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PROGRESS RELATING TO CIVILIAN APPLICATIONS
DURING JUNE, 1957

by

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REPORTS RELATED TO CIVILIAN APPLICATIONS
ISSUED DURING JUNE, 1957

- BMI-1184 "Fabrication of Dispersed Uranium Fuel Elements Using Powder-Metallurgy Techniques", by Stan J. Paprocki, Donald L. Keller, and George W. Cunningham.
- BMI-1187 "The Constitution of Zirconium-Uranium Alloys Containing Oxygen or Nitrogen", by Arthur A. Bauer, Glenn H. Beatty, Frank A. Rough, and Ronald F. Dickerson.
- BMI-1189 "Progress Relating to Civilian Applications During May, 1957", by Russell W. Dayton and Clyde R. Tipton, Jr.

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A. DEVELOPMENT OF MATERIALS FOR HANFORD REACTORS

F. R. Shober

Assembly of equipment to determine the feasibility of making thermal-conductivity and electrical-resistivity measurements on irradiated specimens is being completed. The creep properties of Zircaloy 2 and Zircaloy 3A are being investigated at elevated temperatures. The burst strengths of welded zirconium and Zircaloy 2 tubes are being determined at room and elevated temperatures.

Several lengths of nickel-plated solid uranium cylinders have been successfully clad with aluminum by a coaxial extrusion-cladding technique. A good bond was obtained between the nickel plate and the aluminum cladding. Additional aluminum single crystals have been prepared by the strain-anneal technique. Materials and equipment are being prepared for the growth of molybdenum and zirconium single crystals.

Thermal Conductivity of Uranium

H. W. Deem and C. F. Lucks

A new apparatus is being assembled to determine the feasibility of making thermal-conductivity and electrical-resistivity measurements on irradiated specimens.

Shop work has been completed and all parts of the apparatus have been received. Assembly of the apparatus and preliminary testing should be completed by the end of July.

Mechanical Properties of Zirconium and Zircaloy 2

F. R. Shober, L. L. Marsh, and J. A. VanEcho

The creep properties of Zircaloy 2 and Zircaloy 3A are being determined at elevated temperatures. The differences in properties existing from lot to lot of material are being investigated. The burst strength of welded Zircaloy 2 tubes is being determined at elevated temperatures for comparison with data obtained from seamless tubes.

The results of the creep tests are shown in Table A-1. In each case where data exist from the two lots of material at the same stress, they are tabulated. In some instances, data from duplicate specimens from the same lot are given for comparison. A test in progress for 4200 hr at 650 F and 12,500 psi shows a total elongation of 0.43.

The burst strengths of several welded Zircaloy 2 tubes have been determined at room temperature and 300 F and are given in Table A-2. The failures generally occurred in the vicinity of the weld. Additional tests will be made at 450 and 650 F.

TABLE A-1. CREEP PROPERTIES OF ANNEALED ZIRCALLOY 2 AND ZIRCALLOY 3A

Lot	Specimen	Temperature, F	Stress, psi	Time on Test, hr	Deformation at Indicated Time, per cent						Load Off	Creep Rate ^(a) , per cent per hr
					Load On	50 Hr	100 Hr	500 Hr	1000 Hr	Total		
<u>Zircaloy 2</u>												
1	6-25	300	25,000	1005.0	0.385	1.50	1.65	2.17	2.45	2.50	--	0.0005
2	20-1	300	25,000	700 (b)	0.25	3.10	3.40	4.0	--	--	--	--
2	31-2	300	25,000	1000.0	0.23	2.90	3.15	3.60	3.87	--	--	0.00054
1	6-10	450	23,000	1000.0	0.30		2.05	2.13	2.15	2.15	--	0.00002
2	1-5	450	23,000	675 (b)	0.10	3.68	3.74	3.76	--	--	--	--
2	1-6	450	20,000	750 (b)	0.25	2.35	2.38	2.40	--	--	--	--
2	40-7	550	17,500	1103.9	0.50	1.13	1.15	1.155	1.18	1.181	0.981	0.000025
2	20-3	550	20,000	250 (b)	2.10	3.02	3.03	--	--	--	--	--
2	1-2	550	24,000	1032.8	4.00	16.2	16.2	16.25	16.29	16.29	16.01	0.000040
1		650	9,000	3462	0.032	0.06	0.072	0.072	0.081	0.091	--	<0.000010
2	1-3	650	9,000	850 (b)	0.075	0.088	0.092	0.1	--	--	--	--
2	3-2	650	9,000	3770.2	0.06	0.09	0.10	0.117	0.117	0.117	0.085	<0.000010
1	6-13	650	12,500	1000	0.17	0.42	0.428	0.448	0.46	0.46	--	0.000024
2	3-1	650	12,500	4200 (b)	0.15	0.23	0.245	0.282	0.335	0.43	--	0.00005(c)
2	1-4	650	12,500	1029.1	0.10	0.15	0.155	0.192	0.220	0.22	0.113	0.000028
2	15-1	650	15,000	75 (b)	0.35	0.65	--	--	--	--	--	--
<u>Zircaloy 3A</u>												
-	3-9	300	23,000	575 (b)	0.20	2.15	2.36	2.9	--	--	--	--
-	3-10	300	30,000	114.3	0.30	26.0	--	--	--	59.2	--	--
-	3-7	400	20,000	750 (b)	0.25	2.70	2.86	3.14	--	--	--	--
-	3-8	400	17,500	675	0.25	1.58	1.62	1.67	--	--	--	--

(a) Based on creep deformation between 500 and 1000 hr.

(b) Tests in progress.

(c) Based on creep deformation between 3000 and 4000 hr.

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TABLE A-2. BURST STRENGTH OF WELDED ZIRCALOY 2 TUBES AT ROOM TEMPERATURE AND 300 F

Specimen	Material	Nominal OD, in.	Nominal Wall Thickness, in.	Measured OD, in.	Measured Wall Thickness, in.	Cold Work, per cent	Burst Pressure, psi	Burst Stress, psi (a)	Circumferential Elongation, per cent
<u>Room Temperature</u>									
486-2-1	Zirconium	1.5	0.020	1.510	0.0250	10	3900	113,650	23.1
486-2-2	Ditto	1.5	0.020	1.505	0.0250	10	3850	112,190	17.1
483-1-1	Zircaloy 2	1.5	0.035	1.505	0.0385	10	5300	99,520	17.8
483-1-2	Ditto	1.5	0.035	1.503	0.0380	10	5600	105,221	16.6
<u>300 F</u>									
486-2-3	Zirconium	1.5	0.020	1.505	0.0250	10	3200	93,060	30.5
486-2-4	Ditto	1.5	0.020	1.506	0.0250	10	3200	93,250	12.9

(a) Calculated by Barrow's thin-wall formula, $S = \frac{PD}{2t}$ where S = hoop stress, P = internal pressure, D = inside diameter, and t = wall thickness.

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Zirconium Cladding

F. R. Shoher and R. F. Dickerson

Hot hydrostatic pressing is being investigated as a method for cladding zirconium to nickel-plated and to electropolished uranium. The cladding has been successfully bonded to both of the differently prepared cores.

Several additional specimens are being pressurized under conditions to produce a sound bond. These specimens, when completed, will be sent to Hanford for testing and evaluation.

Extrusion Cladding of Round Bars

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

Several exploratory tests were made to investigate the application of the coaxial extrusion-cladding procedure to the cladding of solid, cylindrical HAPO fuel elements. A four-port mandrel was used with a mandrel-to-die distance of about 0.065 in.

In the first test, a core length of about 26 in. was clad with a 0.050-in. cladding of 1100 aluminum. This length was obtained by connecting two nickel-plated uranium round bars with an M-355 aluminum powder-metallurgy-product plug, and attaching a steel plug at the front end and an M-355 plug at the back end. Threaded joints were used to connect the sections together. No difficulty was experienced in cladding at 1050 F and a speed of about 10 in. per min. The maximum pressure was about 38,000 psi on the billet. Metallographic examination of cross sections of the clad bars indicated that a uniform wall thickness was obtained, and also showed evidence of diffusion layers at the aluminum-nickel and nickel-uranium interfaces. A qualitative "chisel and peeling" test showed that a good bond was obtained between the aluminum and the nickel-plated uranium. The bond between the aluminum and the M-355 end plug was not quite so good.

The cladding experiment was repeated using only uranium and M-355 end plugs. By sawing through the clad bar at the proper points, it will be possible to produce individual clad elements consisting of uranium with M-355 end plugs at each end for other evaluation studies.

One other experiment was made in which the objective was to clad over a uranium bar that was followed through the die by a steel end plug resting on, but not mechanically connected, to it. A separation of about 1/8 in. was caused at the junction between the two bars by aluminum penetration but, in spite of this, the extruding aluminum gripped the steel core and pulled it through the die. This is an indication that it may be possible to clad successive fuel elements and end plugs with only simple connections between them, since it appears that relatively little pressure is required to hold them in contact as they pass through the mandrel into the die.

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The results obtained in these exploratory experiments have demonstrated that it is possible to extrusion clad the cylindrical fuel elements. Thus, the objective of this preliminary project has been achieved, and future work can be concerned with new designs and development of optimum operating conditions, specifically for HAPO fuel-element configurations.

Preparation of Aluminum, Molybdenum, and Zirconium Single Crystals

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

Single crystals of high-purity aluminum, molybdenum, and zirconium are to be prepared for irradiation studies. Aluminum single crystals have been grown from thin sheet by the strain-anneal method.

Additional single crystals of high-purity aluminum (99.99 per cent) have been grown in the form of standard 0.505-in.-diameter tensile bars by the strain-anneal technique. Excellent results were experienced, as an above-average yield of single crystals was obtained. These crystals will be shipped to HAPO.

Construction of the modified Andrade furnace is nearly complete. Molybdenum rods for this growth have been fabricated.

Chemical analyses indicate that the zirconium stock obtained is of high purity. Standard 0.125-in.-diameter tensile bars are being made for use in a strain-anneal method.

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B. DEVELOPMENTS FOR ALUMINUM-CLAD FUEL ELEMENTS

R. J. Carlson

Work has continued on the evaluation of variables involved in the extrusion-cladding process. Preheating of steel cores to 1050 F appeared to eliminate to some extent the erratic pressure variations previously noted and also resulted in lower cladding pressures.

The casting of full-size extrusion billets of aluminum-uranium alloys containing up to 50 w/o uranium has been delayed pending the receipt of suitable molds. However, melting studies have been conducted, using small right-cylinder molds. The use of a Zirconite sand mold fired at 1800 F yielded material comparable to that poured into graphite or metal molds.

The related study concerned with the effect of specific impurities, i.e., magnesium and silicon, on the casting properties of uranium-aluminum alloys with a high uranium content has been initiated.

Extrusion Cladding of Flat Plates

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

Work was continued toward the evaluation of variables involved in the extrusion-cladding process.

Mandrel Tip 5, designed with a heavy center web that was intended to reinforce the mandrel slot, was modified a second time to increase the metal flow toward the center of the core to be clad. The test results showed that the metal flow was still restricted and further modification of this mandrel tip is required.

Several tests were made which indicated that the temperature of the core had an appreciable effect on the extrusion-cladding pressure. Preheating steel cores to about 1050 F seemed to eliminate some of the erratic pressure variations obtained in the past, and also resulted in lower cladding pressures. It was also observed that the die temperature did not decrease as usual during the course of the extrusion. This probably is an indication that some of the pressure increases noted previously were related to chilling of the aluminum by the cooler core and conduction of heat away from the die.

Further progress was made on the design of the "right-angle" extrusion-cladding assembly, and most of the details have been worked out.

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Study of the Liquidus in Aluminum-Uranium Alloys

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

The aluminum-uranium system is the subject of an investigation to determine the liquidus line between 16 and 50 w/o uranium. Also being studied is the effect additions of silicon and magnesium may have on the liquidus temperatures. Techniques being used in this investigation include thermal analysis, differential thermal analysis, electrical-resistivity measurements, and metallography.

Alloys containing 12, 13.2, 14, 17.2, 20, 30, 40, and 50 w/o uranium have been prepared. Silicon additions up to 1 w/o are being made at each composition through use of a series of silicon-uranium-aluminum alloys containing 50 w/o silicon. Additions of magnesium up to 0.5 w/o are also being made to each base alloy.

Preparation of Aluminum-Uranium Alloys

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

The development of melting and casting techniques capable of producing sound homogeneous aluminum-uranium extrusion billets containing up to 50 w/o uranium is the primary objective of a program of research now in progress. Of further interest is the maximum uranium content that can be incorporated into such castings by production techniques.

The casting of full-size extrusion billets has been delayed pending the receipt of suitable molds. However, during the interim, melting studies have been conducted on the high-uranium alloys using small right-cylinder molds. Molding materials investigated included graphite, metals, and ceramics. The use of a Zirconite sand mold fired at 1800 F yielded material comparable to that poured into graphite or metal molds. Melts weighing 6 kg each have been prepared and will be cast into graphite molds to evaluate various methods of gating and feeding.

The related study concerned with the effect of specific impurities on the casting properties of the high-uranium alloys has been initiated. The impurities, i.e., magnesium and silicon, are being introduced into the melts in the form of master alloys. Ingots in the form of 1-in.-diameter rounds containing up to 1 w/o of impurities are required for these studies.

Development of a Mechanical Extensometer

F. R. Shoher and R. F. Dickerson

An extensometer of sufficiently high sensitivity and range to determine the transverse mechanical properties of uranium tubes is being developed.

Several modifications of an existing extensometer have been considered, and studies of other types are being made prior to building and testing.

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C. PLANT ASSISTANCE TO MCW

A. A. Bauer

Reports covering the gas-metal and uranium oxides studies are being prepared.

The effect of nitrogen on the electrical properties of uranium dioxide has continued to receive study with attempts being made to add nitrogen in the form of UNH to the oxide.

Corrosion tests of Type 304 ELC stainless steel in fluoride- and chloride-contaminated nitric acid solutions have continued. With the exception of specimens exposed to a 30 w/o nitric acid solution containing 0.50 w/o chloride, corrosion rates in 5, 18, and 30 w/o nitric acid solutions have remained essentially constant under long-term tests. Specimen corrosion rates have remained unchanged after seven 48-hr exposure periods to 45 w/o nitric acid solutions containing chlorides.

Investigation of Uranium Oxides

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

A paper summarizing the past year's work on the reactivity of uranium oxides, UO_3 and UO_2 , was prepared, and is being submitted for publication. Further studies on the pore structure and the nature of bonding of nitrogen atoms in the uranium oxides are being planned.

The Electrical Properties of Uranium Dioxide

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

The study of the effects of nitrogen on the electrical properties of uranium dioxide was continued. During June, an attempt to dope the oxide with nitrogen by adding uranyl nitrate was made. It was expected that the nitrate would decompose when the specimens were annealed at high temperatures and that nitrogen would be absorbed by the oxide. However, the specimens were lost during the hydrostatic pressing process when the rubber tubes burst. Additional specimens are now being prepared.

The Corrosion Resistance of Selected Stainless Steels

C. L. Peterson, W. C. Baytos, F. W. Fink, and R. S. Peoples

The investigations of the corrosion of Type 304 ELC stainless steel by nitric acid solutions that have been contaminated with chlorides and fluorides, similar to solutions

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which might occur in the nitric acid reconcentrator at Weldon Springs, have been continued.

Specimens have now been exposed for 3500 hr in 5.0 and 5000 hr in 18.0 and 30.0 w/o nitric acid solutions. The corrosion rates are remaining essentially constant, with two exceptions. Both of these occur in the case of specimens exposed in 30.0 w/o nitric acid solution containing 0.50 w/o chloride. As reported in BMI-1189, the corrosion rate of the specimen positioned at the interface is leveling off to a value of 1 to 2 mils per month. The specimen submerged in the liquid shows a corrosion rate which started to increase at 4000 hr and which has now reached 0.38 mil per month. There has been no serious corrosion in solutions containing 0.10 w/o chloride or less.

The corrosion rate of the specimen submerged in the liquid of the 5.0 w/o nitric acid solution containing 0.30 w/o chloride has begun to decrease and stands at 0.51 mil per month following 1500 hr exposure.

A total of seven 48-hr exposure periods were completed with 45.0 w/o nitric acid solutions containing chloride additions. The results after seven periods were no different from those reported in BMI-1189 following the completion of five exposure periods.

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D. PROCESSING OF FEED MATERIALS

E. L. Foster, Jr.

The studies of interest in the processing of feed materials which were conducted during the past fiscal year have been concerned with two subjects. These were the development and testing of salt baths for the beta heat treatment of uranium and a study of the factors contributing to galling in dies used for the compacting of uranium tetrafluoride-magnesium mixtures.

The motivation of the work concerned with the salt-bath developments was the detrimental effect of hydrogen in uranium. In the fabrication of fuel slugs, the uranium is heat treated to obtain a beta structure. During this heat treatment, hydrogen is picked up by the uranium from the salt bath.

In the course of this investigation, two salt baths were chosen for their property of reducing the quantity of hydrogen picked up by uranium heat treated in them. The baths were tested with controlled additions from baths now in use at NLO.

Results from tests indicated that the all-chloride salt bath (50 w/o KCL-50 w/o NaCl) was more effective in reducing the hydrogen picked up by uranium than the chloride-carbonate bath (50 w/o KCl-50 w/o NaCO₃). In both baths, the weight loss of uranium and corrosion attack by the salt on containers and electrodes were slight.

Testing was extended into the high-humidity summer months with specimens being heat treated in the all-chloride bath and in the bath presently used at NLO. Conclusions on the results of these tests have not been drawn since the analyses for hydrogen contents of the specimens heat treated are not yet available.

The study of factors contributing to galling in dies used for the compacting of uranium tetrafluoride-magnesium mixtures has also been completed.

Both of the above subjects will be reported in detail in topical reports now being prepared.

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E. GENERAL FUEL-ELEMENT DEVELOPMENT

F. A. Rough

During June, two different methods were used to prepare UO_2 -BeO diffusion couples to determine the diffusion of uranium in dense UO_2 . Although these methods appeared promising, a complete evaluation has not been concluded to date.

Research and development on UO_2 -graphite fuel elements was continued with the successful preparation of uniform UO_2 -graphite cored graphite-clad fuel elements.

Current investigations of the properties which affect the sinterability of BeO showed that unexpected large amounts of sulfate were retained by the ammonia-precipitated hydroxides and by the oxides made from them.

Research on uranium alloys for high-temperature-reactor fuel applications has now been concluded and a topical report covering the work is being prepared.

Successful fabrication techniques have been devised to produce UN and UC dispersed in stainless steel and clad with stainless steel, and work is now in progress to fabricate enriched specimens of this material for irradiation experiments.

Data have been obtained for the activation energy for temperatures above 100 C for the aqueous uranium corrosion reaction.

CERAMICSRefractory Fuel Elements

G. B. Engle, M. J. Snyder, J. F. Quirk, and A. J. Roese

Current research on refractory fuel elements is aimed at measuring the high-temperature diffusion of UO_2 in densely sintered BeO. Also, the evaluation of UO_2 -graphite fuel elements was continued with the fabrication of elements containing dispersions of UO_2 nodules. These elements will be evaluated for fission-product retention and uranium loss.

BeO

In previous attempts to measure the diffusion of uranium in dense BeO tubes, UO_2 coatings applied as one element of the diffusion couple did not adhere satisfactorily to the BeO tube during heating at 2800 F in hydrogen.

During June, in the attempt to fabricate reliable diffusion couples, UO_2 -BeO composites were prepared by two methods: (1) finely ground UO_2 was packed into half the length of the bore of a dense BeO tube, and (2) a closely fitted sintered UO_2 rod was inserted in half the length of the bore of a BeO tube and finely ground UO_2 was introduced as an aqueous slurry, to fill the annular space between rod and tube.

The couple prepared with the packed UO_2 powder core was heated at 2400 F for 48 hr in vacuo. During heating, the UO_2 sintered and shrank away from the tube bore, leaving only a narrow band of contact inside the tube. A chord section was removed by grinding away a portion of the tube wall adjacent to the contact band, producing a flat extending the length of the tube. The ground portion of the tube, with a minimum wall thickness of 0.024 in. at the center of the flat, was placed in contact with an alpha-sensitive photographic plate for 7 days. Examination of the plate showed a band of alpha exposure uniform in density over the entire length of the chord section. Since the diffusion couple extended only half the length of the tube, the significance of the experiment is not yet known. Evaluation of this sample is being continued.

The couple prepared with a sintered UO_2 core was heated at 2400 F for 48 hr in vacuo. After heating, the UO_2 core appeared to be still firmly in contact with the tube bore. This couple will be examined by the autoradiographic method.

Graphite

In continuing the evaluation of UO_2 -graphite fuel elements for possible use in a nitrogen-cooled power reactor, a new batch of fuel-element prototypes were fabricated utilizing UO_2 nodules dispersed in a graphite matrix.

The graphite elements were prepared by extruding a mixture of pregraphitized coke, UO_2 nodules of about 200 to 300 μ in diameter, and a resin binder. The soft extruded cores then were coextruded with additional graphite mix which contained no UO_2 . The resultant elements were heated at 400 F to cure the binder. X-ray photos showed the UO_2 -graphite cores to be centrally located within the outer graphite sheath and the UO_2 to be uniformly distributed within the cores. The elements will contain about 2.7 w/o UO_2 after the binder is carbonized.

Evaluation of these elements for fission-gas retention and uranium loss is planned.

Characterization of Sinterable Oxide Powders

J. F. Quirk and N. Mosley

Basic properties that affect the sinterability of oxide powders are being assessed. Currently, this work involves an investigation of BeO made from Be(OH)₂. Previous work indicated that impurities in the starting hydroxide, as well as calcining temperature, have an important influence on sinterability of the BeO.

In May, six samples of $\text{Be}(\text{OH})_2$, prepared by ammonia precipitation from BeSO_4 solution, were analyzed to determine sulfate content and loss on ignition. During June,

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similar analyses were made on the BeO samples obtained by calcining the hydroxides for 2 hr in air at 1470 F. Also, sinterability of the calcined oxides was assessed by measuring the density of compacts sintered 1 hr at 2600 F in an atmosphere of 10 volume per cent hydrogen-90 volume per cent argon. The purpose of these experiments was to characterize the oxide powders in terms of their volatile impurities. The results of the experiments are given in Table E-1.

These analyses showed that unexpectedly large amounts of sulfate were retained by the ammonia-precipitated hydroxides and by the oxides made from them. Repeated washing of the precipitated hydroxides reduced the sulfate content from about 40 to 7.6 w/o, but did not significantly affect sinterability of the calcined oxides. Sulfate contents of the oxides calcined at 1470 F ranged from 2.1 to 6.1 w/o, and were, in general, higher than their weight loss on ignition. This indicates that the sulfate was not completely removed by heating (ignition) for 3 hr in air at 1650 F. It was noted, as confirmation of previous results, that purification by recrystallization of the starting $\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$ gave decreased sinterability of the oxide. Also, deliberate contamination of the starting BeSO_4 solution with MgSO_4 and CaSO_4 gave improved sinterability.

A readily sinterable BeO made from commercial NaOH-precipitated hydroxide was used as a reference material in these experiments. This material had a much lower sulfate content and loss on ignition than did the comparable ammonia-precipitated materials.

Effort next month will be directed toward identifying anion constituents of the commercial hydroxide and its oxide calcination product. Also, the effect of various calcining and sintering atmospheres on removal of sulfate contaminant will be studied.

METALLURGYUranium Alloys for High-Temperature Application

W. E. Murr, A. A. Bauer, and R. F. Dickerson

The project concerned with elevated-temperature properties of binary uranium alloys for use as power-reactor fuels is being concluded. A topical report which will summarize the developmental work of the program is being prepared. The report will include the elevated-temperature mechanical properties of alloys of uranium-3.5 through 12 w/o molybdenum and uranium-3 through 20 w/o zirconium as well as the physical and mechanical properties of the 7 through 15 w/o niobium alloys.

Compartmentalized Fuel Elements

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

Preparation of irradiation specimens of UN and UC dispersed in stainless steel and clad with Type 347 stainless is in progress. In one series of specimens the core is

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TABLE E-1. VOLATILE CONSTITUENTS IN BeO POWDER

Compound Analyzed	BeSO ₄ ·4H ₂ O Starting Material	Precipitant	Number of Washings	Calcining Temperature, F	SO ₄ Content, w/o	Loss on Ignition for 3 Hr at 1650 F, w/o	Bulk Density of Sintered BeO Compacts, per cent of theoretical density
Be(OH) ₂	Brush cp	NH ₃	None	--	(40) ^(a)	65.3	--
BeO	Ditto	--	--	1470	4.8	3.5	89.3
Be(OH) ₂	Ditto	NH ₃	5	--	15.5	50.0	--
BeO	Ditto	--	--	1470	5.3	3.8	87.7
Be(OH) ₂	Ditto	--	20	--	7.6	52.1	--
BeO	Ditto	--	--	1470	4.8	3.4	88.7
Be(OH) ₂	Recrystallized	NH ₃	None	--	43.5	71.5	--
BeO	Brush cp	--	--	1470	6.1	3.9	82.4
Be(OH) ₂	Contaminated Brush cp ^(b)	NH ₃	None	--	34.2	63.3	--
BeO	Ditto	--	--	1470	5.6	4.9	95.8
Be(OH) ₂	Contaminated Brush cp ^(c)	NH ₃	None	--	43.5	71.1	--
BeO	Ditto	--	--	1470	2.1	4.6	94.7
Be(OH) ₂ ^(d)	Brush technical grade	NaOH	Washed	--	0.03	41.1	--
BeO	Ditto	--	--	1470	0.03	0.56	96.5

(a) Estimate from incomplete analysis—being analyzed.

(b) The sulfate was contaminated by addition of 0.5 w/o CaSO₄ and 0.5 w/o of MgSO₄.(c) The sulfate was contaminated by addition of 0.67 w/o CaSO₄ and 0.33 w/o of MgSO₄.

(d) Hydroxide Lot C5659, from Brush Beryllium Company, was used as a reference material.

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E-5 and E-6

to contain 24.2 w/o UN, while in the second series the core will contain 24.3 w/o UC. These loadings are equivalent to a 27 w/o UO_2 dispersion in stainless steel. Specimens are to be irradiated to burnups of 5, 10, 15, and 20 a/o of uranium-235.

Fabrication of dummy specimens has been completed. Metallographic examination indicates that uniform dispersions and sound core-to-cladding bonds may be obtained by current fabrication techniques. The UN (minus 200 plus 270 mesh)-stainless cores are roll clad at 2000 F, while the UC (minus 100 plus 150 mesh)-stainless cores are roll clad at 1800 F. Type 302B stainless is used as matrix powder in both cases, and picture frames are machined from Type 347 stainless.

UC and UN powders have been prepared from fully enriched uranium metal (93.14 w/o uranium-235), and irradiation specimens will be fabricated during the next report period.

CORROSIONMechanism of Uranium Corrosion

J. B. Schroeder, C. M. Schwartz, H. D. Hannan,
P. D. Miller, and R. S. Peoples

The mechanism of aqueous uranium corrosion is being studied as part of a program to develop corrosion-resistant alloys for reactor fuels.

A paper discussing a possible corrosion mechanism at temperatures up to 100 C was written and submitted for publication.

Experimental work on the corrosion of dingot material in moist-argon atmospheres at temperatures above 100 C was continued. Data obtained at 100, 150, and 200 C indicate an activation energy of 8.9 kcal for the corrosion reaction.

F-1

F. STUDIES OF ZIRCONIUM-URANIUM ALLOYS

F. A. Rough

This section reports the progress of the research on the fundamental properties, corrosion resistance, and radiation stability of zirconium-uranium alloys.

Corrosion data on clad zirconium-22 and 50 w/o uranium elements indicate that water quenching the clad material from the rolling temperature destroys the corrosion resistance of the clad samples.

Results of corrosion tests on heat-treated uranium-zirconium-niobium alloys are reported.

Long-term irradiations are now under way for uranium-50 w/o zirconium specimens.

Heat Treatment and Mechanical Properties of Zirconium-Uranium Alloys

D. L. Douglass, L. L. Marsh, Jr., and G. K. Manning

Experimental work has been completed in the determination of the time-temperature-transformation curves of zirconium-uranium alloys containing oxygen. A topical report is being prepared.

Corrosion of Zirconium-Uranium Alloys

W. Chubb, W. E. Berry, R. S. Peoples, and R. F. Dickerson

One phase of the program on determining properties of zirconium-uranium alloys is the evaluation of their corrosion behavior in high-temperature water. A study is being made of the effect of postcladding heat treatment on the corrosion behavior of Zircaloy 2-clad fuel elements in 680 F water.

Cross sections of fuel elements with zirconium-22 or 50 w/o uranium cores are being exposed to follow the progress of corrosion in the core and in the alloy-diffusion zone at the core-cladding interface. The core in the zirconium-70 w/o uranium fuel elements is being exposed through a 0.040-in.-diameter hole drilled through the cladding and halfway through the core. Samples were given the following postcladding heat treatments:

- (1) None (as roll-bonded at 800 C)
- (2) 800 C, 1/2 hr, water quench
- (3) 400 C, 10 min, air cool
- (4) 650 C, 1 hr, air cool
- (5) 900 C, 1 hr, air cool
- (6) 800 C, 1/2 hr, slow furnace cool.

Areas of intermittent bonding between core and cladding or cladding and cladding were discovered in samples after roll cladding. Interpretation of corrosion results is difficult because of this. After a 42-day exposure in 680 F water, attack at the core-cladding interface has caused the cladding to bulge slightly on all samples with 50 w/o uranium cores. Several samples with 22 w/o uranium cores exhibited bulging of the cladding from oxide buildup in a confined area where separation at the Zircaloy 2-Zircaloy 2 bond line allowed water to reach the core. The trend in the results obtained to date indicates that water quenching from the rolling temperature destroys corrosion resistance. Cracking or rapid attack at the core-cladding interface occurred within a 14-day exposure on water-quenched samples with 22 and 50 w/o uranium cores. Cracking at the core-cladding interface also was observed on a 22 w/o sample which had been aged 10 min at 400 C after roll bonding.

Only the as-roll-bonded sample of those with 70 w/o uranium cores has survived 42 days of exposure in 680 F water. A pimple, observed at the defect in this sample after 31 days of exposure, is 1/16 in. in diameter after 42 days of exposure. The core in the water-quenched sample was completely oxidized and the cladding ruptured after 3 days of exposure. Samples which were slow cooled from 800 C or were air cooled from 650 C were removed from test after 9 and 35 days of exposure, respectively, because of general oxidation of the core outward from the defect. The samples which were aged 10 min at 400 C or were air cooled from 900 C failed from oxidation of the core at faulty Zircaloy 2-Zircaloy 2 bond lines at the edges of the samples.

Microsections are being made on failed samples to ascertain the progress of attack.

Development of Corrosion-Resistant 70 w/o Uranium Alloys

V. W. Storhok, A. A. Bauer, W. E. Berry, and R. S. Peoples

An investigation designed to improve the corrosion resistance of the zirconium-70 w/o uranium alloy either by heat treatment of the base alloy or by ternary alloying additions is being conducted. Ternary additions of molybdenum, niobium, silicon, and titanium were made with the idea of either tying up the free alpha uranium found in the 70 w/o alloy or of stabilizing the gamma phase.

The molybdenum and silicon ternary alloys along with the uranium-zirconium alloy have been clad with Zircaloy 2 and are undergoing heat treatment. These clad alloys will be corrosion tested in 680 F water; the cladding will be artificially defected with 40-mil-diameter pinholes.

Corrosion testing of bare uranium-zirconium-niobium alloys is continuing. Preliminary results show that after several different heat treatments were investigated, specimens of uranium-23.7 w/o zirconium-5.3 w/o niobium are most corrosion resistant after a heat treatment of 24 hr at 900 C followed by an air cool. An 11 w/o niobium-17.9 w/o zirconium alloy was most corrosion resistant after being quenched from 800 C and aged at 360 C for 15 min or 500 C for 24 hr. Additional uranium-zirconium-niobium alloys containing 3 and 5 w/o niobium have been prepared and will be corrosion tested bare, as well as clad, in 680 F water.

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Radiation Stability of Zirconium-Uranium Alloys

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

Research is continuing on a program of study of the effects of radiation on zirconium-uranium alloys. A temperature-controlled capsule which contains two specimens of zirconium-50 w/o uranium alloy of two different heat treatments is currently at the MTR awaiting reactor test space. The most recent schedule has the capsule listed for insertion into the reactor on August 5, 1957.

Work has been initiated for the construction of a test capsule not temperature controlled which will contain two specimens of zirconium-50 w/o uranium. It is planned to irradiate these specimens to a burnup of 2 a/o.

G-1

G. CORROSION STUDIES OF ZIRCONIUM

R. S. Peoples

The AEC Reactor Development Division research reported in this section pertains to a study of the long-term corrosion behavior of zirconium-base cladding alloys in elevated-temperature water and steam.

Corrosion studies have been continued in 600 and 680 F degassed water and 750 F steam.

Corrosion Studies of Zirconium and Zirconium Alloys

W. K. Boyd and R. S. Peoples

The corrosion samples representing seven different melts of Zircaloy 2 have been exposed up to 38 months in 600 F degassed water. Although total weight gains are of the order of 50 mg per dm², the samples still exhibit a dark tarnish film and there is no indication of transition or breakway.

In other studies, intentionally contaminated Zircaloy 2 samples are being evaluated in 680 F degassed water. After approximately 25 months of exposure, corrosion results are as follows:

- (1) Oxygen contents in excess of 6000 ppm or nitrogen contents of 200 ppm or higher result in a complete loss of corrosion resistance.
- (2) Hydrogen in the range 500 to 1500 ppm has no effect on the corrosion resistance of Zircaloy 2 in 680 F water.

Zirconium Corrosion Committee Program

In cooperation with the Zirconium Corrosion Committee program of alloy development, modified Zircaloy 2 compositions, i. e., lower tin with iron and/or nickel, are being studied in 680 F degassed water and superheated steam (750 F, 1500 psi). Corrosion rates based on up to 639 days of exposure are tabulated in Tables G-1 through G-3. Results may be summarized as follows:

- (1) The corrosion resistance of the alloys under investigation markedly decreases with increasing nitrogen levels above 70 ppm.
- (2) Alloys containing 0.25 to 0.5 w/o tin and 0.25 to 0.5 w/o iron and/or nickel have good resistance to both 680 F water and 750 F steam. The data, however, indicate that the iron-containing alloys are somewhat more resistant to 680 F water than are the tin-nickel alloys, whereas the reverse is true for 750 F steam.

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- (3) Heat treatments consisting of either slow cooling from 775 C or water quenching after a soak at 900 C do not result in any significant change in the corrosion properties of either the tin-iron or tin-nickel alloys.
- (4) Aluminum additions of 200 ppm decrease the corrosion resistance of the modified Zircaloy 2 compositions.

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TABLE G-1. CORROSION OF SPONGE-BASE ZIRCONIUM ALLOYS IN 750 F STEAM AND 680 F DEGASSED WATER

Alloys Vacuum Annealed 1 Hr at 775 C and Furnace Cooled

Heat	Alloy Composition (Balance Zirconium), w/o	750 F Steam			680 F Water		
		Exposure Time, days	Weight Gain, mg per dm ²	Remarks(a)	Exposure Time, days	Weight Gain, mg per dm ²	Remarks(a)
556	0.05Fe-0.002Ni-0.007N	105	Lost weight	W, F	333	105	M
585	0.05Fe-0.002Ni-0.007N	105	Lost weight	W, F	305	193	W, F
557	0.05Fe-0.15Ni-0.007N	207	99	M	501	110	M
558	0.05Fe-0.15Ni-0.015N	172	111	M	305	107	M
559	0.05Fe-0.15Ni-0.0225N	159	114	M	277	114	M
560	0.05Fe-0.15Ni-0.030N	118	137	M	221	140	M
563	0.05Fe-0.15Ni-0.007N-0.5Sn	166	104	M	473	112	M
564	0.05Fe-0.15Ni-0.015N-0.5Sn	159	103	M	361	103	M
565	0.05Fe-0.15Ni-0.0225N-0.5Sn	159	113	M	305	103	M
566	0.05Fe-0.15Ni-0.030N-0.5Sn	136	119	M	305	130	M
567	0.25Fe-0.25Sn-0.007N	207	89	M	501	92	M
568	0.25Fe-0.25Sn-0.015N	172	109	M	445	118	M
569	0.25Fe-0.25Sn-0.0225N	172	127	M	305	101	M
570	0.25Fe-0.25Sn-0.030N	159	114	M	277	115	M
573	0.40Fe-0.50Sn-0.007N	172	110	M	501	106	M
574	0.40Fe-0.50Sn-0.015N	172	119	M	501	105	M
575	0.40Fe-0.50Sn-0.0225N	136	104	M	361	105	M
576	0.40Fe-0.50Sn-0.030N	124	116	M	305	109	M
579	0.20Fe-0.20Ni-0.50Sn-0.007N	159	110	M	445	105	M
580	0.20Fe-0.20Ni-0.50Sn-0.015N	159	132	M	445	125	M
581	0.20Fe-0.20Ni-0.50Sn-0.0225N	118	120	M	305	119	M
582	0.20Fe-0.20Ni-0.50Sn-0.030N	118	108	M	305	104	M
561	0.05Fe-0.15Ni-0.007N-0.02Al	159	132	M	221	126	M
571	0.25Fe-0.25Sn-0.007N-0.02Al	136	112	M	249	121	M
577	0.40Fe-0.50Sn-0.007N-0.02Al	124	114	M	305	119	M
583	0.20Fe-0.20Ni-0.50Sn-0.007N-0.02Al	136	112	M	445	127	M
562	0.05Fe-0.15Ni-0.007N-0.01Ti	159	105	M	305	113	M
572	0.25Fe-0.25Sn-0.007N-0.01Ti	118	115	M	277	112	M
578	0.40Fe-0.50Sn-0.007N-0.01Ti	159	119	M	473	114	M
584	0.20Fe-0.20Ni-0.50Sn-0.007N-0.01Ti	136	108	M	445	122	M

(a) Key to remarks: M - milky; W - white oxide; F - flaking (heavy oxide).

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TABLE G-2. CORROSION OF ZIRCONIUM-BASE ALLOYS MELTED

Heat	Alloy Content					750 F Steam		Remarks(a)
	Sn, w/o	Fe, w/o	Ni, w/o	N, ppm	Al, ppm	Exposure Time, days	Weight Gain, mg per dm ²	
								Vacuum Annealed 2 Hr at
996	0.24	0.25	--	44	58	200	103	M, WP
974	0.25	0.25	--	48	63	200	102	M, WP
997	0.26	0.25	--	33	46	186	98	M, WP
984	0.26	0.25	--	47	68	212	109	M, WP
1001	0.26	0.27	--	55	62	181	145	M, WP
1003	0.48	0.41	--	219	43	135	120	M, WP
975	0.49	0.36	--	57	45	186	115	M, WP
1000	0.49	0.38	--	43	50	186	130	M, WP
983	0.50	0.39	--	53	70	194	118	M, WP
998	0.51	0.38	--	48	47	186	111	M, WP
990	0.48	0.25	0.23	47	56	186	111	M, WP
985	0.49	0.20	--	47	64	194	109	M, WP
1007	0.50	0.21	0.19	50	51	135	110	M, WP
999	0.53	0.20	0.18	64	46	186	123	M, WP
995	0.65	0.22	0.19	38	57	186	116	M, WP
1024	0.25	0.25	--	63	68	181	115	M, WP
1026	0.25	0.25	--	63	67	186	121	M, WP
1030	0.50	0.44	--	58	47	186	123	M, WP
1028	0.49	0.38	--	71	38	181	116	M, WP
1014	0.25	0.25	--	60	145	147	112	M, WP
1012	0.25	0.25	--	50	179	147	107	M, WP
1013	0.26	0.26	--	54	275	135	170	M, WP, S
1010	0.51	0.39	--	46	88	135	105	M, WP
1009	0.48	0.38	--	57	148	147	118	M, WP
1011	0.50	0.39	--	62	275	135	123	M, WP
1025	0.50	0.21	0.19	65	62	186	114	M, WP
1023	0.53	0.22	0.20	63	60	181	119	M, WP
1017	0.49	0.21	0.19	53	135	181	137	M, WP
1016	0.51	0.21	0.18	48	154	135	107	M, WP
1015	0.50	0.21	0.19	51	265	147	148	M, WP

(a) Key to remarks: M, WP - milky with a white oxide pattern; W - white oxide; F - flaking (heavy oxide); S - segregated.

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AT THE BUREAU OF MINES FOR ZIRCALOY 3 EVALUATION

680 F Water			750 F Steam			680 F Water		
Exposure Time, days	Weight Gain, mg per dm ²	Remarks ^(a)	Exposure Time, days	Weight Gain, mg per dm ²	Remarks ^(a)	Exposure Time, days	Weight Gain, mg per dm ²	Remarks ^(a)
775 C and Furnace Cooled			Heated 2 Hr at 900 C and Water Quenched					
546	114	M, WP	170	118	M, WP	588	86	M, WP
406	108	M, WP	170	106	M, WP	588	94	M, WP
462	107	M, WP	182	111	M, WP	588	88	M, WP
434	106	M, WP	170	102	M, WP	588	90	M, WP
378	21	M, WP, F	182	111	M, WP	588	103	M, WP
294	125	M, WP	135	136	M, WP, S	448	100	M, WP
462	108	M, WP	170	110	M, WP	588	96	M, WP
434	109	M, WP	170	135	M, WP	532	103	M, WP
434	104	M, WP	170	107	M, WP	588	93	M, WP
434	106	M, WP	170	132	M, WP	588	95	M, WP
462	105	M, WP	170	111	M, WP	560	105	M, WP
490	104	M, WP	170	107	M, WP	588	107	M, WP
294	160	M, WP	135	113	M, WP	364	106	M, WP
434	103	M, WP	170	134	M, WP	532	117	M, WP
546	120	M, WP	170	106	M, WP	560	105	M, WP
378	109	M, WP	158	134	M, WP, S	532	121	M, WP
406	106	M, WP	170	127	M, WP, S	560	104	M, WP
546	87	M, WP, F	158	127	M, WP	532	109	M, WP
406	100	M, WP	158	125	M, WP	532	110	M, WP
322	106	M, WP	158	128	M, WP	476	100	M, WP
322	116	M, WP	123	112	M, WP	448	105	M, WP
182	Lost weight	M, WP, F	105	108	M, WP	364	122	M, WP
266	118	M, WP	105	107	M, WP	448	118	M, WP
294	120	M, WP	105	112	M, WP	448	105	M, WP
266	110	M, WP	93	115	M, WP	364	111	M, WP
378	97	M, WP	158	132	M, WP	532	113	M, WP
378	104	M, WP	158	142	M, WP	448	100	M, WP
378	121	M, WP	105	105	M, WP	448	105	M, WP
322	114	M, WP	93	98	M, WP	420	121	M, WP
266	125	M, WP	93	108	M, WP	364	141	M, WP

TABLE G-3. CORROSION OF ARG-MELTED (ROCKING-HEARTH METHOD) SPONGE ZIRCONIUM IN 750 F STEAM AND 680 F DEGASSED WATER

Alloys Melted at Battelle

Heat	Alloy Composition (Balance Zirconium), w/o	750 F Steam			680 F Water			750 F Steam			680 F Water		
		Expo- sure Time, days	Weight Gain, mg per dm ²	Remarks(a)	Expo- sure Time, days	Weight Gain, mg per dm ²	Remarks(a)	Expo- sure Time, days	Weight Gain, mg per dm ²	Remarks(a)	Expo- sure Time, days	Weight Gain, mg per dm ²	Remarks(a)
Heated 1 Hr at 775 C in Vacuum and Furnace Cooled							Heated 1 Hr at 900 C in Vacuum and Water Quenched						
Tin and Iron													
462	Unalloyed	1-1/2	768	W	210	211	M, F	10	1680	W	28	Lost weight	W, F
463	Unalloyed	1-1/2	839	W	210	210	M, F	10	1325	W	28	66	W, F
464	0.22Sn-0.28Fe	106	108	M	630	105	M	136	109	M	616	105	M
465	0.23Sn-0.30Fe	106	105	M	630	98	M	136	109	M	616	89	M
466	0.22Sn-0.38Fe	136	100	M	630	97	M	136	104	M	616	113	M
467	0.22Sn-0.39Fe	136	100	M	630	92	M	136	100	M	616	92	M
469	0.25Sn-0.27Fe	112	102	M	630	90	M	184	105	M	616	112	M
468	0.25Sn-0.28Fe	112	105	M	630	94	M	136	100	M	616	77	M
470	0.25Sn-0.33Fe	136	102	M	630	94	M	147	100	M	616	102	M
471	0.25Sn-0.34Fe	112	107	M	630	89	M	136	105	M	616	106	M
473	0.27Sn-0.25Fe	106	105	M	630	82	M	184	108	M	616	99	M
472	0.29Sn-0.25Fe	106	115	M	630	84	M	147	96	M	616	94	M
475	0.27Sn-0.28Fe	148	109	M	630	86	M	147	102	M	616	98	M
474	0.28Sn-0.34Fe	106	104	M	630	87	M	136	100	M	616	105	M
477	0.47Sn-0.44Fe	112	112	M	630	83	M	147	111	M	588	112	M
478	0.51Sn-0.47Fe	142	109	M	630	88	M	136	113	M	616	87	M
481	0.52Sn-0.47Fe	136	106	M	630	90	M	136	105	M	588	105	M
479	0.50Sn-0.50Fe	136	95	M	630	87	M	136	110	M	588	115	M
476	0.49Sn-0.51Fe	124	100	M	630	88	M	147	106	M	588	111	M
480	0.51Sn-0.53Fe	124	102	M	630	88	M	136	104	M	588	118	M
Tin and Nickel													
488	Unalloyed	1-1/2	680	W	126	136	M	26	Lost weight	W, F	42	251	W
489	Unalloyed	1-1/2	570	W	154	111	W	26	Lost weight	W, F	42	231	W
491	0.17Sn-0.03Ni	249	114	M	434	109	M	206	105	M	392	134	W, S

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TABLE G-3. (Continued)									
750 F Steam					680 F Water				
Expo-	Alloy Composition	Time,	Weight	Expo-	Alloy Composition	Time,	Weight	Expo-	Alloy Composition
days	sure	days	Gain,	days	sure	days	Gain,	days	sure
Remarks(a)	mg per dm ²	Remarks(a)	mg per dm ²	Remarks(a)	mg per dm ²	Remarks(a)	mg per dm ²	Remarks(a)	mg per dm ²
Heated 1 Hr at 775 C in Vacuum and Furnace Cooled									
Tin and Nickel									
(Continued)									
490	0.18Sn-0.04Ni	231	103	M	434	102	M	242	117
492	0.13Sn-0.08Ni	249	109	M	630	97	M	158	Lost weight
493	0.18Sn-0.08Ni	189	112	M	630	100	M	242	113
495	0.17Sn-0.19Ni	249	107	M	630	109	M	242	116
494	0.19Sn-0.19Ni	249	115	M	574	107	M	206	102
496	0.21Sn-0.04Ni	267	78	W, F	574	118	M	261	Lost weight
497	0.22Sn-0.04Ni	249	117	M	462	102	M	261	Lost weight
498	0.22Sn-0.07Ni	213	102	M	630	99	M	166	Lost weight
499	0.22Sn-0.08Ni	207	115	M	630	96	M	242	106
501	0.21Sn-0.20Ni	213	100	M	630	127	M	201	111
500	0.22Sn-0.41Ni	249	113	M	630	108	M	206	107
502	0.27Sn-0.03Ni	213	108	M	518	114	M	140	Lost weight
503	0.27Sn-0.04Ni	213	108	M	518	117	M	206	Lost weight
505	0.27Sn-0.08Ni	213	100	M	574	107	M	261	Lost weight
504	0.28Sn-0.08Ni	213	100	M	630	106	M	242	110
506	0.26Sn-0.19Ni	213	103	M	574	110	M	206	103
507	0.25Sn-0.20Ni	207	146	M	574	110	M	206	117
508	0.56Sn-0.04Ni	249	106	M	434	102	M	206	108
509	0.56Sn-0.04Ni	213	102	M	574	115	M	206	139
510	0.54Sn-0.07Ni	213	103	M	574	113	M	206	131
511	0.56Sn-0.07Ni	207	117	M	574	104	M	206	141
513	0.51Sn-0.18Ni	207	120	M	518	112	M	201	126
512	0.54Sn-0.20Ni	207	118	M	574	121	M	206	133

TABLE G-3. (Continued)

Alloy Composition (Balance Zirconium), Heat w/o		750 F Steam			680 F Water			750 F Steam			680 F Water		
		Expo- sure Time, days	Weight Gain, mg per dm ²	Remarks(a)	Expo- sure Time, days	Weight Gain, mg per dm ²	Remarks(a)	Expo- sure Time, days	Weight Gain, mg per dm ²	Remarks(a)	Expo- sure Time, days	Weight Gain, mg per dm ²	Remarks(a)
Heated 1 Hr at 775 C in Vacuum and Furnace Cooled								Heated 1 Hr at 900 C in Vacuum and Water Quenched					
Tin, Iron, and Nickel													
483	0.51Sn-0.16Fe- 0.090Ni	124	104	M	490	110	M	136	148	M	420	154	M
482	0.49Sn-0.23Fe- 0.097Ni	148	110	M	602	103	M	136	105	M	588	109	M
485	0.47Sn-0.27Fe- 0.13Ni	136	102	M	574	134	M	136	107	M	532	109	M
484	0.48Sn-0.31Fe- 0.13Ni	136	102	M	630	104	M	136	104	M	588	115	M
486	0.48Sn-0.38Fe- 0.18Ni	148	106	M	602	100	M	130	107	M	588	116	M
487	0.48Sn-0.44Fe- 0.18Ni	148	116	M	574	106	M	130	107	M	588	138	M

(a) Key to remarks: M - milky; W - white oxide; F - flaking (heavy oxide); S - segregated.

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H. REACTOR-MATERIALS DEVELOPMENT

F. A. Rough

This section reports the varied research being done on reactor materials for the AEC.

It is reported that promising results have been obtained by firing a thin film of phosphate enamel on uranium.

Investigation of the oxidation resistance of niobium-zirconium alloys has shown that zirconium increases the oxidation rate at low zirconium concentrations, but decreases the rate at high zirconium concentrations.

Research is being concluded on the determination of the phase diagram for the uranium-rich portion of the uranium-titanium phase diagram, and a topical report on the uranium-titanium alloy system is being prepared.

Progress is reported in the results of coating the inside and outside of a small-diameter stainless steel tube with niobium using the moving-temperature-gradient coater.

Enameling of Special Metals

B. W. King, E. D. Dietz, and M. C. Brockway

This program is concerned with the development of enamel coatings for metals of interest to the AEC. Coatings have been developed for zirconium which are adherent, protective, and resistant to thermal-cycling tests. The coatings developed to date for uranium have not been completely free of crazing defects.

Efforts were continued in June to reduce crazing in phosphate enamels applied to uranium. Reducing the thickness of enamel application, fusing the enamel in air and completing the firing in argon, or mill addition of NH_4Cl to the enamel slip were procedures employed. Reduction of enamel thickness was most effective in reducing crazing.

Firing a thin film of phosphate enamel on uranium which had not been electro-polished in the usual pretreatment procedure produced a coating of considerable interest. As observed visually, this coating was rather dull and rough. Viewed at a magnification of 60 diameters, the coating appeared to consist of a continuous glass phase which was relatively free of bubbles or cracking. Adherence was excellent.

Future work is planned to include further development of the thin enamel coating discussed above. If the irregularity in coating thickness could be smoothed out, this might be a particularly useful coating so long as thicknesses greater than 1 mil are not required.

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Oxidation-Resistant Niobium Alloys

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

The objective of this investigation is to determine the factors which influence the rate and mechanism of oxidation of niobium and niobium-base alloys. Continuous-weighting oxidation tests are being conducted on niobium alloys in air at 1000 C.

Oxidation tests in dry air have been completed on niobium-25 a/o vanadium and on niobium-10, 25, and 35 a/o zirconium. Data are given in Table H-1. Niobium-25 a/o vanadium oxidized linearly at a high rate, in contrast to alloys containing up to 12.5 a/o vanadium, which oxidized parabolically at low rates. The appearance of the oxidized sample and the high oxidation rate indicate that the vanadium oxidized to liquid V_2O_5 , which accelerates oxidation. Weight-gain data indicate that loss of V_2O_5 by volatilization was negligible.

Niobium-10, 25, and 35 a/o zirconium formed white slablike scales when oxidized, similar to pure niobium. Niobium-10 a/o zirconium oxidized parabolically, but with a high rate [$4075 \text{ (mg/cm}^2)^2 \text{ per hr}$] compared to parabolic rates observed on niobium-molybdenum and niobium-vanadium alloy [$43 \text{ to } 227 \text{ (mg/cm}^2)^2 \text{ per hr}$]. Alloys containing 25 and 35 a/o zirconium oxidized linearly with decreasing rates. Thus, zirconium increases the oxidation rate at low concentrations but decreases the rate at high concentrations. Niobium-45 a/o zirconium has also been prepared for testing.

Future work includes:

- (1) Continuation of continuous-weighting air-oxidation tests on binary niobium alloys containing chromium, tungsten, vanadium, and zirconium.
- (2) Comparison of the oxidation behavior of metallographically polished pure niobium with previous results obtained on niobium abraded with silicon carbide metallographic paper.

Uranium-Titanium Alloy Studies

D. L. Douglass, L. L. Marsh, Jr., and G. K. Manning

The experimental work on the determination of the phase diagram for the uranium-rich portion of the uranium-titanium phase diagram has been completed. An additional series of hot-hardness determinations for the compound U_2Ti was made for both heating and cooling cycles. It was found that, upon heating, the compound decomposed congruently to the gamma body-centered-cubic solid solution of titanium in uranium at 900 C. This agrees closely with published values of 890 C. The results for the cooling cycle are as yet incomplete.

TABLE H-1. OXIDATION DATA FOR BINARY NIOBIUM ALLOYS IN DRY AIR AT 1000 C

Sample	Composition, a/o	Parabolic Rate Constant, (mg/cm ²) ² per hr	Transition to Linear Rate, hr	Linear Rate Constant, (mg/cm ²) per hr	Weight Gain, mg per cm ² , After Indicated Time					
					1 Hr	2 Hr	3 Hr	4 Hr	5 Hr	6 Hr
0-30 ^(a)	100 Nb	--	0.1	30.0	30.0	61.3	89.8	114.2	136.9	158.6
0-59	Nb-25V	--	--	303	(b)	--	--	--	--	--
0-61	Nb-10 Zr	4075	--	--	62.4	89.8	(c)	--	--	--
0-63	Nb-25 Zr	1480	0.5	27.0	42.5	69.3	94.8	117.0	133.7	--
0-65	Nb-35 Zr	1120	2.2	9.22	33.0	47.7	57.6	66.8	76.2	84.6

(a) Data from previous test included for comparison.

(b) Sample was completely oxidized in 42 min.

(c) Sample lost in oxidation furnace.

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Additional work has been performed by the use of quantitative metallography in the determination of the $\gamma/\gamma + \text{U}_2\text{Ti}$ transus.

A topical report is being prepared.

Vapor Deposition of Molybdenum Coatings
on Stainless Steel Tubing

C. F. Powell, D. M. Rosenbaum, R. B. Palmer,
and I. E. Campbell

Development of processes for vapor depositing molybdenum and niobium on the inside and outside of small-diameter Type 304 stainless steel tubing for ANL is continuing.

In prior work, adherent uniform coatings were applied to the outside of 20-in. lengths of small-bore tubing, but uniform coatings could not be obtained over the inside surface of tubing of this length.

Work during June was devoted to further tests with the moving-temperature-gradient coater in depositing niobium on the inside and outside of the small-bore tubing.

Operation of the moving-temperature-gradient coater was described in BMI-1189. This coater was designed to coat the inside and outside surfaces of the small-bore tubing simultaneously. Several specimens were coated with niobium in this unit, at a deposition temperature of 850 C. These coatings, black in some areas and slightly powdery in others, were poorly adherent and nonuniform. At a deposition temperature of 1000 C, the coating was bright and adherent. The coating varied in thickness from about 1 mil at one end of the specimen to 0.1 to 0.2 mil at the other end, indicating a large variation in the NbCl_5 vaporization rate due to insufficient capacity in the NbCl_5 vaporizer.

Only one run was completed in the moving-temperature-gradient coater at 1000 C before part of the tapped furnace winding burned out. The tapped furnace section has been rebuilt, using heavier resistance wire, and the vaporizer section of the coating unit has been enlarged to hold more NbCl_5 . Additional specimens of the small-bore tubing will be coated with niobium in the moving-temperature-gradient coater during the coming month.

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I-1

I. PHYSICAL METALLURGY

F. A. Rough

Programs sponsored by the AEC Division of Research in the general area of metallurgy and ceramics are reported in this section.

The determination of physical properties of uranium compounds is presently concerned with UBe_{13} , uranium silicides, UC, and UN. The determinations of properties are in various stages of progress.

The study of the tensile transition in alpha uranium is being concluded, and a topical report is being prepared.

The study of the constitution of ternary uranium alloys is concerned with portions of the systems uranium-molybdenum-zirconium and uranium-zirconium-silicon. The evaluation of the former is in final stages, while only preliminary results are available for the system uranium-zirconium-silicon.

The study of bonding fundamentals is continuing, but no new data are available.

A study of the system niobium-hydrogen includes both kinetics measurements and the accumulation of further equilibrium data.

PROPERTIES OF URANIUM COMPOUNDSPreparation, Fabrication, and Physical Properties

A. B. Tripler, Jr., and M. J. Snyder

The preparation of UBe_{13} by induction melting of a stoichiometric mixture of UH_3 and beryllium powder under an argon atmosphere was reported in BMI-1189. Due to accidental overheating of a part of the specimen, two portions were recovered, a melted portion and a sintered portion. Metallographic examination showed the melted portion to be mostly a single phase of UBe_{13} with a small amount of an unidentified second phase along the grain boundaries. UBe_{13} was the only phase identified by X-ray diffraction. The bulk density of the melted portion was 107 per cent of theoretical. Two unidentified phases, in addition to the predominant UBe_{13} , were observed metallographically in the sintered portion of the specimen. X-ray diffraction identified the UBe_{13} and detected a second phase which might have been uranium carbide, oxide, or a mixture of the two. The bulk density of the sintered portion was 89 per cent of theoretical. Next month, attempts will be made to eliminate the extraneous phases in the UBe_{13} preparations, and samples will be prepared for heat-of-formation measurements and gas reactions.

I-2

Sintered specimens of U_3Si_2 , USi , USi_2 , and USi_3 were prepared for tinning experiments. Tinned specimens are needed for thermal-conductivity measurements. Electrotinning experiments are under way but, thus far, adherent tin coatings have not been achieved. Attempts to electroplate the silicides (and specimens of other compounds as they become available) will continue. The so-called "loaded-wheel" technique will also be tried for tinning. Essentially, it consists of "loading" an abrasive wheel with tin (other metals may be used) and then abrading the surface to be coated with the wheel. Oxide films or scales are removed and a layer of tin is deposited on the cleaned surface.

An arc-melt of UC_2 was made, powdered, and analysed. Fabrication experiments will be undertaken and a sample will be submitted for heat-of-formation studies.

To speed up the preparation of bulk specimens, a method is being considered whereby arc melts can be cast directly into a desired shape. The crucible material must not react with the uranium compound at its melting point. Thermodynamic calculations show that UO_2 and ZrO_2 are good prospects for the crucible material. Graphite may be used for some of the compounds.

Work on UB_2 and UB_4 will be renewed during July.

Reaction With Gases

W. M. Albrecht and M. W. Mallett

Fundamental studies are being made of certain uranium compounds to determine their reactions with several of the common gases.

The reaction rates of USi , USi_2 , USi_3 , U_3Si_2 , and UAl_2 with oxygen and nitrogen in the range 150 to 600 C were compared with the published information on the reaction of uranium with the gases.

The rate of reaction of oxygen with uranium* increases rapidly with time, indicating that there is no protective film formed. In contrast, the rates of oxidation of the uranium silicides, with the exception of USi , initially decrease with time due to the formation of a protective film. The initial rate of oxidation of the uranium was lower than that of any of the uranium compounds. However, the rate increased so rapidly that it surpassed the oxidation rate of the compounds (except USi) in about 30 min.

The reaction of uranium with nitrogen** follows a parabolic law (formation of a protective film) which is the same as that found for the uranium silicides. However, the rate of reaction is much slower for uranium than for any of the uranium silicides.

A topical report covering this work is being prepared.

*Cubicciotti, Daniel, JACS, 74, 1079 (1952).

**Mallett, M. W., and Gerds, A. F., J. Electrochem. Soc., 102, 292 (1955).

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Heat-of-Formation Measurements

J. W. Droege, A. W. Lemmon, Jr., and R. B. Filbert, Jr.

As a part of the experimental study of the fundamental properties of refractory uranium compounds, the heats of formation are being investigated. If the heats of combustion of a compound and of its constituent elements are known, the heat of formation may be found. Attempts are in progress to measure these heats of combustion by means of the oxygen-bomb calorimeter. The use of the sodium peroxide-fusion calorimeter is also being investigated, as an alternate technique. So far in the program, the emphasis has been placed on the combustion of USi_2 , but work has now been started on UC and UN.

Since for USi_2 the peroxide calorimeter seems to show the greater promise, experiments have been conducted to determine the most satisfactory amounts of USi_2 and combustion aid, benzoic acid, to use in the combustion. Determinations have also been made on benzoic acid alone.

Several combustions of UC and UN have been carried out in the oxygen bomb. The product in each case was a fused black mass apparently close to the composition U_3O_8 . Analysis of the products is not yet complete, however. Since there is some evidence that these compounds would be slightly oxidized if exposed to the high-pressure oxygen in the bomb, a method is required for protecting the sample until ignited. Small envelopes of Mylar which can be filled and sealed in a protective atmosphere have been found to be satisfactory.

Determinations have been made of the heat of combustion of Mylar and of the cotton thread used for a fuse.

In addition to continuing these experiments with USi_2 and UC, the oxygen calorimeter will be calibrated with standard samples of benzoic acid, and the heat liberated in the fuse wire will be determined.

INVESTIGATION OF THE TENSILE TRANSITION
IN ALPHA URANIUM

L. L. Marsh and G. K. Manning

The effect of grain size and strain rate on the tensile properties and the metallography of the plastic deformation of uranium are being studied over the temperature range defining the tensile transition.

This program is being brought to a close. Data from tensile tests at various temperatures and strain rates are being analyzed. A topical report summarizing the results of this study is being prepared.

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CONSTITUTION OF URANIUM ALLOYSUranium-Base Ternary Alloys

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

Determination of the phase relationships between the delta phases of the uranium-zirconium and uranium-molybdenum binary systems is proceeding.

High-temperature X-ray diffraction studies are nearly completed. Results thus far show no indication of the questionable transition phases that were present in heat-treated and quenched specimens. The high-temperature X-ray investigation also shows that, on cooling, decomposition of the gamma phase in alloys of intermediate composition is very sluggish. As reported earlier, the gamma phase of the uranium-zirconium system is stabilized to about 450 C by molybdenum additions.

A study of the phase relationships of the uranium-rich corner of the uranium-zirconium-silicon ternary system is being conducted. Preliminary X-ray data indicate that Zr_3Si_2 and Zr_2Si are in equilibrium with gamma uranium, and that U_3Si and U_3Si_2 are in equilibrium with Zr_3Si_2 . Further investigation is progressing by means of metallographic and X-ray diffraction examinations.

STUDY OF BONDING FUNDAMENTALSJ. B. Melehan, F. C. Holden, H. R. Ogden,
and R. I. Jaffee

Efforts in the month of June were directed to preparation of a report which will summarize the research of the past year, and preparation of equipment and materials for further research.

NIOBIUM-GAS REACTIONS

W. M. Albrecht, W. D. Goode, and F. W. Boulger

A fundamental study is being made to determine the kinetics and mechanisms of the reaction of hydrogen with niobium.

An equilibrium study of the temperature-pressure-composition diagram of the niobium-hydrogen system has been completed. The data were obtained in the ranges 100 to 900 C, 10 to 1000 mm of mercury, and atomic ratios of hydrogen to niobium of 0.01 to 0.85. A topical report summarizing these data is being prepared.

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The rate study of the reaction to produce a solid solution having a composition with a hydrogen-to-niobium atomic ratio of 0.05 was continued at temperatures of 700 and 800 C. At these temperatures the reaction rates were difficult to reproduce. Rates varied by about 50 per cent. Before further rate studies can be made, the cause of such variances must be determined so that reproducible data can be obtained.

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J. CORROSION PROBLEMS ASSOCIATED WITH THE
RECOVERY OF SPENT REACTOR FUEL ELEMENTSC. L. Peterson, P. D. Miller, E. L. White, W. D. Beasley,
G. L. Ericson, and R. S. Peoples

The study of the corrosion problems connected with various recovery processes for spent reactor fuel elements was continued during June.

Tests simulating the cyclic conditions of the Zircex hydrochlorination-acid dissolution are under way. Favorable results are being obtained with both Type S-816 and Haynes 25 alloys. Over twenty cycles have been completed.

Titanium, Type S-816, and Haynes 21 are still behaving well in the Darex dissolution tests.

The Zircex Process

In the Zircex process, uranium will be recovered from spent reactor fuel elements by solvent extraction of the nitric acid solution of the residue remaining after hydrochlorination of the fuel elements. Hydrochlorination is used to volatilize away any zirconium cladding or alloy.

The evaluation of candidate materials of construction for both the hydrochlorinator and the chloride stripper has been continued during June.

The cyclic tests, which are being used to examine possible materials for the construction of a combined hydrochlorinator-dissolver vessel, have been run for more than 20 cycles. The specimens from these tests have been examined following the 15th and 20th cycle. The most corrosion-resistant material which has been found for any of the three acid dissolution solutions is the Type S-816 heat-resistant cobalt-base alloy. Good results are also being obtained with Haynes 25, another cobalt-base alloy. The other materials under test, such as Carpenter 20, Haynes 21, Type S-590, and Type 304 ELC stainless steel, have already shown such high corrosion rates that they are no longer being seriously considered for this use.

Currently, studies are under way to determine whether the hydrochlorination or dissolution part of the cycle contributes more to the corrosion of the various specimens. The results thus far indicate that either may contribute more, depending upon the particular material under test. Certainly, the cyclic repetition of these two conditions seems to produce more corrosion than prolonged exposure in either condition alone.

The specimens which have been exposed to boiling 5.0 M HNO₃ Zircex elimination-test solutions containing 500 ppm chloride have now been exposed for over 16 weeks. The corrosion rates continue to remain fairly constant at values less than 1 mil per month, as reported in BMI-1176.

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Tests in simulated chloride-stripper solutions have been running for over 16 weeks and are being continued. Both welded and unwelded titanium specimens are exposed.

The Darex Process

In the Darex process, fuel elements containing stainless steel are dissolved by a dilute solution of nitric and hydrochloric acids; the chlorides are stripped by concentrated nitric acid and the uranium is removed from the resulting nitric acid solution by solvent extraction.

The exposure of specimens of various materials in the presence of dissolving stainless steel has been continued. A total of 1846 hr of exposure have been completed in the continuous dissolver and 1659 hr in the batchwise dissolver. To date, specimens of titanium, welded titanium, and tantalum have shown no attack. There has been a slight corrosion of specimens of Type S-816 and Haynes 21, but this is less than 1 mil per month.

Welded and unwelded titanium specimens have been exposed for over 16 weeks to simulated chloride-stripper solutions. These tests are being continued.

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K. EVALUATION OF A REFLECTOR-CONTROLLED
HETEROGENEOUS BOILING REACTOR

J. W. Chastain

This program is investigating the use of a reflector scheme control for long-time unmanned operation of a heterogeneous boiling-water reactor. In this method of control the annular water reflector is connected to a pressurized surge tank and the reactor core. If the power demand is increased the steam pressure above the core drops, permitting water to be forced into the reflector from the surge tank. The increased reflector height increases the reactivity of the core and, hence, the power. For a decrease in power, the process takes place in reverse. A critical-assembly experiment is being run to give information on the reactivity worth of the reflector and the formation of voids with power. A hydraulic mock-up of the system driven by a reactor simulator is being run to investigate the dynamics of the system.

During June, the critical assembly was operated in the hot (417 F) non-power-producing condition. Some instabilities in the hydraulic system have developed and are being investigated.

In July, the boiling (power producing) case will be started on the critical-assembly experiments. These data will be supplied to the simulator.

Critical Experiment

W. C. Ballowe, D. A. McPherson, R. G. Wilson,
F. J. Jankowski, D. A. Dingee, and J. W. Chastain

The experimental results obtained in this experiment will be used to evaluate the reflector system of control so that the feasibility of such a system in practice may be determined. The results are being used in the reactor simulator to provide long- and short-term transient-behavior data.

The effort during the month of June was twofold. Experimental work was done on the hot nonboiling case (moderator and axial and radial reflector voided to have nuclear properties equivalent to water at 417 F and 300 psi). This is now nearing completion. Effort was also directed toward the preferential voiding of the moderator and upper-axial-reflector plastic strips for the power-producing nonhomogeneous boiling case. These materials should be ready for loading into the assembly in early July.

The experiments on the boiling case are expected to start during July.

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Simulator Studies

B. B. Gordon and J. J. Stone

During June the hydraulic system mock-up was operated, using stainless steel wool for damping. This proved to be unsatisfactory because, after a short period of operation, the stainless steel wool packed together and effectively constricted the pipe. All runs made under these conditions were unstable for positive changes in reactivity or power demand.

The system was operated using a section of 1-in. pipe in the 2-in. line between the simulated reflector tank and the surge tank. This system also was unstable for a positive disturbance in power demand.

During the next period, a 2-in. pipe containing several 90-deg elbows will be used in an attempt to produce the damping necessary for optimum operation.

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L. STUDIES OF SODIUM-TANTALUM COMPATIBILITY
AT ELEVATED TEMPERATURES

J. H. Stang

Research is being conducted for Los Alamos Scientific Laboratory to obtain information pertinent to the tantalum-sodium materials combination that is to be present in LAMPRE I. This is a fast reactor system which features an all-tantalum tube-to-header core configuration; the coolant is high-temperature sodium.

Three 4-week flow-loop experiments to evaluate the resistance of tantalum specimens to attack by 1200 F peak-temperature sodium were in progress at the end of June. A few analytical data from prior loop experiments were obtained during the month which indicated the following two interesting points:

- (1) The amount of tantalum in sodium does not increase during a corrosion run despite the fact that the exposed specimens display pronounced weight losses. To date, there is no evidence that cold-zone mass transfer occurs.
- (2) Specimens fabricated from arc-cast material seem to display a higher weight loss and a greater reduction in oxygen content after exposure than samples fabricated from sintered material.

Creep testing of annealed and thermally degassed tantalum sheet from sintered material at 1200 F was continued. The annealed material has a greater strength than the thermally degassed material in a ratio varying from almost 2 to 1 at lower creep rates (0.0001 per cent per hr) to about 3 to 1 at higher rates, above 0.1 per cent per hr.

During June, several miniature tantalum tube-to-header assemblies were fabricated successfully. Subsequent helium leak-detection tests showed that these assemblies were leakfree; metallographic examinations of the welds involved indicated that they were relatively free from porosity and generally sound.

A recent analysis of a sodium sample made by gettering the sample at 760 C with titanium foil (later analyzed by vacuum fusion) showed a higher oxygen content (52 ppm) than that found by the amalgamation method (about 30 ppm). This result indicates the possibility of contamination from the walls of the test bomb, a factor which must be scrutinized carefully in the over-all appraisal of the gettering technique.

Tantalum-Sodium Compatibility Studies

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

The behavior of tantalum exposed to sodium in polythermal forced-circulation loops is being investigated. The peak sodium temperature is 1200 F, and the nominal fluid-temperature cycle is 500 F. The loops are constructed of Type 316 stainless steel and contain removable tantalum specimens in the furnace zone. Each loop has built in

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sodium-purification facilities consisting of cold-trapping and gettering (employing zirconium foil) circuits.

Work during June was along two lines: (1) the conduction of three 4-week corrosion experiments and of one loop experiment in which the tantalum specimens are being exposed for subsequent mechanical-property determinations; and (2) the collection of additional analytical data for three tests completed earlier and reported in detail in BMI-1189. One of the current corrosion runs is being made with the sodium containing about 20 ppm oxygen, and the other two, with the fluid at the >10-ppm oxygen level. The mechanical-property specimens are also being exposed in a very-low-oxygen environment, estimated to be in the 2 to 3 ppm range as a result of an exceptionally rigorous cold-trapping and gettering schedule. The objective here is to obtain specimens which are quite low in oxygen (30 ppm and, perhaps, lower) but which have not experienced the grain growth that accompanies thermal degassing.

Recent information pertinent to prior corrosion runs (8-, 11-, and 15-day exposures with oxygen levels of >10 ppm and 80 ppm) has indicated the following points of particular interest:

- (1) The tantalum level in sodium, indicated by spectrographic analyses to be in the fractional ppm range, did not change in the case of one loop exposure. Some change was expected here since (a) the tantalum specimens lost up to 0.2 per cent of their original weight during the experiments (without intergranular attack), and (b) evidence of cold-zone mass transfer has not been found, although the inspection of all loop components is not quite complete. Thus, at present, there is no indication of the final disposition of the metal removed from the test material.
- (2) As indicated in BMI-1189, two specimens fabricated from arc-cast material exhibited somewhat higher weight losses than neighboring samples fabricated from sintered sheet. A posttest vacuum-fusion analysis of one of these specimens (exposed in sodium containing <10 ppm of oxygen) indicated that it also lost more oxygen during its exposure than did an adjacent sample of sintered stock (a decrease from 113 ppm oxygen to 16 ppm for the former compared with a decrease from 118 ppm to 64 ppm for the latter). It is somewhat surprising that arc-cast material would exhibit this substantially different behavior than sintered material. Further experiments with arc-cast material are now contemplated and should yield additional information concerning this behavior.

High-Temperature Mechanical Properties of Tantalum

G. F. Huber, J. G. Dunleavy, C. J. Slunder, and A. M. Hall

Static creep tests are being conducted in helium-atmosphere (4 psi) furnaces to obtain data for tantalum specimens at 1200 F. Specimens tested to date have been fabricated from sintered sheet (original oxygen content about 210 ppm) which is either

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annealed or thermally degassed. The former treatment yields a fairly fine-grained product which probably has an oxygen content somewhat lower than the original (this point is now being checked); the latter yields a coarse-grained product which contains >5 ppm of oxygen (nitrogen and hydrogen are also driven off during the degassing). Recent experiments have placed emphasis on the low-oxygen material, inasmuch as it is expected that oxygen (as well as other gases) will be removed from the tantalum in the core of LAMPRE I by a strong gettering action of the fuel mixture. In the reactor, however, temperature-dependent grain growth, such as takes place during high-temperature degassing, should not occur.

Table L-1 presents a summary of creep data accumulated thus far in this research. It may be seen that the annealed material is considerably stronger than the thermally degassed material, as might be expected. For example, to obtain a practical minimum creep rate, such as 0.00001 per cent per hr, the stress allowable would be about 10,000 psi in the case of annealed material, but would be only 6,000 psi for thermally degassed material. It is of interest to note that, at higher loads and, consequently, higher creep rates, this spread in creep strength of the two materials increases.

It is expected that, during July, creep tests will be started on the following two kinds of material:

- (1) Annealed sheet 0.030 in. thick fabricated from arc-cast material.
- (2) Material exposed to 1200 F peak-temperature sodium to obtain low-oxygen samples, as discussed in the previous subsection.

Weldability of Tantalum for High-Temperature Systems

S. M. Silverstein, R. P. Sopher, and P. J. Rieppel

The objective of this study is the development of techniques (using the inert-gas tungsten-electrode arc-welding process) for welding of an all-tantalum tube-to-header can assembly. Work during June consisted of the final fabrication and testing of miniature tantalum tube-to-header assemblies. The materials used were 20- and 30-mil virgin arc-cast tantalum sheet for the can walls and tube headers, respectively, and 15-mil-wall virgin arc-cast tantalum tubing. As mentioned in BMI-1189, some difficulty was encountered in making the header-to-can joint because of warpage and consequent burn-through. However, in recent fabrication work, this difficulty has been alleviated by the use of a colletting system.

A tantalum tube was joined to the side wall of each tube-to-header assembly to facilitate helium leak testing of the welded joints. The subsequent leak-detection tests showed that the assemblies were leakfree (within the limit of detection of the helium mass spectrometer used). Metallographic studies made on the tube-to-header assemblies indicated that the tantalum welds were sound and relatively free of the porosity which is peculiar to some lots of tantalum. In addition, these studies showed that good filleting was obtained between the tantalum tubes and header wall. This should not only increase the strength of the joint but should also reduce the possibility of crevice corrosion in the heat exchanger.

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TABLE L-1. CREEP STRESS-RUPTURE DATA FOR TANTALUM SHEET FABRICATED FROM SINTERED STOCK AND TESTED AT 1200 F IN HELIUM

Stress, psi	Time in Progress, hr	Rupture Life, hr	Time, hr, to Reach Indicated Percentage Deformation			Total Deformation, per cent	Minimum Creep Rate, per cent per hr
			1.0	2.0	5.0		
			<u>Annealed Material^(a)</u>				
32,000	To failure	3.6	--	--	--	88.5	~10
28,000	To failure	16.0	--	--	0.1	65.5	~1.1
24,000	To failure	460.8	--	0.2	3.0	73.1	0.06
20,000	475 ^(b)	--	2.2	4.0	69	9.5 ^(b)	0.01
15,000	1011 ^(b)	--	46.5	775.0	--	2.2 ^(b)	0.000035
12,500	Test just started						
10,000	325	--	--	--	--	0.05	~0.00001
<u>Thermally Degassed Material^(c)</u>							
20,000	Failed on loading						
10,000	To failure	21.1	--	0.9	2.2	40.1	0.023
8,000	540	--	2.6	27.0	256.0	9.6	~0.01
6,000	800	--	--	--	--	0.525	~0.00001

(a) Annealed in vacuum at 2650 F for 8 min; recrystallization occurred.

(b) Test discontinued at the time indicated.

(c) Heat treated in vacuum at 4500 to 4800 F for 5 hr; average grain diameter over 1 mm.

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L-5 and L-6

As mentioned in previous reports, welding studies on sintered and arc-cast tantalum sheet have indicated that the higher the oxygen content of tantalum the more weldable the material. Future work will be performed in order to determine the validity of this postulation. These studies will consist of ingassing an unweldable lot of tantalum (31 ppm oxygen) to higher levels of oxygen. Trial welds made at each level will be given thorough metallurgical inspections to ascertain their quality.

Determination of Oxygen in Sodium

P. H. Goble, W. M. Albrecht, and M. W. Mallett

A study is being made to develop a new and more sensitive analytical method for the determination of oxygen in sodium. The method consists of gettering oxygen from the sodium by a metal which, in turn, is analyzed by vacuum-fusion techniques.

In BMI-1189, an experiment was reported in which the analytical results were incomplete. In that experiment, 10-mil sheet titanium was heated in contact with filtered sodium for 2-1/2 hr at 690 C. This sodium had been analyzed by the amalgamation method and found to contain 40 ± 10 ppm oxygen. In the gettering experiment, the titanium picked up 21 ppm oxygen from the sodium. However, it is believed that this low value can be attributed to an incomplete vacuum-fusion analysis of the titanium sample.

Further experiments were made during June using a new supply of sodium. A single analysis of this sodium by the amalgamation method indicated that its oxygen level was 30 ± 10 ppm. It should be pointed out, however, that several additional analyses by the amalgamation method will be made to obtain a more reliable base-level value. Using this sodium, a gettering run was made for 2 hr at 760 C. To improve its heating characteristics, the bomb was charged with 0.4 atm of argon which is equivalent to about a 1-1/2-atm pressure at the temperature of the run (normally, the gettering reactions are carried out in vacuum). In this experiment, the titanium picked up 52 ppm oxygen from the sodium. Although this value may represent the actual oxygen concentration in the sodium, it was slightly higher than anticipated. There is a possibility of oxygen contamination from the argon or from the walls of the bomb at the high temperature used for this run. It is planned to check these possible sources of contamination very carefully.

M-1

M. DEVELOPMENTAL STUDIES FOR THE PWR

R. W. Dayton

Preparations for further flow studies on the PWR model were completed, and testing will begin in July.

Studies of a production test for weld porosity and uranium segregation are in the apparatus stage.

The literature survey of construction materials for in-pile pressurized water loops is progressing on schedule, and is expected to be completed during July.

Reactor Flow Studies

L. J. Flanigan and H. R. Hazard

Air-flow studies in a quarter-scale model of the PWR are being conducted to determine the effects of lower-plenum design on mixing and flow distribution in Core 2. Previously reported work includes design of an aluminum casting for a core extension, preparation of the model for flow studies, and improvement of gaseous-tracer technique for studying mixing.

In June, the aluminum core extension was machined and installed in the model. The perforations in the new flow baffle were designed and the baffle was machined and installed in the model.

Twenty additional static-pressure taps were installed in the lower dome of the model for a detailed study of the effect of the extended core on pressure distribution. Eight static-pressure taps, manifolded together by a recessed piezometer ring, were installed in the downstream side of the flow baffle. These will be used in measuring flow-baffle pressure loss.

In July, assembly of the model will be completed and a shakedown test will be run, and the distribution of flow in the model core and mixing in the lower plenum will be studied in detail.

X-Ray Photometric Examination of Fuel Elements

J. B. Schroeder and C. M. Schwartz

A nondestructive production test for weld porosity and uranium segregation is being developed. Shielding and collimators are being made of Mallory 1000 alloy (tungsten-6 w/o copper-4 w/o nitrogen) and should be completed in about 60 days. Procurement proceedings are under way for the purchase of the required X-ray generator.

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M-2

High-Strength Materials for
Pressurized-Water In-Pile Tubes

F. H. Lyon and H. J. Grover

The objective of this program is to collect and evaluate pertinent physical-property information on materials suitable for in-pile pressurized-water tubes. The survey is aimed at supplying the information for an experimental program for the construction and testing of a prototype tube.

BMI-1189 listed the materials being considered under four general classifications: alloy steels, chromium steels, precipitation-hardening stainless steels, and superalloys. During June, a large quantity of factual information has been collected on the materials in the four classifications. It has been obtained from more than 40 selected references and contains almost all of the information directly available at Battelle.

Most of the pertinent information about alloy steels has been located in the literature. Data on corrosion-resistance properties, however, have not been found thus far in the survey.

The information on chromium steels is nearly complete in regard to the stainless steels, but for Croloy 16-1 nothing was found. The corrosion-resistance properties of all the stainless steels, and the creep strength, rupture strength and notch sensitivity of AISI 410 and 414 may require experimental determination.

For AM-350, a precipitation-hardening stainless steel, the largest gaps in available information involve the welding and corrosion-resistance characteristics, creep strength, and thermal conductivity. No information has been found for AM-355, although its properties should be nearly those of AM-350.

In the group designated superalloys, the information on Inconel X is most nearly complete, lacking only the creep and rupture strengths. No information has been found on W-545, and, for A-286 and Discaloy, the largest gaps are in welding and corrosion-resistance information.

Visits to producers of the various materials are in progress to obtain the missing information and to determine the availability. It is expected that the survey will be completed and a topical report prepared before the end of the next report period.

RWD:CRT/all

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