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PROGRESS RELATING TO CIVILIAN APPLICATIONS
DURING FEBRUARY, 1958

by

Russell W. Dayton
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March 1, 1958

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REPORTS RELATING TO CIVILIAN APPLICATIONS
ISSUED DURING FEBRUARY, 1958

- BMI-1249 "The Effect of Primary Alpha on the Beta Decomposition of Zirconium-Uranium-Oxygen Alloys", by David L. Douglass and Lyle L. Marsh, Jr.
- BMI-1253 "Progress Relating to Civilian Applications During January, 1958", by Russell W. Dayton and Clyde R. Tipton, Jr.

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A-1

A. DEVELOPMENTS FOR ZIRCONIUM-CLAD FUEL ELEMENTS

F. R. Shober

Investigations of the effect of irradiation on the thermal conductivity and electrical resistivity of encapsulated uranium and bare UO_2 are in progress. Materials to be evaluated in these investigations have been received and testing will be started. Creep tests to evaluate the creep strength of 15 per cent cold-worked Zircaloy 2 at elevated temperatures have been in progress approximately 3000 hr. Interruption of a test does not appear to have any deleterious effects on the creep properties of Zircaloy 2 at 345 C.

Aluminum cladding was successfully bonded to nickel-plated I and E type fuel elements by techniques utilizing gas pressure and temperature. Sound bonds were formed under pressures of 5,000 and 10,000 psi at 950 F for 1 hr. Additional work is being directed toward determination of the minimum pressure necessary for formation of a metallurgical bond. Preparation of zirconium alloys for an alloy-development program has started. Selected alloys containing up to 4.0 w/o tin, 2.0 w/o molybdenum, and 3.0 w/o niobium will be screened for corrosion resistance and strength.

In the program to develop additional high-strength corrosion-resistant zirconium alloys, the initial selection of alloys to be investigated has been completed. Materials have been obtained, and arc melting of the alloys has begun.

Thermal Conductivity of Uranium and UO_2

H. W. Deem and C. F. Lucks

A program directed toward determining the effect of irradiation on the thermal and electrical conductivities of uranium and on the thermal conductivity of uranium oxide is continuing.

Uranium

The specimens of unirradiated Zircaloy 2-clad uranium have been received. A radiographic examination showed voids in the NaK filling which were probably introduced during shipping. The specimens will be vibrated in a sequence of positions in an effort to remove the voids from the NaK. Measurements of thermal conductivity and electrical resistivity on the unirradiated canned specimens will be made in existing apparatus, but the apparatus was severely damaged recently. Thus, measurements of the clad specimens will be delayed somewhat while repairs of this apparatus are being made.

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UO₂

An apparatus for making thermal-conductivity measurements on UO₂, both before and after irradiation, has been completed. A steady-state absolute method is being used. Briefly, accurately measured power is introduced into the top part of the specimen and the heat flows through the specimen into a heat sink. Compensated thermocouples at known positions measure the thermal gradients. The thermal conductivity of the specimen will be calculated from the heat flow, cross-sectional area, and the thermal gradients over measured lengths. Guarding to prevent stray heat flows has been provided.

Measurements on fused quartz (a material which has a thermal conductivity significantly lower than UO₂ and which provides a severe test for the apparatus) have shown the guarding to be inadequate. At power levels necessary to raise the apparatus to moderate temperatures this radiant-heat exchange between the input heater and the guard has introduced stray heat flows which are large compared with the small specimen heat flows.

During March, the guarding will be modified to better control the stray heat flows, and calibration runs will be continued.

Mechanical Properties of Zirconium Alloys

F. R. Shober and J. A. VanECHO

The creep properties of 15 per cent cold-worked Zircaloy 2 are being evaluated over the 232 to 399 C temperature range. The limiting deformation of interest is that associated with the initiation of third-stage creep. The increased strength achieved by cold working is apparently retained under condition of creep at the temperatures of concern. The ductility is reduced slightly by the cold working.

Tests in air at 232 C have been discontinued and replaced by tests at 40,000 psi and 288 C and 15,000 psi and 399 C. The additional eight tests have been in progress for approximately 2500 hr and have shown no indication of third-stage creep except the test at 30,000 psi at 343 C which showed an increasing creep rate after 1000 hr. The results are shown in Table A-1. This particular test had been discontinued at 2015 hr, when the load was removed and the specimen furnace cooled under vacuum. The specimen remained in that condition for a period of approximately 1 month. As a result of interest in longer test times and the effect of discontinuous loading and heating, it was suggested that the test specimen be brought to temperature and reloaded at the same stress. This was done and appropriate measurements were made. Deformations associated with unloading and reloading are given in Table A-2. The total deformation of reloading was slightly less than that amount obtained in the first 2015 hr of tests, but within 20 hr creep was occurring at the same rate as before unloading. The time-versus-deformation curve shows very little indication of the discontinuity of load, and, on the basis of this, it would appear that no deleterious effects are experienced by interruption of the test. All tests will continue until third-stage creep or failure is indicated.

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TABLE A-1. CREEP PROPERTIES OF 15 PER CENT COLD-WORKED ZIRCALOY 2

Specimen	Temperature, C	Stress, psi	Time on Test ^(a) , hr	Deformation at Time Indicated, per cent								Creep Rate ^(b) , per cent per hr
				Load On	100 Hr	500 Hr	1000 Hr	1500 Hr	2000 Hr	2500 Hr	Load Off	
7-1-2	232	40,000	2666.4(c)	0.400	0.512	0.545	0.560	0.567	0.580	0.583	0.301	<0.00001
7-1-3	232	30,000	2614.9(c)	0.280	0.337	0.350	0.355	0.0355	0.355	0.358	0.104	<0.00001
7-4-2	288	40,000	150	0.269	0.550	--	--	--	--	--	--	--
7-2-2	288	35,000	2850	0.353	0.520	0.595	0.625	0.675	0.692	0.705	--	0.000026
7-2-3	288	30,000	2750	0.275	0.385	0.415	0.440	0.455	0.470	0.476	--	0.00001
7-3-1	288	25,000	2950	0.205	0.282	0.305	0.323	0.330	0.335	0.340	--	0.00001
7-3-2	288	20,000	2830	0.178	0.227	0.245	0.250	0.265	0.265	0.270	--	0.00001
7-1-1(d)	343	30,000	2475	0.315	0.592	0.745	0.875	1.025	1.183	1.395	--	0.00023(e)
7-2-1	343	25,000	2650	0.230	0.420	0.510	0.548	0.580	0.612	0.645	--	0.000066
7-3-3	343	20,000	2650	0.180	0.325	0.380	0.397	0.417	0.427	0.440	--	0.000026
7-4-1	343	15,000	1775	0.110	0.205	0.245	0.265	0.275	--	--	--	0.00002(f)
7-4-3	399	15,000	300	0.134	0.330	--	--	--	--	--	--	--

(a) Tests in progress.

(b) Creep rate based on creep deformation from 2000 to 2500 hr.

(c) Tests discontinued.

(d) Test discontinued at 2015.6 hr and reloaded after a 6-week dormant period.

(e) Minimum creep rate.

(f) Creep rate based on creep deformation from 1000 to 1500 hr.

A-3

A-4

TABLE A-2. CREEP DEFORMATION OF SPECIMEN 7-1-1 UPON REHEATING TO 343 C AND RELOADING AT 30,000 PSI

Time of Test, hr	Condition	Deformation, per cent
2015.6	Before load off	1.183
2015.6	Load off	0.924
2015.6	After reheating, before load on	0.901
2015.6	Reloading, load on	1.173
2020	--	1.180
2030	--	1.195
2050	--	1.197
2100	--	1.220

Zirconium Cladding

S. J. Paprocki, C. B. Boyer, E. S. Hodge, and C. C. Simons

A technique utilizing gas pressure and temperature to achieve bonding of aluminum to nickel-plated uranium cores is being investigated as a method of cladding internally and externally cooled fuel elements.

A series of I and E type slugs was pressure bonded in an effort to determine optimum bonding parameters for this fuel element. In addition, dimensional-control studies were made on the bonded fuel elements to determine the amount of deformation that can be expected during a pressure-bonding cycle. Metallographic examinations, peel tests, and thermal-shock tests were used to evaluate bond strengths and bond integrity.

Specimens were pressure bonded for 5 min at 950 F at 10,000 psi and for 1 hr at 950 F at 5,000 and 10,000 psi. All three specimens appeared well bonded as evaluated by metallographic examination and peel tests. The specimens have been subjected to thermal-shock tests; however, metallographic results are not completed.

Dimensional-control studies were made in detail on the specimen bonded at 750 F for 1 hr at 5,000 psi. The compiled data indicate that excellent dimensional control may be expected with the pressure-bonding process if the procedure developed for assembly of components is employed. The external diameter of the tubular aluminum can changed less than 0.0005 in. and the internal diameter changed only 0.002 to 0.003 in. at the maximum. The length of the slug and the core-end configurations remained the same throughout the bonding cycle. The aluminum and the nickel barrier layer had deformed along the uneven contour of the uranium to fill in any pits in the core material.

Additional specimens are being bonded to determine the minimum pressure-bonding parameters that can be used and still obtain a bond interface that is not susceptible to thermal shock and is continuous at all points along the bond interface.

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A-5 and A-6

Development of High-Strength Corrosion-Resistant Zirconium Alloys

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

Zirconium and certain of its alloys are used as cladding materials for fuel elements due to their low cross section for thermal neutrons, good fabrication and mechanical properties, and excellent corrosion resistance in many liquids. As a result of this, considerable attention has been given to their properties and to the development of better properties.

A program has been initiated to develop zirconium alloys having at least twice the strength of Zircaloy 2 at 570 F and good corrosion life in 570 F water. The nuclear properties, the thermal conductivity, and the expansion coefficient should be similar to those of Zircaloy 2. The initial portion of the program will consist of the screening of selected alloys containing up to 4.0 w/o tin, 2.0 w/o molybdenum, and 3.0 w/o niobium. These alloys are being prepared by arc melting using sponge zirconium. The screening will consist of hardness testing of annealed zirconium alloys and of corrosion testing for 1000 hr in 570 F high-purity water. The hardness-strength relationship will be used in order to select alloys falling within the strength requirements. Hot-hardness data to determine the relative fabricability of the alloys will be obtained on all alloys which show favorable properties in the screening operation. Time permitting, work on the mechanical and physical properties will be initiated.

A list of alloys to be investigated has been compiled. It consists of permutations of zirconium-base alloys with additions of 2.0 to 4.0 w/o tin, 0.5 to 2.0 w/o molybdenum, 1.0 to 3.0 w/o niobium, 0.1 w/o iron, and 0.5 w/o nickel. All of the materials have been obtained, and arc melting of alloys has commenced.

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B. DEVELOPMENTS FOR ALUMINUM-CLAD FUEL ELEMENTS

R. J. Carlson

Testing of the "right-angle" extrusion-cladding equipment has resumed using a newly designed, stronger mandrel. Thirty-mil claddings were applied successfully to uranium cores; however, M-355 aluminum (19 w/o Al_2O_3) connectors and end plugs failed by cracking and rupturing. Attempts to apply a 20-mil cladding were successful on a 10-ft steel core, but only 1 to 2-ft sections of uranium could be clad without rupture. Evaluation of billet-to-billet welds is not yet complete.

The feasibility study of centrifugal casting as a possible technique for the preparation of aluminum-uranium alloy tubes is nearing completion. Results to date are encouraging and indicate that additional development work should be done.

An investigation has been instigated to develop a uranium alloy having a thermal-neutron cross section not exceeding that of a uranium-4 w/o zirconium alloy but with superior corrosion resistance to a uranium-2 w/o zirconium alloy. Several uranium ternary and quaternary compositions have been chosen for evaluation on the basis of increasing the alloy content of the alpha-uranium phase.

Extrusion Cladding of Flat Plates

R. J. Fiorentino, D. C. Drennen, C. J. Slunder, and A. M. Hall

Tests on the development of the "right-angle" extrusion-cladding process for flat plates were resumed with a newly designed, stronger mandrel. Thirty-mil claddings were applied successfully to uranium and steel cores. However, M-355 aluminum (19 w/o Al_2O_3) connectors and end plugs did not withstand the extrusion forces, and failed by cracking and rupturing. Sections of the clad uranium bars were examined for signs of billet-to-billet welds. These were observed under the microscope, but they have not been evaluated as yet.

In some additional experiments, a 20-mil cladding was applied to 10-ft lengths of steel, but only 1 to 2-ft sections of uranium could be clad without rupture. The appearance of the bulged, oversize cladding indicated that the core was being restrained. Also it was quite obvious that there was a tendency for the core to be cocked edgewise as a result of the imbalance of forces exerted by the aluminum. Apparently, the aluminum can flow more readily from the top portholes which tends to push the core downward until it binds in the mandrel. A small load applied on the core to counterbalance the cocking force minimized this trouble. However, change in design of the mandrel and die should be considered for improved results.

Additional sections of the 20-mil clad steel bars will be examined for evidence of billet-to-billet welds.

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Preparation of Aluminum-Uranium Alloys

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

Aluminum-uranium alloys have proved themselves as useful reactor fuels in low-temperature, water-moderated, water-cooled reactors, and it would be desirable to increase the uranium loading of such reactors by increasing the uranium content of the fuel. Therefore, a program of research concerned with the casting of aluminum-uranium alloys containing high concentrations of uranium is in progress. The objective of this program is to examine techniques capable of yielding reproducibly sound, homogeneous extrusion blanks requiring a minimum of cleanup.

A brief feasibility study of centrifugal casting as a possible technique for the preparation of aluminum-uranium alloy tubes is nearing completion, although all the chemical analyses have not been completed; this initial study indicates strongly that the centrifugal process shows much promise and additional development work would be well worthwhile. Refinements in mold design and the pouring mechanism would be specific areas of study.

An extrusion billet of aluminum-25 w/o uranium containing 3 w/o silicon has been prepared utilizing static casting techniques. This billet will be forwarded to a cooperating laboratory for evaluation.

Development of a Mechanical Extensometer

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

Evaluation of a mechanical extensometer designed and constructed to have sufficient sensitivity to determine the transverse mechanical properties of 3-in. -OD uranium tubing is continuing. As a result of hazards involved in handling uranium tubing which has been exposed to irradiation, the usual methods of measurement of mechanical properties cannot be used. This has made necessary the development of a mechanical extensometer which can be attached to the uranium ring and operated remotely.

Calibration of the extensometer which consisted of raising the specimen holder of the apparatus a known amount with push-rod micrometer and recording the displacement on an SR-4 strain gage indicator has been completed. For every one division movement observed on the SR-4 strain-gage indicator there is a corresponding movement of 0.000014 in. on the diameter of the ring. The transverse properties of aluminum, yellow brass, copper, Type 304 stainless steel, and uranium tubes are being measured in order to evaluate as completely as possible the response of the extensometer over various ranges of deformation. All testing has been done at room temperature. Additional testing at 300 F is planned to further evaluate the extensometer.

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B-3 and B-4

Development of a Natural Uranium Fuel Alloy
With Improved Corrosion Resistance

M. S. Farkas, A. A. Bauer, and R. F. Dickerson

An investigation has begun to develop a uranium alloy having a thermal-neutron cross section not exceeding that of a uranium-4 w/o zirconium alloy but with improved corrosion resistance over that of a uranium-2 w/o zirconium alloy in 300 C water. Improvement in corrosion resistance in terms of hours is being sought for the purpose of providing the necessary time for removal of a defective element, after initial detection of failure, before swelling of the defective element can clog the fuel-element channel.

Most alloy compositions being investigated consist of uranium-2 w/o zirconium plus either aluminum, chromium, molybdenum, nickel, niobium, platinum, ruthenium, titanium, or vanadium. The amount of the third element included is the maximum allowable as determined by cross section. Several other uranium ternary and quaternary alloys of combinations of low-cross-section metals have been chosen, the approach being to improve corrosion resistance by increasing the alloy content of the alpha-uranium phase.

The alloys are to be heat treated and then corrosion tested in 300 C water. An autoclave which permits visual observation of the corrosion process is being employed. Alloys for short-time corrosion-rate determinations will be selected upon the basis of initial visual observations of the rate of corrosion.

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C-1

C. PLANT ASSISTANCE TO MCW

D. A. Vaughan

Density measurements in the uranium dioxide-oxygen system are being made to aid in understanding the miscibility gap between UO_{2+x} and U_4O_9 . Data presented indicate a decreasing density with increasing oxygen content through the UO_{2+x} single-phase region, while for higher oxygen contents the density increases.

Study of the electrical properties of UO_2 to which divalent, tetravalent, and hexavalent metals were added as oxides have been continued. Addition of divalent calcium as CaO to UO_2 increases the p -type conductivity of UO_2 and reduces somewhat the extrinsic activation energy of UO_2 . The tetravalent and hexavalent metal ions have shown little or no effect upon the electrical properties of UO_2 . However, the hexavalent ion (MoO_3) was probably reduced during sintering of the mixed oxides in hydrogen.

Corrosion studies on materials of construction for the nitric acid reconcentrator and for a proposed solvent-extraction process are being continued. The low corrosion rates of Type 304 ELC stainless steel in 38 w/o nitric acid solution are unaffected by sulfate additions up to 5000 ppm. Corrosion tests of several construction materials in the synthetic solutions comparable to the raffinate stream have been discontinued. These tests will be repeated in a composite of the raffinate stream from Weldon Springs to allow for the presence of foreign ions normally found in raffinate.

Apparatus for the study of hydrogen permeability through molten slag is being developed. Hydrogen leaks and failures in the reaction tube have delayed measurement of permeability.

Apparatus for surface-tension measurements of molten magnesium fluoride has been completed and will be tested on materials of known surface tension during the next period.

Investigation of Uranium Oxides

D. A. Vaughan, J. R. Bridge, and C. M. Schwartz

A study of uranium oxides has been made and a topical report is being prepared on the active and inactive uranium dioxide-oxygen systems. The investigation of methods for determining nitrogen in uranium dioxide has been discontinued.

During February a study was made of the effect of oxygen content on the density of oxygen-enriched uranium dioxide. Samples were prepared in the composition range $\text{UO}_{2.04}$ to $\text{UO}_{2.25}$ by heating in oxygen at 130 C. Compositions of $\text{UO}_{2.01}$ and $\text{UO}_{2.02}$ were made by reduction at 1000 C. The oxygen-enriched materials were annealed at 450 C for 100 hr. Helium density measurements were made, and the results are given in Table C-1.

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TABLE C-1. HELIUM DENSITIES OF URANIUM OXIDES

Oxygen-Uranium Ratio		Helium Density, g per cm ³
Before Density Measurement	After Density Measurement	
2.013	2.024	10.94
2.025	2.039	10.54
2.043	2.063	10.27
2.081	2.086	10.69
2.124	2.133	10.53
2.249	2.246	10.89

On the basis of these limited data, it is suggested that the density of UO_{2+x} decreases with oxygen additions up to about $\text{UO}_{2.13}$ (the limit of the UO_{2+x} single-phase region at 450 C), and then increases in the $\text{UO}_{2+x} + \text{U}_4\text{O}_9$ two-phase region. The densities of several intermediate compositions are being measured to verify these results.

The Electrical Properties of Uranium Dioxide

J. W. Moody, R. K. Willardson, and H. L. Goering

The study of the effect of metallic impurities on the electrical properties of UO_2 is being continued. Previous reports have described the preparation of sintered specimens of UO_2 containing small amounts of calcium, molybdenum, and zirconium, and the effects of these impurities on the room-temperature electrical conductivity of UO_2 . It will be recalled that additions of calcium enhanced the p -type conductivity of UO_2 , while additions of molybdenum and zirconium had little effect. These results were interpreted by assuming that the metallic impurities enter into the fluorite lattice of UO_2 by substituting for uranium atoms. Thus, the accommodation of Ca^{+2} in a site normally occupied by U^{+4} introduces additional acceptor states, and the p -type conductivity of UO_2 is increased. Zirconium enters into the lattice as Zr^{+4} , and has no detectable effect on the electrical properties of sintered specimens. Molybdenum behaves in much the same manner as zirconium even when the Mo^{+6} ion is used as the doping agent. Evidently Mo^{+6} is reduced by UO_2 and molybdenum enters the lattice as Mo^{+4} .

The temperature dependence of the conductivity of the doped samples has been measured from room temperature to about 850 C. As might be expected, the samples containing calcium had an extrinsic activation energy (0.32 ev) somewhat less than that measured for a pure sample of UO_2 (0.38 ev). The lower activation energy is consistent with an increase of positive current carriers. The temperature dependence of samples containing zirconium was almost identical to that of undoped UO_2 , while the curves of samples containing molybdenum showed an anomalous decrease in conductivity at about 800 C. This anomaly is somewhat reminiscent of the effect observed in relatively dense specimens of normal MCW UO_2 , and is not clearly understood.

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C-3

Future plans include the continuation of these experiments. Additional specimens will be prepared covering a wider range of impurity concentrations, and the study will be extended to include impurity ions of +1 and +3 valence.

The Corrosion Resistance of Selected Stainless Steels

C. L. Peterson, W. C. Baytos, and F. W. Fink

Nitric Acid Reconcentrator

The investigations of the corrosion of Type 304 ELC stainless steel on prolonged exposure to chloride-contaminated solutions of nitric acid are still under way. The trends of the corrosion rates already reported in 5, 30, 38, and 45 w/o nitric acid solutions, boiling under reduced pressure, continue essentially unchanged.

A series of seven 48-hr exposure periods was completed in which Type 304 ELC stainless steel specimens were exposed in 38 w/o nitric acid boiling at 250 mm of mercury absolute pressure and contaminated with 0, 5, 20, 100, and 5000 ppm sulfate, respectively. The corrosion rates were all less than 0.1 mil per month, and no effect ascribable to the presence of sulfate was evident.

Container Materials for Proposed Solvent-Extraction Process

The corrosion studies in the three solutions which simulate various points in the solvent-extraction system of the proposed new process for nitric acid recovery were discontinued after 1000 hr of exposure. Specimens of Titanium 75A, Carpenter 20 and Carpenter 20 Cb stabilized alloys, and Types 304, 304 ELC, 316, and 347 stainless steel showed very low corrosion rates: of the order of less than 0.01 mil per month at the end of this period.

Future research with this system will investigate the corrosion of Type 304 stainless steel in solutions similar to the synthetic ones already studied, but made by proper manipulation of a composite of the raffinate stream. This will allow a determination of the corrosion rates in the presence of the foreign ions normally found in the raffinate.

Gas-Metal Studies

W. R. Hansen, M. J. Trzeciak, and M. W. Mallett

As part of the program for investigating the fundamental aspects of the process for producing dingot uranium, a kinetic study is being made of factors affecting yield and purity of product.

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Permeability of hydrogen through molten magnesium fluoride slag is being measured to obtain information on how hydrogen moves inside the thermite reactor following reaction. This will contribute toward determining effective means for lowering the hydrogen content of the dingot-uranium product.

Also, the surface tension of molten magnesium fluoride is being studied. Product yield may be affected by a high surface tension which prevents some uranium particles from passing through the molten-slag cap covering the molten-uranium product. Measurements of the surface tension of molten slag will be made. In addition, the effect of impurities, such as MgO and UO₂, on the surface-tension values will be determined.

Permeation of Hydrogen Through Molten Magnesium Fluoride Slag

The experimental setup described in BMI-1253 was tried. It was found that even the 1-mil-thick nickel washer inserted between the diaphragm and the reaction tube was not sufficient to prevent leakage of hydrogen. From this experiment it was concluded that the system would not hold at 1300 C with an atmosphere of gas on one side of the diaphragm and a vacuum on the other.

An experiment was tried using argon in place of the vacuum. The purpose was to determine if the argon would convect heat to a critical wax-sealed joint connecting the molybdenum reaction tube to the glass system supplying hydrogen for the permeation study. The presence of argon did not result in melting of the wax. Therefore, a technique will be adopted whereby argon will be flowed around the exterior of the reaction tube to remove the hydrogen permeating the molten slag and molybdenum.

In the course of these experiments the molybdenum reaction tube cracked, making it unsuitable for use. Another reaction tube will have to be fabricated. Work on this is proceeding.

Surface Tension

The experimental system described in BMI-1253 has been constructed and is ready for operation. The diameter of the capillary has been measured at magnifications of 100 and 250X. An average measurement of 0.0665 ± 0.005 cm was obtained for the diameter.

Initial experiments are planned for room-temperature surface-tension measurements on liquids of known viscosity to check out the experimental system.

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D-1

D. PROCESSING OF FEED MATERIALS

E. L. Foster

Two programs concerned with the processing of feed materials are in progress. Both of the programs are in the interest of National Lead of Ohio in their operation of the Feed Material Production Center, and have as their ultimate objective the present or future improvement of fuel-slug quality. One study is of the factors affecting the quality of vacuum-induction-melted uranium, and the other is a theoretical study of the solidification of unalloyed uranium ingots.

Solidification of Uranium

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

A study of the principles involved in the solidification of uranium is in progress. The influence of such variables as superheat, mold shapes, and mold material is being evaluated and, subsequently, a determination of their effect on the soundness of uranium ingots will be made.

Specifically, a mathematical investigation of the heat transfer in the casting and its surroundings is being made. Mathematical models and heat-flow equations have been formulated. These heat-flow equations, upon resolution, describe the time-temperature history of the casting and mold. Analysis of this relationship may indicate the degree of influence of a particular variable upon the solidification behavior of the melt. Examinations of the influence of casting variables will be made by experimental tests in the foundry.

A new program has been set up for mathematical analysis of a 76-cell division of a pie slice of the casting and mold. The program makes provision for the linear variation of the thermal conductivity of the casting and mold materials with temperature, and consideration is given to the change from conductive to radiant heat transfer as the skin of the casting solidifies. The emissivities of the various surfaces at which radiation is occurring are taken into consideration.

Although the "debugging" of this program has not yet been completed, considerable progress has been made; most of the first cycle of computations made by the computer have been checked and found to be correct. The remaining tasks incident to placing the program in operation are to obtain automatic recycling of the program, i. e., to go to the second and subsequent time intervals, and to then examine the results obtained for instability or lack of convergence.

A method of programming known as Symbolic Optimal Assembly Programming has been used to decrease computing time; this routine automatically assigns the location of instructions in the memory storage unit so that computations are made in optimum time.

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Examination of Factors Affecting the Quality
of Induction-Melted Uranium

R. W. Endebrock, E. L. Foster, and R. F. Dickerson

There is some reason to believe that the quality of uranium fuel slugs may be directly related to the amount of nonmetallic impurities in the metal. Because of this possible relationship, a study is being made of reactions between uranium and various process-related impurities that may occur during the vacuum-melting operation. One phase of the study is experimental, centering about a 10-lb-capacity vacuum-melting furnace; another concurrent phase deals with a literature survey.

Experiments made to date indicate that moisture must be considered as an important impurity. Evaluation of the data has implied that, where graphite crucibles are used, a CO-producing reaction (the classical water-gas reaction) between hot carbon and moisture in the furnace offers a mechanism for introducing carbon to uranium during melting. Immediate goals to be achieved by experimentation include determination of the following:

- (1) Baseline melting data under dry-furnace conditions.
- (2) The degree of control necessary for succeeding experiments.
- (3) The role of moisture during melting.

An experimental test series aimed at supplying this information has been completed. Included in the series were moisture-controlled tests with uncoated graphite crucibles containing no uranium, moisture-controlled uranium melts in which exposures of the molten metal in uncoated graphite crucibles could be compared under wet and dry conditions, and melting tests in a graphite-free system in which the effects of a measured addition of CO gas to the furnace could be compared under wet and dry conditions. Since most of the analyses have not yet been completed, an evaluation of the series must be deferred until later. However, observations during the experiments indicate that the reaction rates of the water-gas reaction and the subsequent CO-uranium reaction are much higher than the rate of the H_2O -uranium reaction.

Planned for the immediate future are tests in which impurities stemming from ingot croppings, derby chips, and briquetted turnings are to be evaluated. In addition, tests will be made to evaluate the effectiveness of a dry MgO coating as a barrier to moisture.

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E-1

E. GENERAL FUEL-ELEMENT DEVELOPMENT

F. A. Rough and D. C. Carmichael

Various fuel-element development programs are reported in this section. These studies are supported by the AEC Division of Reactor Development.

Stainless steel-clad dispersions of enriched UC or UN in stainless steel are being encapsulated for irradiation testing; no new results are available from the study.

The properties of cermets containing high loadings of ceramic fuel compound in a metal matrix have been found favorable as compared with those of pure ceramic cores.

The feasibility of a fueled zirconium hydride moderator is being investigated. Additional pressure-composition-temperature studies are being made on the hydride of zirconium-1 w/o uranium alloy, and specimens of the hydride of zirconium-2 w/o uranium alloy are being prepared for irradiation testing.

Development of Dispersed UC and UN Fuel Elements

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

Irradiation specimens of 24 w/o UN or UC dispersed in Type 318 stainless steel powder and roll clad with Type 318 stainless steel are being subjected to preirradiation testing and encapsulation. Specimens of dispersions of UN or UC in Type 347 stainless steel are undergoing tensile tests at room temperature to 1650 F. Data will be reported when available.

Fabrication of Cermet Fuel Elements

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

The initial phase of work concerned with the development and evaluation of cermet fuel elements embodying 60 to 90 per cent ceramic fuel material dispersed in a metallic matrix has been completed. The properties of this material were found to be superior to a pure ceramic material. Additional studies are planned and a program has been formulated for the next fiscal year.

Fueled Zirconium Hydride Moderator

H. E. Bigony and J. B. Vetrano

The hydrides of uranium-zirconium alloys are being investigated as fueled moderators for potential use in gas-cooled reactors. Additional hydrogen-absorption

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isotherms for the zirconium-1 w/o uranium alloy were measured during this period. Encapsulation of specimens for irradiation is still in progress.

Structure and Pressure-Composition-Temperature Studies

The 625 C hydrogen-absorption isotherm was completed and work is in progress on the 534 C isotherm. From Sieverts plots of these data the boundary limits of the two-phase regions were defined as shown in Table E-1.

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

Phase-Boundary Designation	625 C		534 C	
	Hydrogen Sorption, cm ³ per g	Pressure, cm of mercury	Hydrogen Sorption, cm ³ per g	Pressure, cm of mercury
$\alpha, (\alpha+\beta)$	6.7	0.040	--	--
$(\alpha+\beta), \beta$	28.6	0.041	--	--
$\beta, (\beta+\delta)$	91.9	0.142	--	--
$(\beta+\delta), \delta$	156.0	0.140	--	--
$\alpha, (\alpha+\delta)$	--	--	6.4	0.007

During the coming period the effort will be directed toward examining key hydrides of 1 and 50 w/o uranium-zirconium alloys by high temperature X-ray diffraction in an attempt to positively identify the structure of the condensed phases. Composition-pressure-temperature studies will be started on the hydrides of a 25 w/o uranium-zirconium alloy.

Encapsulation and Irradiation

The encapsulation of specimens of the fueled moderator material, the hydride of zirconium-2 w/o uranium, is continuing. Two irradiation capsules, BMI-20-1 and -2, will each contain four fueled and four nonfueled control specimens under 1 atm of hydrogen at room temperature. The design operating temperatures are 1500 F for BMI-20-1 and 1200 F for BMI-20-2.

Specimen encapsulation, hydrogen loading, and NaK loading were completed for BMI-20-2 this month. Subsequent radiographic analysis of the capsule showed that the tube holding the specimens in place was bowed to the extent that the specimens were touching the wall of the inner capsule. This condition does not fulfill the requirements for the heat transfer through the gas annulus. It was thought that the molybdenum tube holding the specimens was caught on a tube support. Thus, two other methods of holding the specimens in position in the capsule during irradiation are being considered.

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F-1

F. STUDIES OF URANIUM AND URANIUM-ALLOY FUELS

F. A. Rough and A. W. Hare

In this section are reported the various research and development programs on uranium alloy fuels which are being sponsored by the Reactor Development Division of the AEC.

Irradiation of Capsules BMI-8-5 and BMI-8-6 containing zirconium-uranium alloy is continuing at the MTR.

As part of the programs to develop a metastable gamma-phase uranium alloy with increased thermal stability, corrosion-testing results in 680 F water are reported.

Routine progress is reported on the program concerned with the development of melting techniques for the preparation of homogeneous uranium-niobium alloys.

Results of tests which were conducted in 200 C steam in which a uranium sample was completely converted to oxide in the absence of air are reported for the study concerned with the mechanism of uranium corrosion.

Radiation Stability of Uranium-Zirconium Alloys

A. W. Hare, A. E. Austin, A. A. Bauer, and R. F. Dickerson

Irradiation of Capsules BMI-8-5 and BMI-8-6 is continuing at the MTR. Capsule BMI-8-6 is a temperature-controlled capsule and contains two specimens of uranium-50 w/o zirconium alloy. Capsule BMI-8-5 contains three specimens of zirconium-22 w/o uranium alloy.

A metallographic examination is currently being conducted on three zirconium-22 w/o uranium alloy specimens which had been irradiated to a burnup of about 0.8 total a/o. Prior to irradiation the specimens were given three different heat treatments to produce three different microstructures. The resultant microstructures were: (1) fine alpha zirconium and epsilon grains; (2) coarse alpha zirconium in an epsilon matrix; and (3) alpha zirconium outlining a fine epsilon and alpha zirconium matrix.

A postirradiation metallographic examination of the three microstructures revealed no significant change due to irradiation. This initial examination was made with light microscopy.

To determine if any subtle changes in the microstructure had occurred because of irradiation that could not be resolved with light microscopy, the microstructures are now being examined with electron microscopy techniques. Replicas of the three microstructures of the irradiated specimens have been made and are being examined. Replicas of unirradiated control specimens which represent the three different microstructures are currently being prepared and will be used as a basis for comparison with

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the results on the irradiated specimens. The control specimens will be examined in both the as-polished and etched condition to determine if etching introduces any artifacts into the original microstructures.

Development of Gamma-Phase Uranium Alloys

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

A project designed to develop a metastable gamma-phase uranium alloy of increased thermal stability by ternary and quaternary alloying is being conducted. Elements being investigated as gamma stabilizers are chromium, molybdenum, niobium, ruthenium, vanadium, and zirconium.

As part of the investigation, thermal analysis is being conducted on all alloys to determine the effect of alloying elements on gamma-decomposition temperature. As might be expected, increasing niobium content of the uranium-20 w/o zirconium-base alloys lower the temperature at which alpha uranium forms in conjunction with gamma uranium, this temperature being 671 C for a 1 w/o niobium alloy and 637 C for a 5 w/o niobium alloy. As yet, the thermal-analysis data on the other alloys are too incomplete to draw any conclusions.

Hot-hardness measurements of the alloys are being made as an indication of hot strength. Of the alloys tested to date, only the uranium-20 w/o niobium-base alloys have shown an appreciable retention in hardness at higher temperatures. A uranium-20 w/o niobium-5 w/o zirconium alloy specimen exhibited hardnesses of 318 DPH at room temperature and 119 DPH at 900 C. Most of the other alloys tested show a rapid decrease in hardness above 500 C.

After 168 hr of corrosion testing in 680 F water, the following alloys have shown the smallest weight changes. All are being tested in the gamma-quenched condition.

<u>Alloy</u>	<u>Weight Change in 168 Hr, mg per cm²</u>
Uranium-10 w/o niobium-1 w/o zirconium	-1.11
Uranium-10 w/o niobium-1 w/o molybdenum	-4.70
Uranium-10 w/o niobium-3 w/o molybdenum	-1.06
Uranium-10 w/o niobium-5 w/o molybdenum	-8.23
Uranium-15 w/o niobium-3 w/o molybdenum	-1.92
Uranium-20 w/o niobium	+0.44
Uranium-20 w/o niobium-3 w/o zirconium	-3.82
Uranium-20 w/o niobium-5 w/o zirconium	+1.88
Uranium-20 w/o niobium-10 w/o zirconium	-3.47
Uranium-7.5 w/o molybdenum-2 w/o ruthenium	-8.12

Most of the other alloys being corrosion tested have either disintegrated or are corroding rapidly.

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As a result of preliminary data, additional uranium-molybdenum alloys containing 7.5, 10, and 12 w/o molybdenum with ternary and quaternary additions of ruthenium and niobium are being prepared for investigation. This investigation will consist mainly of transformation-kinetic studies using hardness measurements and metallography. In addition, uranium-niobium alloys are being investigated further in an attempt to determine the effect of varying aging temperature on rate of transformation.

Preparation of Uranium-Niobium Alloys

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

Alloys of uranium-niobium containing high percentages of niobium are of interest as enriched fuels for use in high-temperature gas-cooled reactors. Among the advantages of alloys in this system is their relatively high strength at elevated temperatures. Though some work has been done in relation to this system, most of the effort has been concentrated on those alloys containing 15 w/o and less of niobium; therefore, a program of study concerned with the preparation and properties of the high-niobium-content alloys is now in progress.

The immediate objective of this program is to develop melting techniques capable of yielding homogeneous material. Recent studies of twice-melted alloys, prepared by the consumable-electrode arc-melting technique, have indicated that the banding prevalent in the uranium-10 w/o niobium ingots will also be found in some degree in the alloys containing greater concentrations of niobium. The extent and severity of this banding is being determined through the use of metallographic and radiographic examination, and by chemical and micro-beam analyses.

The alloys of interest will be fabricated in protective molybdenum cans. These cans will afford protection against oxidation during fabrication and will also increase the likelihood of their fabrication by approximately closed-die forging conditions.

After fabrication, the alloys will be studied to determine the effects of this fabrication on the inhomogeneity. Efforts to homogenize the alloys at elevated temperatures will be made both before and after fabrication. It is believed that prior fabrication will greatly reduce the necessity for homogenizing the alloys.

Constitutional-diagram studies are being contemplated and since these studies will require homogeneous alloys containing a minimum of impurities, melting procedures for the production of homogeneous 100-g melts are being investigated. All of the methods now being investigated utilize the inert-electrode, arc-melting technique together with some variation in casting procedure. The alloys obtained from these furnaces will be evaluated on the bases of the micro- and macrohomogeneity and the quantities and type of contaminants present.

Mechanism of Aqueous Corrosion

E. Adelson, C. M. Schwartz, D. A. Vaughan, O. M. Stewart,
W. E. Berry, P. D. Miller, and F. W. Fink

The reaction between uranium and water or water vapor is being studied at temperatures ranging up to 200 C. A study is being made of uranium valence in the uranium corrosion product formed in water at temperatures below boiling. Previous work on the optical properties and on the L_{III} X-ray absorption edges of UO_2 and UO_3 had indicated differences which might aid in identifying the oxidation state of uranium in the corrosion product.

During the past month, the L_{III} X-ray absorption edge was determined for U, UO_2 , UO_3 , and corrosion product. The corrosion product was prepared by exposing dingot uranium to boiling, deionized water for 24 hr and used immediately for the X-ray measurements. The absorption edges were mathematically analyzed for minima, maxima, and points of inflection and band widths, results of which are given in Table F-1. In all cases, the characteristic values for the corrosion product are intermediate between those for UO_3 and UO_2 , suggesting a valence between 4 and 6 for the uranium. The non-uniform shift in position of the minimum from UO_3 to UO_2 is not understood and will require repeating the measurements. Corrosion product formed at other temperatures will be investigated during the next period. It is also planned to compare these results with the optical properties of the bulk oxides. However, the optical studies will be concentrated on examination of thin adherent oxide films which cannot be investigated by the X-ray absorption method.

TABLE F-1. CHARACTERISTIC POINTS ON THE L_{III} X-RAY ABSORPTION EDGE OF URANIUM METAL, UO_2 , UO_3 , AND URANIUM CORROSION PRODUCT

Sample	Minimum Absorption, angstrom	Inflection Point, angstrom	Maximum Absorption, angstrom	Band Width, angstrom
Uranium metal	0.72648 ₇	0.72253 ₁	0.71857 ₅	0.00791
UO_2	0.72679 ₄	0.72260 ₆	0.71840 ₇	0.00839
Corrosion product	0.72633 ₆	0.72232 ₄	0.71831 ₂	0.00802
UO_3	0.22630 ₇	0.72227 ₁	0.71323 ₆	0.00807

Note: The absorption-edge wavelength was calculated on the basis of LiF crystal with a reported 2d spacing of 4.0275. Measurement of 2d for this particular crystal has not been completed. Although this may modify the absolute wavelength, it will not affect the change due to the valence of the uranium.

Tests have been conducted in 200 C steam in which the uranium sample was completely converted to oxide in the absence of air. The reaction vessel was small enough to permit its being weighed on an analytical balance. Based on weight increase, the freshly formed corrosion product had an oxygen content corresponding to $UO_{2.18}$. However, a stoichiometric quantity of hydrogen was not released during the reaction. The corrosion product, calculated from the hydrogen released, would have been $UO_{1.90}$.

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F-5 and F-6

The oxygen content in the corrosion product rose to $\text{UO}_2.40$ when allowed to oxidize in room temperature air. Ignition in oxygen gave a final uranium-oxygen ratio of $\text{UO}_2.64$, or slightly less than $\text{UO}_2.67$ as found in U_3O_8 . Thus, a small amount of product apparently was lost in the reaction train, since no unreacted uranium was left in the vessel.

Future work will be aimed at accounting for the discrepancy between the amount of hydrogen evolved and metal corroded. Efforts will be made to determine if uranium hydride is in the corrosion product by noting whether hydrogen is liberated during oxidation by room-temperature air.

G-1 and G-2

G. FATIGUE STUDIES OF INCONEL

W. S. Hyler and R. J. Favor

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 Inconel.

The scope of the program includes temperatures of 1200, 1400, and 1600 F. Previously, the data for the 1600 F investigation were reported. During February the investigation at 1200 and 1400 F was nearly completed. During the early part of March it is expected that the experimental phase of this program will be concluded. In addition, data on the static creep properties of Inconel have been received. It is now possible to analyze the effect of dynamic creep superimposed on static creep for this material under the conditions of the program. Some preliminary calculations have been made; however, the final figures will be reported after the experimental phase is completed.

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H-1

H. REACTOR MATERIALS AND COMPONENTS

F. A. Rough and A. W. Hare

The various research programs concerned with reactor materials and components that are supported by the Reactor Development Division of the AEC are reported in this section.

Fabrication results of five new binary alloys and six new ternary alloys are reported. Also reported are the completed results of oxidation tests in dry air on niobium-1 a/o iron at 1000 and 1200 C. This work is for the program designed to study the oxidation of pure niobium and the mechanism by which various alloying additions affect oxidation behavior.

In the study of valence efforts of oxide solutions in UO_2 , preliminary results are reported in the investigation of the stability of $\text{La}_2\text{O}_3\text{-UO}_2$ solid solutions and UO_2 by means of vaporization measurements.

Routine progress is reported on the high-temperature and high-pressure solid-state studies.

The initial results of the preliminary tests on the friction apparatus designed to study the friction behavior of rubbing surfaces immersed in sodium at temperatures up to 1500 are reported. The performance of the apparatus was quite satisfactory.

Routine progress on the basic studies of pressure bonding are reported. Both the analytical and experimental investigations aimed at determining the conditions under which metal surfaces to be bonded attain the sufficiently intimate contact to bond are discussed.

Oxidation-Resistant Niobium Alloys

W. D. Klopp, C. T. Sims, and R. I. Jaffee

The objective of this program is to study the oxidation of pure niobium and the mechanisms by which various alloying additions affect oxidation behavior. Previous tests on binary alloys have shown that the size and valence of the alloying atoms and the properties of new oxides are important. Current work includes preparation, fabrication, and oxidation testing of new binary and ternary alloys.

Five new binary and six new ternary alloys have been consolidated as 35-g ingots. Fabrication by cold or hot rolling into 0.050-in. strip was attempted, with results as given in Table H-1. It can be seen that only the niobium-1 a/o iron alloy was cold fabricable, while six of the remaining ten alloys were hot fabricable. Limits for hot fabricability of binary iron- and rhenium-containing alloys lie between 5 and 15 a/o iron and 5 and 10 a/o rhenium, respectively. Based on as-cast hardnesses, the fabricability

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H-2

of the ternary alloys was lower than that of binary alloys with similar hardnesses. While most binary alloys with as-cast hardnesses below 200 VHN were cold fabricable, none of the ternary alloys could be fabricated cold. Titanium promotes fabricability in ternary combination, as all four ternary alloys containing titanium were fabricable compared to those with molybdenum. The seven fabricable strips were annealed at 1500 C and are now being oxidation tested. The two unfabricable ternary alloys have been remelted and will be tested in the cast condition.

TABLE H-1. HARDNESS AND FABRICABILITY OF NIOBIUM ALLOYS

Composition (Balance Niobium), a/o		Hardness, VHN		Fabricability By Rolling ^(c)	
Nominal	Adjusted ^(a)	Cast	Wrought and Annealed ^(b)	Room Temperature	1000 C ^(d)
5Fe	1Fe	167	127	G	
10Fe	5Fe	262	330	--	F
20Fe	15Fe	455	--	--	NG
5Re	5Re	242	274	--	F
10Re	10Re	336	--	--	NG
12.5Ti-4Mo	10.5Ti-3Mo	171	177	NG	G
19Ti-11Cr	19Ti-2.5Cr	192	235	NG	G
25Ti-15Cr	25.5Ti-6Cr	230	348	--	G
19Ti-9V	17Ti-6.5V	199	262	--	G
5.5Mo-11Cr	4.5Mo-1.5Cr	212	--	--	NG
6Mo-9V	5Mo-7.5V	254	--	--	NG

(a) Compositions adjusted to the nearest 0.5 a/o on basis of melting-loss data.

(b) Rolled to 0.050 in. and annealed 1 hr at 1500 C.

(c) G denotes good fabricability; F, fair, but able to be tested; NG, no good.

(d) Ingots were encapsulated in evacuated stainless steel packs for hot rolling.

Oxidation tests in dry air have been completed on niobium-1 a/o iron at 1000 and 1200 C. Data are given in Table H-2. Iron additions at this level promoted a slight decrease in the 1000 C oxidation rate, and a significant reduction in the 1200 C oxidation rate, although no tendency toward parabolic oxidation behavior was noted. The scales were yellow, but otherwise similar to those formed on pure niobium.

Future work includes:

- (1) Oxidation testing of the remaining six alloys which were hot fabricable (listed in Table H-1)
- (2) Fabrication of five ternary alloys containing titanium-molybdenum, titanium-chromium, and titanium-vanadium, the compositions of which were given in BMI-1253

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- (3) Preparation of additional ingots of niobium-15 and -25 a/o chromium, niobium-11 a/o chromium-5.5 a/o molybdenum, and niobium-6 a/o molybdenum-9 a/o vanadium for oxidation testing in the cast condition
- (4) Preparation, fabrication, and oxidation testing of niobium-12.5 and -15 a/o molybdenum and tungsten to complete investigation of these binary systems.

TABLE H-2. OXIDATION DATA FOR NIOBIUM AND NIOBIUM ALLOYS IN DRY AIR

Sample	Composition, a/o	Test Temperature, C	Parabolic Rate Constant, $(\text{mg}/\text{cm}^2)^2$ per hr	Transition to Linear Rate, hr	Linear Rate Constant, $\text{mg}/(\text{cm}^2)(\text{hr})$	Weight Gain in Indicated Time, mg per cm^2					
						1 Hr	2 Hr	3 Hr	4 Hr	5 Hr	6 Hr
0-30(a)	100Nb	1000	100	0.1	30.0	30.0	61.3	89.8	114.2	136.9	158.6
0-107	Nb-1Fe	1000	--	0	25.2	28.8	51.1	69.6			
0-77(a)	100Nb	1200	--	0	(93) ^(b)	107.7	--	--	--	--	--
0-108	Nb-1Fe	1200	--	0	(36)	48.0	79.2	--	--	--	--

(a) Data from previous tests included for comparison.

(b) Rates in parentheses are estimated, since actual rates decreased gradually as oxidation proceeded.

Valence Effects of Oxide Solutions in Uranium Dioxide

W. B. Wilson, J. Moody, A. Gerds, W. M. Albrecht, and C. M. Schwartz

The use of a tantalum susceptor in the vacuum-induction unit used to fabricate the $\text{UO}_2\text{-La}_2\text{O}_3$ solid-solid solutions has been discontinued, since analyses indicated impurity contents equal to those of the previous techniques. In view of this it appears necessary to return to a graphite susceptor surrounding the sample but arranged in such a way that direct diffusion to the sample is not possible. The only other material permitted in the system in the high-temperature region will be UO_2 , in order to eliminate metal-cation impurities.

Preliminary results have been obtained utilizing microbalance techniques in the study of vaporization of the $\text{La}_2\text{O}_3\text{-UO}_2$ solid solutions and UO_2 . Both materials indicated initial and very rapid oxidation (subject to considerations previously discussed) at 2800 F, followed by approximately the same linear weight-loss curve for each. Conversion to actual fuel loss must await chemical analyses of the samples.

Comparison of the electrical properties before and after oxidation of a solid solution of $\text{UO}_2\text{-50 w/o La}_2\text{O}_3$ indicated a considerable decrease in resistivity after oxidation. The $\text{UO}_2\text{-60 w/o La}_2\text{O}_3$ sample as first prepared in a tantalum susceptor system had resistivities which were too high to be measured with the present apparatus. Regrinding and reprocessing dropped the resistivity, perhaps due to increased tantalum impurity concentration.

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Work will continue to obtain high-purity samples for electrical measurements before and after oxidation and while at temperature, and for comparison of these data to the microbalance oxidation data.

High-Temperature High-Pressure Solid-State Studies

W. B. Wilson and C. M. Schwartz

Minor modifications were undertaken on the carbide inserts of the first (right-circular cylinder) die unit during February. The modifications were made to improve the strength and usability of the unit.

A second unit, utilizing parts interchangeable with the first unit, was assembled and has been tested in a 150-ton press. Initial calibration for pressure indicated that friction is considerable in the right-circular-cylinder die at pressures near and above the first bismuth transition. Nearly 20 per cent excess force is required above the computed value in order to produce the transition and, upon release of the press load, friction tends to maintain considerable pressure temporarily in the die. This unit appears to be satisfactory in its present state to better than 30,000 atm. Work will be undertaken to calibrate temperature as a function of heating power, before actual use.

The finalizing of the geometrical-advantage die design has been initiated, utilizing the data and experience gained with the right-circular-cylinder type.

Rubbing Surfaces in Sodium Environments

J. W. Kissel and J. H. Stang

A series of runs to check out the high-temperature operation of the friction apparatus, designed to study the friction behavior of rubbing surfaces immersed in sodium at temperatures up to 1500 F, was completed during February. The specimens were soaked in a solid lubricant to produce a chemically bonded lubricant film capable of withstanding high temperatures. The 1/2 in.-diameter spherical specimen, which is the stationary rubbing member, was fabricated of type M-1 steel hardened to 64 R_C; the flat specimen, which moves linearly (at a speed of about 0.1 mm per sec for these tests), was fabricated of Type M-2 steel, annealed and lapped. The net contact load, applied normally to the sphere, was 155 ± 2 g. This load corresponds to a maximum stress (Hertzian load) of approximately 65,000 psi. (In BMI-1253, it was stated that net contact loads of 75, 93, and 143 g correspond to Hertzian loads of 80,000, 85,000, and 100,000 psi; these are in error, the correct values being 50,000, 53,000, and 61,000 psi).

During these experiments, an argon atmosphere was maintained in the test chamber. Values of average maximum coefficients of kinetic friction are tabulated below; some "slip stick" was observed at the 470 F level and above.

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H-5

<u>Temperature, F</u>	<u>Coefficient</u>
80	0.128
470	0.132
700	0.149
900	0.167
1000	0.167
1200	0.169

It is interesting to note that up to 900 F the friction increased steadily, but between 900 and 1200 F was almost constant. It has been postulated that, during the lower temperature operation, the chemically bonded film underwent some kind of change which altered its lubricating properties; at 900 F and above, most of the lubricant had disappeared from the surfaces by sublimation. The latter supposition is substantiated by the fact that wear tracks did not appear on the flat specimen until after the 1000 and 1200 F runs; the presence of wear tracks under the conditions of these experiments suggests a complete lubricant failure. With this apparatus, individual wear tracks can be produced, since the flat specimen can be shifted to present a fresh contact surface.

As a whole, the performance of the apparatus during these runs was quite satisfactory, inasmuch as the friction data were reproducible within satisfactory limits. A few modifications of a minor nature were indicated and are being incorporated into the system. Before tests employing molten sodium are initiated, a few more elevated-temperature runs will be made.

Basic Studies of Pressure Bonding

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

An investigation is in progress to analyze the mechanism of bonding surfaces under gas pressure. Emphasis is being placed specifically on determining the conditions under which the metal surfaces to be bonded attain the sufficiently intimate contact to bond.

Analytical Studies of Pressure Bonding

As a simplified physical model for the study of deformation under hydrostatic pressure, the collapse of a thick-walled cylinder is being analyzed. Theoretical predictions of the behavior have been made considering, alternatively, a modified ideal plastic material and the application of creep theory. In each case certain physical parameters remain to be determined experimentally. It is presently conceived that creep theory gives a more valid indication of the actual behavior.

By making a comparison at two different pressure levels of deformation versus time, it is expected that an indication of the interdependence of pressure and time will be obtained. For example, a preliminary examination of limited data indicates that at 500 F the deformation of a 2S annealed aluminum cylinder will be a function of $(p^2 \cdot t)$. If this or a similar relationship is confirmed, it can provide a basis for selection of

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pressure and time. Thus, if a given deformation occurs for some time and temperature, the same deformation is expected to occur for other times and pressures, as long as the value of $(p^{2.8}t)$ is the same. An examination of more complete data is required to verify this tentative conclusion.

Experimental Studies of Pressure Bonding

The experimental phase of the program is directed toward acquiring data which can be correlated with the theoretical values calculated on the basis of postulated mathematical models. Evacuated and sealed thick-walled tubular aluminum specimens are subjected to various sets of pressure-bonding parameters in order to examine the flow characteristics of metals during a hydrostatic pressure-bonding process. To insure reliability of results, duplicate specimens are subjected to each set of pressure-bonding parameters.

In order to determine the effect of the time and pressure parameters on the extent of deformation achieved during the pressure-bonding process, specimens have been subjected to a hydrostatic pressure of 6000 psi at 500 F for periods of 2 and 4 hr. Results of these tests and comparison with previous data at 4000 psi indicates that these parameters have an appreciable effect on the extent of deformation achieved at 500 F. It was also observed that, as the hole size becomes small, the originally circular cross section of the hole becomes distorted. This is as expected due to the polycrystalline structure of the material.

Additional specimens have been prepared and will be used to study the effects of time, temperature, and pressure on the extent of deformation achieved during pressure bonding. These specimens will be subjected to various sets of pressure-bonding parameters as prescribed by the analytical studies.

I-1

I. PHYSICAL RESEARCH

F. A. Rough

Programs supported by the AEC Division of Research are reported in this section. Included are various studies of metallurgy and ceramics.

In BMI-1253, reports were included for various researches into bonding fundamentals, reaction of niobium with hydrogen, and migration of hydrogen in zirconium hydride. The remaining projects are reported this month.

In the study of properties of uranium compounds, the reaction of the important compounds with water is reported. Progress is also being made toward determining the thermal-expansion coefficients and thermal conductivities of these compounds.

The constitution of uranium-base ternary alloys is being studied. A few final details of work on uranium-zirconium-silicon and uranium-zirconium-molybdenum alloys are in progress. This research will be concluded shortly. New research is planned to evaluate the effects of oxygen on the constitution of uranium-niobium alloys.

High-temperature X-ray diffraction studies of the uranium-carbon-nitrogen system are in progress. Under study in particular is the phase field between UC_2 and $U(N, C)$ solid solution.

The structure of UC_2 is being redetermined. Neutron-diffraction data are being analyzed from UC_2 powder samples, and single crystals of UC_2 are being sought experimentally.

Preparation, Fabrication, and Physical Properties
of Uranium Compounds

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

Methods of ceramic fabrication and fundamental properties of some refractory uranium compounds which are potential nuclear fuels are being studied.

The corrosion of UC_2 sintered compacts in boiling water was studied and the preliminary experiments on the corrosion of UC and UBe_{13} in boiling water were repeated, but with more precision. Sintered compacts of UC and UC_2 disintegrated in boiling water within 1 hr. The disintegration was followed by rapid oxidation of the carbides. Both carbides reacted so rapidly that it was not possible to estimate which reaction was more rapid. Analyses of the corrosion products are not complete.

Tests are in progress to determine the effect of boiling water on sintered compacts of UBe_{13} . Samples tested in preliminary experiments disintegrated after several hours. The resulting particles retained their metallic luster, and the weight change after 10 hr was negligible. Testing of a second group of samples indicated that the

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corrosion loss after 36 hr based on the original area of the samples was approximately 3.6 mg per cm². After an additional 36 hr, the original compacts were only about 25 per cent disintegrated. This increased resistance to disintegration may have resulted from changes in fabrication technique. Smaller compacts were used in fabrication of the second group and this resulted in more densely sintered and more uniform samples.

The technique of UBe₁₃ preparation has been further improved by more thorough mixing of the UH₃ and Be powders. This has apparently resulted in a more homogeneous product after firing. The product was ground to minus 325 mesh and sintered. Metallographic examination is pending.

Equipment for measuring thermal expansion by interferometric techniques in vacuum is being set up.

Preparation of specimens for thermal-conductivity measurements is continuing.

Constitution of Uranium-Base Ternary Alloys

M. S. Farkas, A. A. Bauer, and R. F. Dickerson

A study of the phase relationships which occur between the delta phases of the uranium-zirconium and uranium-molybdenum systems is being conducted.

Alloys, believed to be near the eutectoid composition of the uranium-zirconium-molybdenum ternary, are being investigated by means of thermal analysis and by metallographic and X-ray diffraction examination of specimens subjected to long-time heat treatments. The location of this ternary eutectoid is being sought.

The phase relationships existing in the uranium-zirconium-silicon system are being studied in alloys containing high uranium concentrations. The previously reported Zr₅Si₃ phase has been shown by other investigators to be a ternary phase that becomes stable when small amounts of boron, carbon, nitrogen, or oxygen are present. Therefore, this phase does not exist in the pure zirconium-silicon binary, but is stable in place of Zr₃Si₂ when minor impurities are present. Several alloys are being prepared from electrolytic uranium, crystal-bar zirconium, and transistor-grade silicon. The alloys are being prepared to determine whether complete solid solubility exists between U₃Si₂ and Zr₃Si₂. An alloy of the composition Zr₄Si is also being prepared in an effort to determine if such a compound exists.

An investigation of the uranium-niobium phase diagram will begin shortly. The materials to be employed in this study are of the highest purity available. Electrolytic uranium and two grades of ultrahigh-purity niobium are being used. The niobium is being obtained in laboratory quantities by means of bromide- and iodide-decomposition processes developed at Battelle. The bromide niobium will be approximately 99.94 w/o niobium, with the highest impurity element being about 60 ppm oxygen. The iodide niobium will have a somewhat higher impurity level, and from 300 to 500 ppm oxygen.

Alloys will be made to redetermine controversial phase boundaries of the uranium-niobium system and to determine the effect of impurity content on these boundaries.

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I-3

Solid-State Study of Ternary System Uranium-Nitrogen-Carbon

A. E. Austin, A. F. Gerds, C. M. Schwartz

This ternary system is being studied to determine the stability of solid-solution phases of uranium carbides and nitrides, and to investigate the possible existence of ternary phases. Materials were prepared by solid-solid reaction and solid-gas reaction. The earlier results showed that UN and UC formed solid-solutions with a linear variation of unit-cell size. Reactions with U_2N_3 or UC_2 produced a solid solution of U(N, C) with unit-cell size deviating from linearity, thus indicating a slight broadening of the solid-solution field.

Additional reactions were carried out to explore the two-phase field of UC_2 and U(N, C) solid solution. Table I-1 gives the product composition and phase analysis. According to the data, the U(N, C) solid solution for the two-phase field extended from UC to approximately $U(N_{0.3}C_{0.7})$. The observation of U(N, C) existing with free carbon may indicate another two-phase field. Further work is under way on checking this observation and also on seeking the ternary-phase field of U(N, C), UC_2 , and C.

Attempts were made to form U_2C_3 from UN, UC, and UC_2 at 1600 to 1700 C to determine possible nitrogen solid solution. To date, only minor amounts of U_2C_3 phase were produced in compacts of mainly U(N, C) and UC_2 .

Powdered UC and UC_2 were reacted with nitrogen at 1 atm pressure at 1000, 1300, and 1600 C for 2 hr. The products were free carbon and a U_2N_3 -like phase. According to a chemical analysis for combined carbon, there was essentially no carbon solution in the U_2N_3 phase. Further reactions are planned at higher temperatures.

TABLE I-1. COMPOSITIONS AND PHASES IN PRODUCTS OF REACTIONS
UN + CARBON, UC + U_2N_3 , AND UC_2 + U_2N_3

Starting Mixture	Product, a/o			Phases
	Uranium	Carbon	Nitrogen	
UN + 2C	31.4	56.8	11.8	$U(N,C)_{a_0} = 4.9393 \pm 0.0003$ A + C
UC + $2U_2N_3$	51.3	14.5	34.2	$U(N,C)_{a_0} = 4.9096 \pm 0.0003$ A
5UC + U_2N_3	49.9	36.6	13.6	$U(N,C)_{a_0} = 4.9495 \pm 0.0003$ A + ppt. UC_2
5.5 UC_2 + U_2N_3	38.9	58.4	3.1	UC_2 + ppt., $U(N,C)_{a_0} = 4.9565 \pm 0.0005$ A

Structure of UC₂

A. E. Austin and C. M. Schwartz

The structure of UC₂ is being reinvestigated, in order to fix the positions of the carbon atoms in the unit cell, as an aid in interpretation of thermal behavior and type of uranium-carbon bonding. Extraction of single crystals was attempted by etching an ingot which had been slow-cooled through the transformation from the high-temperature cubic form. Although the crystals appeared to be of sufficient size, X-ray examination showed that actual single crystals have not yet been obtained.

Neutron-diffraction data from UC₂ powder have been obtained and are being analyzed.

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J-1

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

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

The study of corrosion problems associated with some of the various processes for the recovery of uranium from spent reactor fuel elements was continued.

Further exposure of titanium specimens to the conditions of the Darex dissolver and to solutions representative of the initial and final conditions in the feed-adjustment system has not indicated any limitation on the use of titanium for these process vessels. A boiling 10 w/o aqueous NaOH solution effectively removes the scale which accumulates in the dissolver.

The corrosion rate of Carpenter 20 stabilized alloy is much lower in the final than in the initial nitric-fluoride solution of the Thorex dissolver. Addition of aluminum to the initial nitric-fluoride solution produces a substantial lowering of the corrosion rates.

It was shown that only a very small potential difference was developed between the container wall and specimen in fused fluoride salts at 650 C, indicating that metal deposition on the corrosion specimen arises from thermal differences rather than galvanic action.

New containers are being constructed from Hastelloy B for further examination of more resistant materials for the fluoride-volatility process.

The Darex Process

Uranium can be recovered from fuel elements containing stainless steel as a diluent or 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 solvent extraction.

Dissolver Studies With Titanium

By the end of February, nearly 800 hr of exposure of plain, welded, and stressed specimens of Titanium 55A and 75A has been completed in the presence of the dissolution of stainless steel in equipment which simulates a continuously operating Darex dissolver. Following 1000 hr of exposure, an extensive metallographic examination of these specimens will be conducted. Thus far, the visual examination given these specimens periodically has not revealed any deleterious effects as a result of corrosion.

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An apparent disadvantage of the dissolution process is the excessive buildup of a siliceous scale on the walls of the dissolver vessel. Preliminary experiments showed that this scale is practically unaffected by boiling solutions of proprietary chelating agents, but is removed easily within an hour by boiling aqueous solutions of about 10 w/o NaOH. As far as could be discerned from these limited experiments, such a treatment had little adverse effect upon the titanium specimens from which the scale was removed.

FAT Studies With Titanium

Plain, welded, and stressed titanium specimens were examined following 1170 hr of exposure in refluxing solutions which simulated the initial conditions in the feed-adjustment tank. Corrosion rates were very low, and no evidence of cracking was observed.

Four experiments are under way in which solutions, representative of conditions in the FAT system, are refluxed by means of heat supplied, in part, by passing 140 C steam through the corrosion specimens. These specimens are titanium tubes, bent in the shape of hairpins, and equipped with a weld bead. One such study with a final FAT solution has over 700 hr of exposure. A large buildup of solids occurred at the liquid line but, underneath this, the titanium appeared to be in good condition. Three other experiments, one with the initial solution and two with the final, now have in excess of 200 hr of exposure. The true resistance of these titanium specimens will not be ascertainable until the end of the exposure period, when sectioning and metallographic examination can be completed.

The Thorex Process

The particular Thorex process under study is one in which stainless steel-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^- . At the end of this dissolution, the final Thorex solution would be about 8.5 M HNO_3 , 0.05 M F^- , and 1.0 M $\text{Th}(\text{NO}_3)_4$. Prior to the study of an actual cyclic process between the two acid systems, specimens of Carpenter 20 stabilized are being evaluated in the nitric-fluoride system.

As mentioned in BMI-1253, Carpenter 20 stabilized had a corrosion rate of about 50 mils per month in the initial nitric-fluoride solution. However, in the final solution, the rate was only of the order of 0.7 mil per month after 184 hr of exposure. This could be caused, in part, by the decreased acidity, but is probably largely the result of complexing of the fluoride by the thorium content of the solution. This suggested the incorporation of a known fluoride-complexing agent, aluminum, into the initial solution and led, in turn, to corrosion rates of about 4 mils per month when 0.1 M Al^{+3} was added, and about 2.7 mils per month in the presence of 0.2 M Al^{+3} during short (24 hr) exposure periods.

J-3 and J-4

Studies of thorium additions to the initial nitric-fluoride solution and of cyclic exposures of the Carpenter 20 stabilized specimens between the sulfuric and nitric acid solutions are planned.

The Fluoride-Volatility Process

Fuel elements containing zirconium as a diluent or cladding can be reclaimed by a fluoride-volatility process. The first step consists of hydrofluorination of the elements in a molten bath of $\text{ZrF}_4\text{-NaF}$ with a stream of HF . A material of construction for this hydrofluorinator is being sought.

Metal crystals were deposited on the corrosion specimen in several of the experiments. Several tests were conducted to obtain a better understanding of the deposition. It was shown that a potential of only 0.006 v was present between the Inconel container wall and the specimen (INOR-8) and support tube (Hastelloy W). No significant difference in potential was noted with nitrogen or HF atmospheres. It appears that the crystal deposition is a result of mass transfer arising from thermal differences between the walls of the container and the corrosion specimen rather than from galvanic attack.

A better assembly would employ a container constructed from the same material as that under examination, or at least a material more resistant to this system than Inconel. At present, INOR-1 and INOR-8, which showed good resistance in preliminary studies, are not available in the sizes necessary. Therefore, three new assemblies have been fabricated from Hastelloy B, which should be much more resistant than Inconel. Long-term exposures of specimens of INOR-1 and INOR-8 are planned in these assemblies.

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K-1 and K-2

K. EVALUATION OF A REFLECTOR-CONTROLLED
HETEROGENEOUS BOILING REACTOR

R. G. Wilson, D. A. Dingee, and J. W. Chastain

A study is being made of the effects of voided regions of various sizes passing completely through a reactor core on various reactor parameters and neutron streaming. This work has been temporarily recessed but the program will be continued as soon as possible.

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L-1

L. STUDIES OF SODIUM-TANTALUM COMPATIBILITY AT ELEVATED TEMPERATURES

J. H. Stang

Research is being conducted to establish the creep characteristics of commercial grades of tantalum in sodium and in inert-gas environments and to investigate factors pertinent to the welding of tantalum. These studies are under the auspices of Los Alamos Scientific Laboratory to supply information to the LAMPRE program.

Stressed tantalum specimens are being exposed to 1200 F sodium circulating in polythermal pumping-loop systems. Initial creep data have indicated that tantalum does not suffer distinguishable loss of strength under the combined stress-corrosion conditions being maintained. The first specimen subjected to test did not display abnormal corrosion effects after 505 hr of exposure; it was stressed to 15,000 psi.

Recently, the research to provide inert-atmosphere creep data has been mainly concerned with creep experiments at 1200 F to establish the strength of specimens having weld beads normal to the load. Several modifications in the degassing apparatus have been of substantial benefit although totally reliable operation has not yet been achieved. Creep experiments with weld-containing specimens continue to indicate that their strength compares well with that of base material.

During February, resistance-welding studies advanced to the point that three tube-to-header joint designs could be chosen for intensive investigations. Other activity in this research includes studies to determine causes of porosity in joints made with some lots of tantalum and an investigation of grain refinement in tantalum welds by ultrasonic excitation.

Tantalum-Sodium Compatibility Studies

G. E. Raines, C. V. Weaver, and J. H. Stang

Five experiments were conducted in the series in which the creep and corrosion behavior of tantalum specimens exposed to 1200 F sodium flowing in polythermal pumping-loop systems (fabricated of Type 316 stainless steel) are being investigated; at the end of February four runs were still in progress. Table L-1 presents creep data now on hand. Comparison of these with data determined for tantalum stressed in helium at 1200 F (reported in BMI-1220 and BMI-1238) indicates that the sodium exposure has very little effect on the strength of the material.

The specimen from the experiment that terminated at 505 hr is being analyzed for carbon, hydrogen, nitrogen, and oxygen. During the exposure, its hardness increased from 165 VHN (5 kg) to 199 VHN, indicating an increase of gaseous interstitials and suggesting that slight contamination by air occurred during the run. It is believed that the blanket-gas (argon) system present in this particular loop was imperfectly sealed and, consequently, permitted the entry of minute amounts of air.

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L-2

TABLE L-1. CREEP DATA FOR TANTALUM DURING EXPOSURE TO HIGH-TEMPERATURE SODIUM

Loop Conditions:								
			Exposure Temperature		1200 ± 10 F			
			Fluid Temperature Cycle		Approximately 60 F			
			Flow Rate		Approximately 1 fps			
Stress, psi	Time in Progress hr	Time to Reach Indicated Percentage Deformation, hr					Total Deformation, per cent	Minimum Creep Rate, per cent per hr
		0.1	0.2	0.5	1.0	2.0		
<u>Annealed Sintered Tantalum</u>								
15,000	505 ^(a)	1/4	3/4	4	10	64	3.5	0.0026
12,500	360	1-1/3	12	65	--	--	0.75	0.00068
<u>Annealed Arc-Cast Tantalum</u>								
18,000	576	3	7	41	--	--	2.4 ^(b)	0.00056
18,000	168	3	40	--	--	--	0.3	0.006
								(estimated)
12,500	720	6	288	--	--	--	0.3	0.00005

(a) Terminated by a failure of a high-temperature-limit thermocouple.

(b) A power failure after 288 hr shut this experiment down; at this time the specimen elongation was 0.8 per cent. During the startup period, an inadvertent overstressing of the specimen caused a rapid elongation from 0.8 per cent to 2.25 per cent.

Weight-loss information for the above specimen indicates a somewhat higher degree of corrosion (equivalent to 4.9 mils per year of penetration) than is normal for unstressed tantalum in 1200 F flowing sodium. Most of the increase, however, is probably attributable to the contamination that was present. The specimen did not exhibit unusual effects, such as edge cracking or intergranular penetration, which would suggest that stress-corrosion phenomena had occurred.

High-Temperature Mechanical Properties of Tantalum

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

Experiments investigating the thermal degassing of tantalum have continued. A thin (70 mils) tube of tantalum was selected as the specimen to be degassed; previously a sheet specimen was used, but, with this geometry, it was difficult to obtain an adequate loading of the Megatherm and reach the temperature (approximately 4800 F) necessary for degassing. Experiments with the new configuration are scheduled for early March.

Creep tests at 1200 F have been completed on tantalum specimens which were fabricated from commercial as-rolled arc-cast sheet and had a bead weld normal to the direction of load; data are presented in Table L-2. Although greater initial deformation occurs in a welded specimen, the creep rate after about 50 hr appears to be

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L-3

essentially the same as in unwelded as-rolled material. The higher initial deformation in welded specimens may stem from the fact that the short weld zone is in an unworked condition at the start of the test and an initial elongation is required to work harden it.

TABLE L-2. CREEP DATA FOR TANTALUM WELDMENTS AT 1200 F

Specimen	Stress, psi	Time in Progress, hr	Time to Reach Indicated Percentage Deformation, hr			Total Deformation, per cent	Minimum Creep Rate, per cent per hr
			0.1	0.2	0.5		
<u>Welded Specimens of As-Rolled Arc-Cast Tantalum Sheet</u>							
W1	15,000	1724	0.0	11	--	0.391	0.000015
W2	17,500	1010	0.0	5	175	0.700	0.00016
<u>As-Rolled Arc-Cast Tantalum Sheet (Control Specimens)</u>							
A1	15,000	1656.2	11	825	--	0.238	0.00004
A2	15,000	335 ^(a)	16	--	--	0.185	0.00003

(a) Test in progress.

Recently, creep tests of annealed arc-cast tantalum sheet have been started at 900 and 1500 F. These are the first creep experiments with tantalum at a temperature level different from 1200 F.

Weldability of Tantalum for High-Temperature Systems

R. L. Koppenhofer, S. M. Silverstein, and R. P. Sopher

Studies to determine the causes of porosity in joints made with some lots of tantalum continued. Various pass sequences and welding techniques were used in an attempt to concentrate the porosity normally found adjacent to the heat-affected base metal. Although there was no sizable concentration of porosity created by the multi-pass welding, a change in microstructure occurred. Samples of tantalum weld-metal and unwelded material have been submitted for complete constituent analyses. If possible, compositional differences will be interpreted in terms of factors causing porosity.

During recent weeks, a magnetostrictive transducer with a 1-kw rating at a no-load resonance of 19.4 kc has been built for use in a study of the effects of ultrasonic vibration on grain refinement of arc welds in tantalum. This device is designed to force drive a specimen (clamped to a conical horn fastened to the transducer) during welding. In exploratory studies of ultrasonic effects, it was observed that some grain refinement was produced when excitation was supplied throughout the welding cycle. However, precipitation of a second phase at the grain boundaries was noted. This phase appears to be similar to a second phase precipitated at grain boundaries of tantalum excited immediately after welding. Further study should indicate if these phases are similar

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L-4

and also if they can be eliminated, since this precipitate might cause weld-zone embrittlement.

Exploratory resistance-welding tests were completed on all of the small-bore tube-to-header joint designs that were initially proposed. The most promising designs have been selected, and work to develop satisfactory welding conditions and procedures for them has been initiated. The following are the designs which merit further investigation:

- (1) Flanged tantalum tube spot welded to a tantalum header
- (2) Flared tantalum tube inserted in and spot welded to a countersunk hole in the tantalum header
- (3) Simple tube-to-header butt joint.

At present, difficulty is being encountered in producing uniform bonding throughout the entire circumference of the tube-to-header joint. Experiments to develop techniques for eliminating this difficulty are in progress.

The problem of electrode sticking when a molten zone is produced in a resistance weld has not been solved. Of the three electrodes (RWMA Classes 1 and 2, and tungsten) evaluated, none has shown satisfactory results. Further studies to solve this problem are planned.

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M-1

M. DEVELOPMENTAL STUDIES FOR THE PWR

R. W. Dayton

No studies of flow in the PWR model are now in progress while preparations are being made for mixing studies in a 7-1/2-ft core design.

Adjustments of the X-ray photometer have improved its performance so that it appears capable of detecting changes of uranium concentration of about 0.3 w/o in a gamma-phase niobium alloy.

A number of pressure-bonded specimens have been made from belt-abraded cold-rolled Zircaloy 2. Good bonding with complete grain growth was obtained at 1550 F. In a single test at 1500 F, grain growth was incomplete. Grain growth, even for material which is somewhat contaminated, is achieved by following the alpha-phase bonding with a short beta-phase treatment.

Reactor Flow Studies

L. J. Flanigan and H. R. Hazard

Studies in a quarter-scale air-flow model of the PWR are being conducted to determine the effects of lower-plenum geometry on mixing and flow distribution in Core 2. Previously reported work includes completion of all flow studies with the 9-ft core design.

Drawings of the flow baffle for the quarter-scale model to simulate a 7-1/2-ft core design were revised to include instrumentation for pressure measurement and a layout of the hole pattern for the bottom of the baffle. Flow studies for this core design will begin on delivery of the flow baffle.

X-Ray Photometric Examination of Fuel Elements

J. B. Schroeder and C. M. Schwartz

An X-ray photometer has been developed for the inspection of fuel elements by means of X-ray density measurements. The apparatus has been assembled and tested and is essentially complete.

The photometer consists of a 150-kv constant-potential X-ray generator with a fine-focus X-ray tube (0.4-mm² focus) rigidly mounted on a lathe bed whose saddle supports the specimen and translates it across the X-ray beam. The beam is collimated by a 0.060-in. aperture in a shield of Mallory 1000 alloy (tungsten-6 w/o nickel-4 w/o copper), placed between the sample and the detector. A scintillation probe with a thallium-activated NaI crystal serves as the detector. The collimator shield and

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detector assembly can be aligned transverse to the X-ray source by means of a pair of translation screw adjustments. The signal from the probe is fed from a preamplifier to a linear amplifier and pulse-height analyzer, and then to a ratemeter and a strip-chart recorder. All power is obtained from a Stabiline line-voltage regulator. In operation, it has been found that good intensity and an excellent ratio of beam-to-background intensity is obtained by setting the pulse-height analyzer to a window width of 50 per cent. This discriminates almost completely against the excessive background due to Bremsstrahlung. Satisfactory operation of the pulse-height analyzer required the acquisition of an ultrastable high-voltage supply for the photomultiplier tube.

The apparatus has been tested on a uranium-10 w/o niobium filler plate, nominally 0.135 in. thick, having a series of milled steps each approximately 0.001 in. high by 0.1 in. wide. These steps were readily detected at a specimen-translation speed of 1/2 in. per min. At 145 kv and 3 ma, the counting rates were of the order of 8×10^5 cpm, and the change in intensity per mil step was about 5×10^4 cpm, or better than 6 per cent. The percentage probable error at this counting rate is about 1 per cent, so that a change in thickness of, say, 0.2 mil should be detected. This is equivalent to a change in uranium concentration of about 0.3 w/o.

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

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

An investigation is being conducted to determine the feasibility of cladding compartmented uranium dioxide with Zircaloy 2 by a gas-pressure-bonding technique. Studies have continued on the preparation of Zircaloy 2 surfaces for bonding and the prevention of fuel-cladding reaction during pressure bonding.

Results of the investigation to determine a suitable surface preparation for obtaining good Zircaloy 2-to-Zircaloy 2 bonds have indicated that belt-abraded material consistently produces satisfactory bonding. Techniques have been developed for belt abrading both hot-rolled and cold-rolled Zircaloy 2 to achieve excellent bonds, as evidenced by 100 per cent grain growth across the original bond interface and no bond-line contamination. Belt abrading is accomplished by passing the material to be abraded under a fast-moving abrasive belt. The process is suited to production as material can be processed at relatively high speed for a reasonable cost.

A series of cold-rolled and belt-abraded specimens has been run over a period of several weeks to determine the reproducibility of bonding this type surface. All specimens that were bonded at 1550 F and 10,000 psi for 4 hr yielded 100 per cent grain growth across the interface, and no contamination was visible at any point. A specimen prepared in a similar manner but bonded at 1500 F showed no visible contamination and 75 per cent grain growth across the interface. This specimen was annealed at 1500 F for 3 hr to determine if a longer bonding time would have increased the bond-line grain growth. Metallographic examination revealed that the growth across the interface was not substantially increased by this treatment. Therefore, unless

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annealed and belt-abraded material would give improved results, pressure bonding at 1500 F or lower temperatures to avoid reaction problems should be followed by a short heat treatment at a higher temperature to obtain complete grain growth across the bond interface, as discussed later.

Inconsistent bonding of specimens with machined surfaces is believed due to contamination of some of the surfaces resulting from variations in the machining process or the starting material. Electron-diffraction examination of such surfaces has disclosed zirconium dioxide, which may have resulted from overheating the thin cladding during machining. Examination of other oxidized surfaces after pressure bonding has indicated that the oxide did not completely diffuse into the Zircaloy 2 during bonding. In an attempt to determine if bonds with only 80 to 90 per cent grain growth obtained during pressure bonding of previous irradiation specimens resulted from slight contamination from the machined surfaces or from the uranium dioxide cores, two additional specimens were prepared from the same material. One contained outgassed steatite cores for simulated fuel; the other, which was run in a separate can at the same time, consisted of two flat plates with no cores. After bonding, both specimens had about 80 per cent grain growth across the bond interface and slight contamination along the bond line. These results are identical with those of the specimen containing uranium dioxide cores, which indicated that the bonding was not perfect due to contaminated machined surfaces. It should be pointed out that, although the machined-surface bonds were not perfect and therefore not considered satisfactory for this program, such bonds do not fail in mechanical-peel tests or show bond-line attack during corrosion testing.

It has been found that specimens which do not have an optimum surface preparation and produce bonds with less than 100 per cent grain growth across the interface can be heat treated for a short time in the beta-phase region to obtain 100 per cent growth. A similar treatment could also be given belt-abraded specimens if it becomes desirable to have added insurance that all specimens have complete grain growth across the original interface. The results of this program have indicated that this procedure would result in very reproducible bonds having no contamination and 100 per cent grain growth across the interface. Excellent results have been obtained by placing a specimen in a salt bath at 1850 F for 2 min. The use of the salt bath for annealing eliminates excessive heating and cooling times that would greatly increase the fuel-cladding reaction.

The results of the study being conducted to determine a suitable barrier layer to prevent fuel-cladding reaction during bonding have demonstrated that chromium would be very satisfactory. Purposely defected and sectioned corrosion specimens having a 0.0005-in. electroplated chromium barrier on the uranium dioxide cores have shown no growth or visible attack after 6 weeks in 750 F steam and 6 weeks in 680 F water. Results also indicate that chromium is an effective barrier layer even if an as-bonded sample is heat treated at 1850 F for 15 min.

The amount of reaction occurring between the Zircaloy 2 cladding and Ti-Namel spacers during pressure bonding has been found to depend upon the temperature of bonding and the surface preparation of the Ti-Namel. The regulation of these variables may eliminate this reaction problem; however, in case a barrier layer is required between these materials, suitable ones are available. A stainless steel shim can be

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included between the cladding and spacers, or a thin coating of chromium can be electroplated on the spacers to prevent reaction between the cladding and spacer.

Specimens have been run to determine the effect of oversized and undersized cores on the extent of bonding obtained; results are not yet available. Components for a group of large flat-plate specimens (11 by 2.25 by 0.08 in.) are being prepared and will be used to develop further the techniques studied in this program. The annealed Zircaloy 2 for these specimens is being belt abraded and core-insert holes will then be stamped in the receptacle plates. The specimens will be used to attempt to establish the reproducibility of the pressure-bonding process using developed procedures without a subsequent beta-phase anneal, and also with an anneal and, possibly, use of a lower bonding temperature to minimize or eliminate reaction problems.

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N. EVALUATION OF URANIUM MONOCARBIDE AS A REACTOR FUEL

F. A. Rough

Considerable progress is reported for the first phase of an evaluation of uranium monocarbide as a fuel for the SRE.

Samples are being prepared by casting techniques with considerable success. These samples are being used for compatibility tests to select capsule materials for irradiation and for necessary physical tests.

Meanwhile, further development of the casting techniques is in progress to permit controlled preparation of enriched specimens at the proper time.

Casting Techniques for the Preparation of Uranium Monocarbide

A. C. Secrest, E. L. Foster, R. F. Dickerson

During the past month, considerable progress has been made on the first phase of a program concerned with the evaluation of uranium monocarbide as a fuel material. The monocarbide is attractive because of its refractory nature, its reported high thermal conductivity, and the possibility that it can be fabricated to fuel-element shapes by casting techniques. Irradiation data on UC are very limited. The data reported are for sintered, porous samples, and the results are not necessarily meaningful for cast materials.

Small, well-formed castings which did not fracture on cooling have been prepared in a reproducible fashion. The material for these 3/8-in.-diameter by 2-in.-long castings was melted in a bench-scale, arc-melting furnace and cast in graphite molds under a helium atmosphere. Analytical results indicate that the castings contain the stoichiometric amount of carbon. They also indicate that no significant increase in carbon content results from casting in graphite thimbles.

The castings were ground without fracturing by using an Alundum grinding wheel and a water-base cutting fluid. No detrimental effects were observed on the ground specimens as a result of using the water-base cutting fluid. However, it should be noted that the castings prior to and immediately after grinding were kept in a desiccator. The need for keeping the monocarbide in a dry atmosphere has not been firmly established. However, on the basis of one test, there is evidence that UC disintegrates when left in water overnight.

Nearly all of the machine-ground specimens needed for compatibility studies with NaK at elevated temperatures have been prepared. Radiographic examinations, using a cesium-137 source, are planned for all specimens to determine soundness.

The average density, based upon weights and dimensions, was 13.3 g per cm³ or 97.5 per cent of theoretical. The density is being determined also by the more sophisticated method of weighing the specimens in liquids.

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Holes for the insertion of thermocouples have been drilled in cylinders of cast UC by using ultrasonic drilling techniques. There was no evidence of specimen cracking as a result of drilling.

The first few castings made during the month were cast in either solid graphite or solid copper molds. Castings made in this way were not well formed and cracked during cooling because the casting cooled too rapidly. The casting quality has been improved considerably by using thin-walled graphite thimble molds that are suspended by a graphite collar in a cavity in a copper mold. Work directed toward adapting an additional arc-melting unit to produce larger castings of uranium monocarbide is proceeding.

A number of materials and techniques have been examined in an effort to coat UC for thermal-conductivity tests. Of these, only indium applied mechanically gave a reasonably satisfactory coating. Thus, indium will be used until a better coating material is found.

The specimen size for the irradiation program has been revised. On the basis of thermocouple size, capsule size, center-line temperature, and flux requirements, a 3/8-in. -OD by 2-in. -long specimen was selected. The specimens will be enriched to 8.3 w/o uranium-235.

Immediate plans are to supply additional machine-ground specimens needed for compatibility studies of carbide with NaK at 600 C. Effort will also be directed toward supplying machine-ground specimens for thermal-conductivity and thermal-expansion tests. As soon as the specimens for these investigations are completed, emphasis will shift to producing cast specimens suitable for irradiation. A study of the physical properties of UC as they vary with composition will be made as part of a program directed toward evaluating the effect of such variations on the quality of the castings.

Irradiation-Capsule Design for Uranium Monocarbide

R. B. Price, R. H. Barnes, E. M. Chandler, and G. D. Calkins

Preliminary studies under way will guide the design of the irradiation capsule. NaK compatibility tests have been planned with uranium-4.8 w/o carbon specimens in stainless steel containers as tabulated below.

Basket	Exposure Time, weeks	Temperature, F
Molybdenum	2	1100
Molybdenum	8	1100
Molybdenum	2	1300
Stainless steel	2	1300

Several preliminary specimens have been received. Radiographic examination techniques have been tried with promising results. Obvious surface defects in the specimens were observable in the radiographs.

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First attempts to drill center-line holes in specimens ultrasonically appear to be successful. Holes of 0.077-in. diameter, 0.33-in. depth were drilled in 0.25-in.-OD by 1.0-in.-long specimens.

Sample thermocouples were plated with 0.005 in. of molybdenum. This is one approach that is being considered to prevent contact between the stainless steel thermocouple sheath and the specimen. Arrangements have been made to obtain molybdenum sheet of 0.010-in. thickness and to have this sheet rolled to an approximate 0.001-in. thickness for specimen basket material.

Several basket designs being considered will allow the basket to be disassembled and the specimen to be observed intact after irradiation if it has fractured.

Potential liquids for use in density measurements, fabrication for machining, and NaK disposal are being investigated. Compatibility tests of UC specimens in water, ethyl alcohol, butyl alcohol, benzene, xylene, and air were done. Preliminary results of exposures of 1, 3, and 5 hr indicated little or no weight change and no obvious physical change except in the case of water after 3 and 5 hr of exposure. Bubbles of acetylene gas were given off and some fragmentation occurred in the water-exposure test.

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O-1

O. DEVELOPMENTS FOR NMSR

A. W. Hare and R. F. Dickerson

The program of study concerned with the effect of irradiation on UO_2 fuels has been initiated in cooperation with The Babcock & Wilcox Company. The fuel to be studied is that proposed for the Nuclear Merchant Ship Reactor (NMSR). This reactor is a pressurized-water type to be fueled with uranium pellets clad with stainless steel tubes containing pelletized uranium.

As an initial portion of this program, the preparation of suitable pelletized UO_2 specimens for an in-pile test program has begun. Pellets with a density of 98.2 per cent of theoretical have been prepared. As lower bulk densities are required, shorter ball-milling times and lower forming pressures will be used. The dummy fuel-pin assemblies have been prepared for an engineering test loop at the MTR.

Research is also being conducted on the safety aspect of foreseeable accident types in connection with the NMSR program. During the past month an analysis of the continuous-rod-withdrawal accident has been undertaken.

Fabrication of Urania Fuel Pellets for Loop-Test Studies

H. D. Sheets, C. Hyde, and A. G. Allison

The objective of this program is to prepare suitable, pelletized UO_2 specimens for in-pile-loop test program. The specimens are to contain 3 to 7-1/2 w/o enriched UO_2 and have sintered bulk densities of about 91 per cent of theoretical.

Four lots of UO_2 of varying degrees of enrichment have been received.

A 100-g batch of UO_2 containing 2.83 per cent U^{235}O_2 was ball milled for 15 hr in a 1-qt natural rubber-lined mill with Al_2O_3 balls. The milled powder was mixed with 3 w/o camphor and pressed at 20,000 psi in a steel die. Pressed density of the pellets was about 6.85 g per cm^3 . The pellets were sintered for 1 hr at 3000 F in flowing hydrogen. Sintered bulk density was about 10.69 g per cm^3 (98.2 per cent of theoretical).

Shorter ball-milling time and lower forming pressures and sintering temperatures will be used in an effort to obtain lower bulk density. Some differences in physical properties can be expected depending on the fabrication techniques which are chosen.

Sinterability of the other lots of UO_2 available for this program are presently being investigated.

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Fabrication of Loop-Test Fuel Pins

S. Alfant, A. W. Hare, A. A. Bauer, and R. F. Dickerson

The fabrication of dummy fuel-pin assemblies which are to be used in the engineering test loop at the MTR has been initiated. The unfueled prototype assemblies will be used to determine flow conditions in the loop prior to the first loading of fueled subassemblies.

The fuel pins for the irradiation tests will consist of tubes containing pelletized UO₂. The tubes, end caps for the tubes, and the components for fuel-pin holders are fabricated from Type 304 stainless steel.

Preliminary work on the dummy assemblies has consisted of developing end-capping techniques and assembly procedures for both the fuel pins and the fuel-pin assemblies.

As soon as the techniques for the fabrication of the fuel pins and fuel-pin assemblies have been developed, three short pin assemblies and one long pin assembly will be completed and shipped to the MTR for the flow tests. Upon satisfactory completion of these tests, work will be initiated on the fabrication and assembly of the fueled subassemblies.

Simulation of Various Accident Considerations for the
NMSR Program Using Analog Techniques

J. J. Stone, Jr., B. B. Gordon, and R. S. Boyd

Research is being conducted on the safety aspects of foreseeable accident types in connection with the Nuclear Merchant Ship Program. Four accidents have been selected for analysis by analog simulation techniques. These are: continuous rod withdrawal, startup, loss of coolant flow, and cold water accident.

An analysis of the continuous-rod-withdrawal accident has been undertaken. Three phases have been considered: (1) no safety system in operation, (2) high-flux trip, and (3) period trip. Each of these operations was observed under a variety of parameter variations including reactor power level, coolant flow rate, rate of negative-reactivity insertion, period for scram, delay in period circuitry, flux scram level, flux scram delay time, reactor temperature coefficients, and total negative reactivity available for shutdown. In this simulation, only the reactor, and not the associated heat-transport system, was considered.

Future work will involve analysis of the startup accident and the initial setup of the loss-of-coolant-flow study.

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