld porosity and were merely opened and enlarged by the acid exposure. This will be determined by metallography.

The tube exposed to the Middle Darex solution was found to be virtually unattacked when examined after 1068 hr and was returned for continued exposure.

The Sulfex-Thorex Process

The evaluation of various metals and alloys for use as a container material for the dissolution step of the proposed Sulfex-Thorex process has continued. In this particular process, stainless-clad fuel elements of thorium or thoria would be dejacketed by dissolution in sulfuric acid. Following this, the thorium or thoria would be dissolved by a solution of 13.0 M HNO_3 , 0.05 M F^- , to give a final solution of about 8.5 M HNO_3 , 0.05 M F^- , and 1.0 M $\text{Th}(\text{NO}_3)_4$. Experience has shown that the addition of 0.2 M Al^{+3} to the Thorex solutions will aid in preventing excessive corrosion of several materials. Such an addition will probably be made during operation of this process.

Experiments With Carpenter 20 Cb

No experiments were conducted this month with Carpenter 20 Cb.

Experiments With Ni-o-nel

Stabilized Ni-o-nel was heat treated 2 and 10 hr at 1650 F and air quenched, prior to welding. Specimens cut from this welded material have been exposed to boiling Initial Thorex solution for a total of 288 hr. The corrosion rates have been about 1.5 mils per month, or less, and the outward appearances are very good. Isolated areas of localized attack along the edge of the weldment, more prevalent on specimens from the 2-hr heat, have been observed. However, none of these areas show very severe attack and exposure is being continued.

Specimens of stabilized Ni-o-nel, welded by the manufacturer, were heat treated for 1 and 24 hr and air quenched. Exposure of these specimens to boiling Initial Thorex solution resulted in a serious localized attack on the weldment. The exposure was halted after 96 hr, with corrosion rates having reached 2.5 to 3.4 mils per month. The composition of the weld metal is now being determined.

A heat treatment at 1850 F for 0.5 hr, followed by an air quench prior to welding, will be evaluated for stabilized Ni-o-nel next.

The corrosion rates of welded specimens of stabilized Ni-o-nel are being determined both in refluxing 6 M H_2SO_4 and in a similar refluxing solution in which batch dissolutions of Type 304 stainless steel are being carried out. These specimens were heat treated at 1850 F for 0.5 hr and air quenched following welding. After 122 hr of exposure, the corrosion rates of the specimens exposed to the vapors above the dissolution were about two to three times as great as the 0.8 mil per month measured during straight refluxing. Specimens exposed to the liquid had about the same rate under either condition: 2.4 and 2.5 mils per month with dissolution, 2.2 and 2.3 without. A liquid-phase specimen in contact with the dissolving stainless rods received protection and showed a rate of only 0.6 mil per month. This evaluation is being continued.

J-3 and J-4

Scouting Experiments With Other Metals

The producers of Illium alloys have suggested that niobium-stabilized Illium 98 be evaluated for the Sulfex-Thorex process. They have also suggested evaluating some of their present production of Illium R using a heat treatment following welding of 1 hr at 2150 F and water quenching. Scouting experiments are planned with these alloys as they are made available.

The Fluoride-Volatility Process

Fuel elements containing zirconium as a diluent or cladding can be recovered by a fluoride-volatility process. The first step consists of hydrofluorination of the elements in a bath of molten fluoride salts using an HF sparge. Various materials are being evaluated for use in the construction of this hydrofluorinator.

A series of runs at 650 C has been completed with the equimolar NaF-ZrF₄ salt. The effect of a HF flow rate of 10 g per hr on specimens at the liquid-vapor interface position has been studied. A sparge of HF alone produced a localized attack at the interface on both INOR-1 and INOR-8 specimens. A 20-ppm oxygen contamination added with nitrogen as a diluent to the sparge produced attack at the interface, but not so much as occurred with HF alone. Hydrogen addition, simulating the 2.4 w/o expected in the effluent, inhibited the localized attack at the interface, as mentioned in BMI-1294. An additional run in this series with HF alone will be attempted at 700 C to investigate the effect of temperature on the corrosiveness of the equimolar salt. It is hoped that this experiment will help establish the importance of temperature and salt composition on the attack at the interface.

A run was completed at 650 C with a salt representative of a possible final composition in the NaF-ZrF₄ system. This composition was 43.5 NaF- 56.4 ZrF₄-0.11 UF₄ (mole per cent). A HF flow of 10 g per hr + 2.4 w/o hydrogen was used during the 199-hr exposure.

All of the corrosion rates were found to measure less than 1 mil per month and no selective attack nor crystalline or metallic deposits were observed with low-power magnification. No serious corrosion difficulties are indicated from this composition with hydrogen present. Flow rates for HF of 10 and 25 g per hr, without hydrogen, are planned for the next two evaluations in this salt.

K-1

K. DEVELOPMENTS FOR SRE, OMRE, AND OMR

J. E. Gates and F. A. Rough

Work reported in this section is part of the Atomics International research program. The objectives of this research are to develop uranium monocarbide as a fuel for the SRE and to perform postirradiation studies of materials of interest to the SRE, OMRE, and OMR programs.

EVALUATION OF URANIUM MONOCARBIDE AS A REACTOR FUEL

F. A. Rough

Experiments are being conducted to determine the suitability of uranium monocarbide as a fuel for the SRE. Two capsules of specimens have been irradiated and examined, and a third is presently in the MTR. Four additional capsules are in various stages of preparation for irradiation.

Reports of progress are included in the irradiation, postirradiation-examination, and gas-releasing study of various capsules and specimens.

Irradiation of Uranium Monocarbide

R. B. Price and W. H. Goldthwaite

The irradiation of Capsule BMI-23-2, containing uranium-5.0 w/o carbon specimens is continuing. The irradiation period has been extended to six cycles. The capsule will be discharged on Cycle 116 shutdown, January 12, 1959. Capsule BMI-23-3 (uranium-5.0 w/o carbon) is at Battelle for thermocouple repairs. Capsule BMI-23-4 (uranium-5.0 w/o carbon) is at the MTR awaiting insertion. It has passed preinsertion tests. The loading of BMI-23-5 and BMI-23-6 (uranium-4.8 and 4.6 w/o carbon) is being delayed by the repair work being done on BMI-23-3.

Typical thermocouple temperatures for Capsule BMI-23-2 during 40-megawatt reactor operation in Cycle 112 are as follows:

K-2

Thermocouple(a)	Temperature, F, on Date Indicated (1958)			
	10/25	10/27	10/29	11/4
1	1170	1180	1225	1325
2	675	685	715	770
3	715	725	750	805
4	360	365	380	410
5(b)	570	630	620	650
6(b)	330	340	385	350

(a) Thermocouple locations:

- 1 In core center of top specimen
- 2 and 3 Touching basket at center of top specimen
- 4 Touching inner capsule wall opposite top specimen
- 5 Touching basket at center of bottom specimen
- 6 Touching inner capsule wall opposite bottom specimen

(b) Suspected defective thermocouples.

Metallographic examination of the Type 304 stainless steel basket and UC specimen used in a compatibility test in NaK at 1100 F for 12 weeks has been completed in an effort to determine the cause of embrittlement of the stainless steel. The surface of the UC specimen appeared normal. Analysis of the stainless indicated that embrittlement was probably caused by a carbide (CrC_4) precipitation at the grain boundaries and possibly by the formation of an unknown phase (possibly sigma) at the grain boundaries and along strain lines within the grains. The use of a stabilized stainless steel, such as Type 347 and the elimination of cold working would minimize embrittlement.

Postirradiation Examination of Uranium Monocarbide

S. Alfant, G. Lamale, A. W. Hare, and F. A. Rough

The object of this program is to study the changes in the physical and chemical properties of uranium monocarbide caused by irradiation in a test reactor.

In order to study these changes, seven capsules were designed and fabricated. One capsule containing twelve specimens was irradiated at the BRR, and six capsules, each containing two specimens, were prepared for irradiation in the MTR. Irradiation of one of these has been completed, while the others are in various stages of progress.

A comparison of the preirradiation and postirradiation density measurements on Specimens AI-2 and AI-3 from Capsule BMI-23-1, irradiated at the MTR, was reported last month in BMI-1301. The capsule operated in one position at a power level of 39.8 megawatts for 12.5 days with Specimens AI-3 and AI-2 in a quoted flux of 0.89 and 0.76×10^{14} nv, respectively. This irradiation produced a burnup of 0.1 a/o. During a midcycle shutdown, the capsule was raised 3 in. In this position at a 39.8-megawatt power level for 11.8 days, the quoted fluxes were 0.96 and 0.89×10^{14} nv, respectively. This produced a burnup of 0.1 a/o of uranium-235 in this position, and a total calculated burnup of approximately 0.2 a/o uranium-235 (1420 MWD/T of uranium).

K-3

The fission-gas analysis indicated that $2.35 \times 10^{-5} \text{ cm}^3$ of krypton-85 was collected inside the capsule after escaping from the specimens. Krypton-85 is approximately one-thirtieth of the total fission gas released under reactor conditions. Based on the krypton-85 analysis, the total gas release was $7 \times 10^{-4} \text{ cm}^3$. The results obtained by calculating the total amount of fission gas released by recoil was $3 \times 10^{-3} \text{ cm}^3$. These results differ by a factor of four, but seem to indicate that gas was released only by recoil from specimen surfaces.

The BRR capsule was irradiated for a period of 43 days at a power level of 1 megawatt. Quoted flux was about $1 \times 10^{13} \text{ nv}$ with a burnup of approximately 700 MWD/T at maximum calculated center and surface temperatures of 660 F and 600 F, respectively. Measurements of density and dimensions were completed on three of the specimens and are given in the following tabulation:

Specimen	Before Irradiation			After Irradiation		
	Density, g per cm^3	Length, in.	Diameter, in.	Density, g per cm^3	Length, in.	Diameter, in.
12A	13.472	0.4973	0.2497	13.43	0.4982	0.2493
15A	13.292	0.4991	0.2499	13.23	0.4998	0.2492
25A	13.500	0.5007	0.2499	13.44	0.5013	0.2493

A comparison of the measurements made before and after irradiation indicates that 0.4 to 0.5 per cent changes occurred in density, while dimensional changes were minor.

At this time, metallography and burnup analyses are being completed on specimens from both the BRR and MTR capsules.

Fission-Product Release From Irradiated Uranium Monocarbide

R. Lieberman, D. N. Sunderman, and M. Pobereskin

The purpose of this work is to determine the rate of release of fission gas from irradiated uranium monocarbide as a function of time and temperature of post-irradiation heat treating.

Two more specimens of uranium carbide will be irradiated in sealed, evacuated quartz capsules. The gas released during irradiation will be collected on charcoal at liquid-nitrogen temperature. The specimens will then be removed from the capsule and heat treated for several days at 1700 to 1900 F and the fission-gas release measured as a function of time and temperature.

Work is continuing on the dissolution of irradiated uranium carbide to measure the quantity of fission gas remaining after heating.

K-4

POSTIRRADIATION STUDIES OF SRE, OMRE, AND OMR FUEL MATERIALS

J. E. Gates

All parts of the program for postirradiation examination of thorium-11 w/o uranium specimens irradiated in Capsules NAA-15-6 and NAA-15-7 have been completed except for metallography. Evaluation of the data is now in progress.

Postirradiation inspection of the uranium dioxide fuel pins irradiated in Capsules NAA-28-1 and NAA-29-1 has been completed. The data obtained from dimensional measurements are presented in tabular form.

Postirradiation examination of the 25 w/o UO₂-stainless steel Fuel Element OMRE-3 has been completed except for additional metallographic examinations. Evaluation of the data is in progress.

The examination and testing of the corrosion, tensile, impact, and U-bend corrosion specimens of structural materials from dummy Fuel Element OMR-1 has been completed. An analysis of the impact and tensile data is in progress.

SRE Fuel Material

J. H. Saling and G. E. Lamale

Thorium-Uranium Specimens

Postirradiation evaluation is in progress on 12 specimens of thorium-11 w/o uranium alloy irradiated in NaK to burnups ranging from 0.57 to 1.5 total a/o at a temperature near 1200 F. All specimens were initially 3/8 in. in diameter and 1-3/8 in. long, and were prepared from 93 per cent enriched uranium. Specimens were irradiated in the swaged condition in the MTR. Inspection of the irradiated specimens revealed six which experienced no gross cracking or swelling. Measurements obtained on these specimens indicated density decreases of 2.6 to 4.9 per cent. The remaining six specimens had cracked, bulged, or crumbled to varying degrees. Four of the six remaining specimens suffered severe bulging only at the top end, suggesting that these four had been incompletely covered by NaK during irradiation. Of the remaining two specimens, one could not be removed from the capsule, and one had failed at the bottom end near the location of the thermocouple.

During the past month, burnup analyses have been performed on six of the twelve specimens. The results of these analyses and dimensional and density data are shown in Tables K-1 and K-2. The decrease in density is greater for the specimens from Capsule NAA-15-7 than for those of Capsule NAA-15-6 even though the burnup of NAA-15-7 specimens is roughly two-thirds that of NAA-15-6 specimens. From the irradiation-temperature data available, this effect is apparently due to the fact that the average temperature of Capsule NAA-15-7 was about 100 F higher than NAA-15-6.

K-5

TABLE K-1. PHYSICAL DIMENSIONS OF THORIUM-11 w/o URANIUM SPECIMENS IRRADIATED IN CAPSULES NAA-15-6 AND NAA-15-7

Capsule	Specimen	Preirradiation Dimensions			Postirradiation Dimensions			Bowling, in.
		Average Diameter, in.	Average Length, in.	Density, g per cm ³	Average Diameter, in.	Average Length, in.	Density, g per cm ³	
NAA-15-7	A	0.3728	1.373	12.14	0.4079	(a)	11.28	0.0042
	B	0.3721	1.374	12.08	0.3798	1.3984	11.49	--
	C	0.3736	1.374	12.06	(a)	(a)	(a)	(b)
	D	0.3733	1.372	12.11	0.3783	(a)	11.63	(b)
	E	0.3730	1.373	12.15	0.3774	1.3902	11.61	0.0022
	F	0.3728	1.379	12.08	0.3743	1.3980	11.74	0.0016
NAA-15-6	1	0.3722	1.374	12.02	0.3772	1.3889	11.71	0.0066
	2	0.3723	1.376	12.16	0.3772	1.3930	11.65	0.0045
	3	0.3724	1.374	12.06	0.3785	1.3929	11.58	0.0036
	4	0.3723	1.372	12.17	0.3811	1.3975	11.60	0.0053
	5	0.3723	1.377	12.16	0.3786	1.4107	11.60	(b)
	6	0.3727	1.376	12.08	0.3767	(a)	11.71	(b)

(a) Specimen was cracked or had crumbled.

(b) Specimen too irregular for bowling measurement.

TABLE K-2. CHANGES IN PHYSICAL DIMENSIONS AND BURNUP OF THORIUM-11 w/o URANIUM SPECIMENS IRRADIATED IN CAPSULES NAA-15-6 AND NAA-15-7

Capsule	Specimen	Burnup, total a/o	Dimensional Change, per cent			Change in Density per Total a/o Burnup, per cent
			Diameter	Length	Density	
NAA-15-7	A	0.72	9.3	--	7.1	9.9
	B	0.63(a)	2.2	1.7	4.9	7.8
	C	0.63(a)	--	--	--	--
	D	0.66	1.3	--	4.0	6.1
	E	0.63(a)	1.1	1.2	4.4	7.0
	F	0.57	1.1	1.4	2.8	4.9
NAA-15-6	1	1.1	1.3	1.1	2.6	2.4
	2	1.1(a)	1.3	1.2	4.2	3.8
	3	1.3	1.6	1.4	4.0	3.2
	4	1.4(a)	2.4	1.9	3.9	2.8
	5	1.5(a)	1.6	2.4	4.6	3.1
	6	0.99	1.1	--	3.1	3.1

(a) The burnup of these specimens was estimated by correlating the radiochemical burnup with dosimetry.

K-6

In the temperature range at which these specimens were irradiated, the swelling rate of thorium alloys increases rapidly with the temperature.* Also included in Tables K-1 and K-2 are data showing the amount of bowing of some of the specimens. The bowing measurements indicate the degree to which the specimens have deviated from their initial right-cylindrical shape.

The data obtained from the mass spectrometric analyses of the gas contained in selected capsules from both trains, and the radiochemical analyses for cesium-137 in the NaK recovered from other capsules, are being evaluated. The next monthly report will contain a summary of these data.

Uranium Dioxide Specimens

The examinations of the tubes containing uranium dioxide pellets irradiated in Capsules NAA-28-1 and NAA-29-1 have been completed. The examinations included sampling of the tube from Capsule NAA-29-1 for fission gas, and visual inspection, photographs, and dimensional measurements of both tubes. Both tubes were sectioned and the fuel pellets examined for internal melting. In addition, a junction box was recovered from NAA-28-1 for examination of the thermocouple connections. The radiochemical burnup determinations and the metallographic examination of selected specimens as originally scheduled have been terminated. The results of the fission-gas sampling have been previously reported. The results of the specimen-tube measurements are included in this report.

The diameter of each fuel pin was measured at intervals of 1 in. along the length of the pin. The pins were then rotated 90 deg and several of the measurements repeated. The results of these measurements are included in Table K-3. Over-all lengths of each pin, and of the NAA-29-1 specimen stack are included below.

	<u>NAA-28-1</u>	<u>NAA-29-1</u>
Over-all length of fuel pin	24.461 in.	24.352 in.
Length of specimen stack	--	18.835 in.

Preirradiation measurements are not available for these pins and an analysis of the data cannot be made.

The junction box recovered from Capsule NAA-28-1 was cut open for examination. The thermocouple connections appeared to be in good condition. However, the box will be returned for final evaluation. An attempt is also being made to extract the hot junction of the thermocouples from the NAA-28-1 specimen stack for examination. This will be completed next month.

Each specimen tube was sectioned and the fuel pellets examined visually at magnifications up to 32X for central melting. This qualitative examination indicated that neither excessive grain growth nor central melting had occurred.

*Hayward, B. R., and Bentle, G. G., "The Effect of Burnup on Metallic Fuel Elements Operating at Elevated Temperatures", 2nd International Conference on Peaceful Uses of Atomic Energy, Paper No. 617, Geneva, Switzerland (1958).

K-7

TABLE K-3. PHYSICAL DIMENSIONS OF URANIUM DIOXIDE FUEL PINS FROM CAPSULES NAA-28-1 AND NAA-29-1

Distance From Reference Mark(a), in.	Diameter of Fuel Pins at Indicated Points, in.			
	Capsule NAA-28-1		Capsule NAA-29-1	
	0 Deg	90 Deg	0 Deg	90 Deg
0	--	0.3771	0.2639	0.2641
1	0.3769	0.3780	0.2639	0.2639
2	--	0.3781	0.2639	0.2637
3	--	0.3775	0.2648	0.2649
4	0.3751	0.3771	0.2628	0.2633
5	0.3785	0.3768	0.2632	0.2638
6	--	0.3764	0.2623	0.2620
7	0.3767	--	0.2633	0.2633
8	0.3768	--	0.2627	0.2630
9	0.3735	0.3779	0.2637	0.2633
10	0.3741	--	0.2637	0.2636
11	0.3751	--	0.2643	0.2636
12	0.3761	--	0.2640	0.2637
13	0.3764	--	0.2638	0.2639
14	0.3757	0.3768	0.2648	0.2642
15	0.3766	--	0.2642	0.2641
16	0.3758	--	0.2641	0.2646
17	0.3772	--	0.2638	0.2634
18	0.3763	0.3764	0.2633	0.2634
19	0.3774	--	0.2628	0.2628
20	0.3768	--	0.2625	0.2626
21	0.3760	0.3771	0.2629	0.2626
22	--	--	0.2638	0.2638

(a) The reference position was 3/8 in. from the bottom of the fuel pin.

K-8

OMRE Fuel Elements

R. J. Burian, D. K. Dieterly, and D. N. Sunderman

All phases of the postirradiation examination of 25 w/o UO₂-stainless steel Fuel Element OMRE-3 have been completed. These include visual inspection, photographs, motion pictures of testing procedures, plate-spacing and plate-thickness measurements, fuel-element dimensional measurements, gamma-activity scans, and radiochemical burnup analyses of slugs punched from selected fuel plates. An attempt to measure the thickness of the organic residue on the plates has also been completed. The results of this measurement were inconclusive. Additional work involving a metallographic examination of two fuel plates is now in progress.

Radiochemical burnup analyses of slugs punched from three different fuel plates have been completed. The results indicate that the burnup ranged from 1.4 to 3/4 a/o of the uranium present, according to location of the fuel plate and of the particular slug in the fuel plate. These data are being correlated with the gamma-scan data to provide an over-all picture of burnup within the element.

Except for the metallographic analyses, no further work is contemplated on this irradiated fuel element.

OMR Fuel and Structural Materials

R. J. Burian and E. G. Bodine

The postirradiation examination of the six different structural materials irradiated in dummy Fuel Element OMR-1 is approaching completion. The examination of the 12 corrosion specimens and the 18 U-bend corrosion specimens has been completed. The testing of 12 tensile and 12 impact specimens has also been completed and the analysis of the data is in progress.

The only work remaining on this experiment is to complete the analyses of tensile and impact data.

L-1

L. TANTALUM AND TANTALUM-ALLOY STUDIES

J. H. Stang

This section outlines the activity in two investigations being conducted for the LAMPRE program at Los Alamos Scientific Laboratory. In the container-materials research, high-quality tantalum and tantalum-tungsten (to 6 w/o) specimens have been prepared by arc melting, and strip-fabrication operations on the specimens have started. If the materials pass metallurgical screening studies, strip specimens will be forwarded to Los Alamos for the next screening step, exposure to plutonium-alloy fuels.

The capsule-design phase of the tantalum-irradiation investigation has been completed and capsule components are being fabricated. Preparation of irradiation specimens will start when parent sheet becomes available.

Development of Container Materials for LAMPRE Applications

D. C. Drennen, M. E. Langston, C. J. Slunder, and J. G. Dunleavy

The initial series of materials for plutonium-alloy compatibility testing at Los Alamos, consisting of unalloyed tantalum and alloys of tantalum plus 1.5, 3, and 6 w/o tungsten, have been produced without difficulty by arc melting in an atmosphere of helium. Radiographic inspection has indicated that the 200-g buttons are sound. Hardness measurements on the unalloyed tantalum specimens have indicated that, once the melting technique was firmly established, the high level of purity of the Temescal tantalum melting stock could be maintained during arc melting. Also, it was found that hardness increased almost linearly with increasing additions of tungsten to tantalum. Typical hardness values for the arc-melted buttons are as follows:

<u>Material</u>	<u>Vickers Hardness Number (10-Kg Load)</u>
Temescal tantalum (melting stock)	90-92
Unalloyed tantalum	92.5-94
Tantalum-1.5 w/o tungsten	108-126
Tantalum-3 w/o tungsten	134.157
Tantalum-6 w/o tungsten	186-214

Chemical analyses and metallographic studies are in progress to determine the purity and homogeneity of the buttons. In the meantime the buttons are being machined in preparation for cold rolling to strip specimens.

Plans are being made to arc melt the second series of alloys which will consist of tantalum plus 1.5, 3, and 6 w/o rhenium.

Irradiation Damage of Tantalum

E. J. Jablonowski, F. R. Shober, F. A. Rough, and R. F. Dickerson

This investigation is directed toward obtaining tensile-property data for irradiated tantalum and comparing these data with those for unirradiated tantalum and tantalum-tungsten alloys. The information sought pertains to the effect of tungsten formed (to about 3 w/o) by transmutation of tantalum. The design of a stainless steel irradiation capsule to contain six 30-mil-sheet tantalum tensile specimens in a NaK environment has been completed. Two such capsules are to be irradiated. Estimates of gamma heating in the test bars indicate that their temperatures during irradiation will be in the neighborhood of 200 F.

According to present plans the capsules will be irradiated at the ETR. They will be exposed to thermal-neutron fluxes of 7×10^{20} and 14×10^{20} nvt, which are the calculated exposures necessary for the transmutation of 1.5 and 3.0 w/o tantalum to tungsten, respectively. While earlier some thought was given to attempting to separate the effect of atom displacement (fast-neutron damage) by evaluating specimens exposed in a cadmium-lined capsule, this plan has now been shelved. In the case of the two capsules that will be irradiated, lattice positions in high thermal fluxes but relatively low fast fluxes will be requested at the test reactor to minimize the fast-neutron damage.

High-purity tantalum will be used in these studies and will be supplied by Los Alamos. Pending its receipt, components of the irradiation capsules and mock-up capsules are being fabricated, and suitable thermal-flux and fast-flux dosimeters are being selected. Also, the intensity of radioactivity of the irradiated tantalum upon discharge from the reactor is being calculated so that the "cooling-down" periods required for the capsules can be estimated.

M-1

M. DEVELOPMENTAL STUDIES FOR THE PWR

R. W. Dayton

Studies of air flow in a PWR model will continue, after a prototype baffling arrangement for the lower plenum has been designed and built.

Gas-pressure-bonded samples of oxide-containing fuel plates have shown that specimens with leak-free compartments can be made when steel jackets are eliminated. The bonding of unjacketed specimens has created a problem of embrittlement from contamination by hydrogen and oxygen in the pressurizing gas. The source of contamination and a practical means for preventing it are being sought.

Reactor Flow Studies

L. J. Flanigan and H. R. Hazard

Air-flow studies in a quarter-scale model of the PWR are being conducted at Battelle to determine the effects of lower-plenum geometry on mixing and flow distribution in Core 2. Previously reported work includes development of an optimum baffling arrangement for the lower plenum with the 7.5-ft core design and submission of drawings of this final configuration to Westinghouse.

During November, Westinghouse designed a similar baffling arrangement suitable for incorporation into the prototype and provided drawings to use for construction of the parts for the quarter-scale model.

In December, a quarter-scale model of the prototype baffling arrangement will be constructed and installed in the model. Complete studies of pressure drop, core-flow distribution, thermal-shield coolant flow, and mixing will be made to verify its performance.

Pressure Bonding of Zircaloy-2-Clad Fuel Elements
Containing Compartmented Oxide Fuel Plates

S. J. Paprocki, E. S. Hodge, D. C. Carmichael, and P. J. Gripshover

A compartmentalized Zircaloy-2-clad flat-plate fuel element containing uranium dioxide cores is being considered for the PWR Core 2. A study is being conducted to determine the feasibility of preparing these elements by gas-pressure bonding. The technique being studied involves fusion welding the edges of the cladding plates to the receptacle plate to form a gastight assembly. The receptacle plates are assembled from strip components.

Defected-compartment corrosion tests have been continued on eight small specimens containing four compartments that were prepared using piece-component frames that were edge welded and then pressure bonded at 1550 or 1750 F, with or without subsequent heat treatment at 1850 F for 5 min. No significant growth from corrosion attack has been observed in any of the specimens after exposure times of up to 28 days in 680 F water. Intercompartmental leak tests have revealed no communication between compartments before or after corrosion testing. The corrosion behavior of the Zircaloy-2 cladding appears good, although some areas of corrosive attack have been observed in the welds of two of the specimens. These areas apparently were due to insufficient cleaning prior to corrosion testing and contamination of the Zircaloy during welding. The corrosion tests are being continued on these specimens.

Several large-scale elements have been prepared by the fusion edge-welding and gas-pressure-bonding technique. These elements are approximately 4-5/16 in. wide and 15 in. long and contain 88 natural uranium dioxide cores with dimensions of 0.100 by 0.250 by 1.500 in. The receptacle plates are constructed entirely from strip components. The end plates of the receptacles are notched to accept the ten 0.040-in.-wide ribs that extend the length of the element and form, with the side plates, 11 full-length compartments. These full-length compartments are further subdivided with 0.040-in.-wide cross ribs to form three compartments over the length of the element. After assembly, the elements are edge welded in a helium atmosphere. A small opening is left in the ends of these specimens so that the helium can be evacuated; the final seal is then made in air. Two specimens of this type were prepared from belt-abraded and machined components and a third contained abraded cover plates and pickled receptacle components. Bonding conditions for the abraded specimens were 1550 F and 10,000 psi of helium for 4 hr; the pickled specimen was bonded at 1750 F and 10,000 psi for 2 hr. After bonding, all of the specimens were heat treated for 5 min in an 1850 F salt bath and cooled between flattening dies.

Internal-pressure leak tests of the components of these elements revealed that all three possessed complete intercompartmental integrity as indicated by burst tests of compartments. The cladding of the specimens, however, exhibited brittle behavior even though high burst strengths were obtained. The specimens bonded at 1750 F were considerably more ductile than the other two specimens; however, these specimens exhibited lower than normal ductility. This brittle behavior is believed to be caused by hydrogen introduced during the bonding cycle. Attempts are being made to eliminate all possible sources of hydrogen from the autoclave before additional specimens are bonded.

Metallographic examination of these specimens indicated that good bonds were obtained. However, it is apparent that air and helium were entrapped during the final sealing of the elements. The entrapped helium results in small voids along the bond interfaces and the entrapped air produces zones of oxygen-rich Zircaloy along the interfaces throughout the specimens. The presence of these oxygen-rich zones complicates the interpretation of core-cladding reaction, since it is not known what portion of the oxygen-rich areas observed around the cores is due to entrapped air instead of core-cladding reaction. It is apparent, however, that the graphite barrier layer is effective in reducing reaction, since no uranium-zirconium reaction zones have been observed.

M-3 and M-4

Heavier graphite coatings are being applied in an effort to completely eliminate core-to-cladding reaction. Specimens now being prepared are also being evacuated during sealing to minimize contamination due to entrapped air.

Areas in which the Zircaloy cladding protrudes above the surface of the oxide cores have been observed in these large-scale elements. This effect apparently resulted from excessive flow of the cladding to accommodate a decrease in the width of the receptacle plate in order to fill void space between the cores and longitudinal ribs. In an attempt to prevent this effect, the notches in the end plates to receive the longitudinal ribs will be eliminated in subsequent specimens so that the ribs are free to move during assembly to minimize the void space between the cores and the ribs. Also the height of the ribs will be reduced to prevent buildup of the excess cladding in these areas.

The most severe problem encountered during bonding of edge-welded specimens has been experienced with cladding embrittlement during the gas-pressure-bonding cycle. Embrittlement results from hydrogen contamination which may be introduced in a number of ways during bonding or heat treating. Possible sources of hydrogen contamination include helium, oil from the compressor, and insulating material in the autoclave. The hydrogen from the helium could be in the form of hydrogen or water vapor; however, dew-point readings on as-received helium indicate the moisture content to be very low. The moisture that is present and the small amount of hydrogen present in the gas may be sufficient to cause embrittlement of thin strips of cladding material. Oil carried over by the gas from the compressor has been observed when the compressor was malfunctioning. Normally, oil is not observed during operation of the compressors. It appears that most of the contamination problems can be minimized by altering the gas-pressure-bonding process to remove possible sources of contamination. Additional specimens are being bonded incorporating several process modifications.

N-1

N. DEVELOPMENTS FOR THE MGCR

W. H. Goldthwaite

Research in two areas of assistance to the Maritime Gas-Cooled Reactor program is reported in this section. The postirradiation examination of metal-clad graphite specimens irradiated in 1000-psi CO₂ at a temperature of 1300 F for 4 weeks followed by 1500 F for 1 week is essentially completed. The postirradiation examination of two stainless steel-clad UO₂ specimens is under way. 2

Metallography of Clad Graphite Specimens
Irradiated in High-Temperature CO₂

W. E. Murr, J. B. Melehan, and F. A. Rough

A one-capsule irradiation program to evaluate the compatibility of graphite and CO₂ with Types 310 and 446 stainless steels and with Inconel is nearing completion.

Metallographic examination of six irradiated metal-clad graphite cylinders has been completed. The six specimens, two clad with each of the above-mentioned cladding materials, were sectioned longitudinally and examined in both the polished and etched conditions. Microhardness traverses were obtained upon each irradiated specimen and on an unirradiated control specimen of each alloy. Microhardness measurements of the irradiated and control specimens are reported in Table N-1, and represent readings from the outside, or gas, interface to the inside, or graphite, interface. The measurements show in every case a decrease in the average hardness of the cladding of the irradiated specimens. The hardness change is most pronounced in the case of the Inconel-clad series. Some specific effects of the experimental irradiation upon the cladding materials as determined by metallographic techniques are noted below.

Examination of longitudinal sections of the irradiated specimens clad with Type 310 stainless steel (A-1 and A-2) revealed that both the inner and outer surfaces of the cladding were slightly rougher than those of the control specimen. In the etched condition, an area approximately 0.0001 to 0.0002 in. wide was observed both on the graphite and the gas sides of the cladding. This area was present in the control specimen and appeared to be associated with a decarburized zone occurring on both the inner and outer surfaces of the cladding. An examination of the interior of the cladding of the irradiated specimens revealed twinning in excess of that observed in the control.

Marked decreases both in grain size and microhardness of the Inconel-clad irradiated specimens (L-2 and L-7) indicate that the Inconel cladding underwent recrystallization during the in-pile experiment. The surface of the gas contact side was rough, indicating a slight corrosion by CO₂. The inner, or graphite contact, side of the cladding was relatively smooth, with no evidence of carbon penetration into the cladding. In a preliminary observation, reported in BMI-1301, evidence of a reaction seemed present at the Inconel-graphite interface. Additional examination revealed that a reaction had not occurred.

Upon examination of the irradiated specimens clad with Type 446 stainless steel (F-1 and F-3), both the inner and outer cladding surfaces appeared rougher than the control-specimen surfaces. Grain size appeared unaffected by the in-pile exposure, although the noticeably lower hardness of the irradiated specimens indicated that some annealing of the cladding had occurred. No carbon penetration was observed on either the inner or outer surfaces of the cladding.

Based on these examinations of the six irradiated and three control specimens, it is believed that no serious carburization or oxidation of the cladding materials occurred during the 5-week BRR exposure. The end-cap weld areas of all nine specimens were intact. There is insufficient evidence from the metallographic examinations for rejecting or selecting any of the three cladding alloys examined.

TABLE N-1. EFFECT OF IRRADIATION ON THE CLADDING HARDNESS OF METAL-CLAD GRAPHITE SPECIMENS

Cladding Material	Specimen	Knoop Hardness(a)									
Type 310 stainless steel	Control	266	257	302	274	270	355	380	492	549	
	A-1	188	222	231	233	256	217	240	263		
	A-2	185	213	211	208	207	207	201	185		
Type 446 stainless steel	Control	202	230	261	284	276	302	261	280	254	
	F-1	215	240	224	229	240	228	224	231	236	220
	F-3	162	192	183	208	186	181	161	191	194	181
Inconel	Control	252	338	327	327	325	327	327	322	335	317
	L-2	177	191	197	207	220	203	217	231	224	183
	L-7	142	171	179	186	174	174	154	174	176	169

(a) Readings from left to right were obtained from outer to inner cladding edges. Knoop hardness numbers were obtained with a 200-g load.

The Postirradiation Examination of GA-BNL Sintered UO₂ Fuel Specimens Clad With Stainless Steel

G. E. Lamale, J. H. Saling, and J. E. Gates

An examination of two stainless steel-clad UO₂ specimens prepared and irradiated at BNL has been initiated. The specimens were approximately 2 in. long by 0.5 in. in diameter. Each specimen was composed of two UO₂ pellets sintered to a density of 93 per cent of theoretical contained in a cladding of stainless steel either 5 or 10 mils thick. The irradiation of the specimen with the 10-mil cladding was conducted in a CO₂ atmosphere. This specimen failed by cladding rupture after 5-1/2 days of irradiation. The second specimen, with a 5-mil cladding, was irradiated for 5 months in a helium atmosphere. The specimens are being examined to determine the cause of failure of the one specimen and to determine the general condition of the other.

N-3 and N-4

Visual examination of the specimen with the 10-mil cladding revealed a large bulge immediately adjacent to a clip fastened around the middle of the specimen. An attempt to remove the clip to permit sampling of the contained fission gas resulted in splitting of the specimen transversely, and the two UO_2 pellets were exposed.

The other specimen appeared to be in good condition externally. The clip was successfully removed for sampling of the fission gas. On making a transverse section of the specimen at the clip end, the face of the UO_2 showed three major radial cracks.

It is planned to complete a dimensional survey and, if feasible, measure the density of the UO_2 specimen with the 10-mil cladding. Each specimen will then be sectioned for a metallographic examination and burnup analysis.

O-1 and O-2

O. ENGINEERING ASSISTANCE TO KAISER ENGINEERS

Reactor Flow Studies

L. J. Flanigan and H. R. Hazard

As part of the Kaiser Engineers' program for the Partially Enriched Gas-Cooled Power Reactor, Battelle is conducting model studies of flow within the pressure vessel.

A quarter-scale model of the reactor vessel will be designed, constructed, and studied using air as a working fluid. The scale and flow rates used for the model study will provide Reynolds numbers in the model which are about 40 per cent of those in the prototype.

The model will be fabricated of glass-fiber-reinforced epoxy resin. The core will be simulated with 1-in. -OD by 3/4-in. -ID aluminum tubes. It will be instrumented to permit measurement of pressure drops throughout the reactor, flow distribution in the core, and velocities and directions of flow in the thermal-shield passage. Provision will also be included for injection of a tracer gas at various points for mixing studies and for use of visual tracers to observe upper- and lower-plenum flow patterns.

During the past month, the general design of the model was established and detailed design was nearly completed. The selection of a vendor for major plastic model parts was begun.

Design and fabrication of the aluminum core should begin next month. Selection of the plastic-parts vendor will be made.

RWD:CRT/all