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by

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January 1, 1956

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A. DEVELOPMENT OF MATERIALS FOR REACTORS

J. R. Keeler

In this program the properties of dilute uranium alloys are being investigated. The mechanical properties of cold-worked zirconium and Zircaloy 2 at temperatures up to 500 C are being determined, and work on the cladding of uranium with zirconium by hydrostatic pressing has been started.

The data obtained by several methods on the solid solubility of uranium in thorium are not in good agreement, and additional data are being obtained.

The study of radiation deformation in graphites at temperatures of 300 C and slightly above has been resumed. Four types of graphite are being prepared for irradiation at Hanford in order to study the factors affecting deformation.

Uranium Alloys for Fuel Elements

H. A. Saller, F. A. Rough, and W. Chubb

A group of dilute uranium alloys has been examined for heat-treatment characteristics, physical, and mechanical properties. The alloys examined were arc-melted uranium, induction-melted uranium, uranium-0.35 a/o chromium, uranium-1.5 a/o silicon, and uranium-0.5 a/o titanium. During the last month, samples of these alloys which had been quenched from 730 C and from 900 C were annealed for 1 hr at 600 C. This treatment produced a marked reduction in hardness, but caused no appreciable change in grain size. The grains became equiaxed, and measurements indicated a small decrease in grain size. As water quenched from 730 C, arc-melted uranium had a hardness of 240 DPH; after annealing at 600 C, the same specimen had a hardness of 204 DPH. This decrease in hardness is interpreted to mean that quenching and transformation strains have been relieved and that the metallurgical stability of the alloys is improved by this treatment. Heat treatments above 730 C merely increase the grain size and hardness of these alloys and do not appear to be promising. No further treatments are planned for these alloys.

Eight biscuit uranium-base alloys containing aluminum, silicon, and zirconium have been prepared by arc melting. The ingots which weighed 100 g were successfully hot rolled at 620 C to 0.10-in. sheet. Hardness data obtained during the fabrication of these alloys are shown in Table A-1.

TABLE A-1. HARDNESS DATA

Nominal Composition (Balance Uranium), a/o	Cast Hardness, Rockwell A	Hot-Rolled Hardness	
		Rockwell A	DPH
0.5 Aluminum	53	59	270
1.0 Aluminum	56	64	287
2.0 Aluminum	62	66	322
0.5 Silicon	61	63	287
1.0 Silicon	63	63	314
1.0 Zirconium	67	63	309
2.0 Zirconium	61	65	297
4.0 Zirconium	67	67	345
Blank	--	54	194

The hot-rolled microstructures of the alloys show evidences of much distortion and orientation of grains as a result of rolling at 620 C. The increase in hardness as a result of rolling also indicates that residual stresses have been left by the rolling operation. The effects of heat treatments upon these alloys will be examined in the near future.

The Constitution of Thorium-Uranium Alloys

H. A. Saller, F. A. Rough, A. A. Bauer, and J. R. Lulay

A study of the thorium-uranium system is under way for Hanford to determine the solubility limit of the thorium-rich solid solution.

Data have now been obtained on the solid solubility of uranium in thorium by hardness tests, metallography, and X-ray diffraction. In general, the data obtained by different methods are not in agreement, but indicate solution of 3 w/o uranium or more in thorium at 710 C and below. There is some indication that the solubility increases at higher temperatures.

Further studies will be designed to improve upon the data in order to obtain more accurate solubility values.

Mechanical Properties of Zirconium and Zircaloy 2

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

A comparison of the mechanical properties of cold-worked zirconium and Zircaloy 2 with those of the annealed material is being made for several

temperatures up to 500 C. A report summarizing the data obtained in this study is being prepared.

Elevated-Temperature Tensile Properties

Four additional tensile tests have been conducted on cold-worked zirconium and Zircaloy 2 after annealing 500 hr at 425 C and 500 C. The rate of straining was 0.02 in. per min (cross head speed). The results are summarized in Table A-2.

TABLE A-2. TENSILE DATA FOR ZIRCONIUM AND ZIRCALOY 2

Properties	Temperature (Argon Atmosphere), C	
	425	500
Cold-Worked Zirconium		
0.2 Per Cent Yield Strength, psi	13,900	5,300
Tensile Strength, psi	19,200	10,500
Elongation, per cent	37	93
Cold-Worked Zircaloy 2		
0.2 Per Cent Yield Strength, psi	34,200	12,900
Tensile Strength, psi	40,900	18,500
Elongation, per cent	21	44.5

It appears that 500 hr at 425 C is not sufficient to completely recrystallize the cold-worked Zircaloy 2, although 500 hr at 500 C was more than sufficient. The knowledge of the softening kinetics already gained at 425 and 500 C for cold-worked zirconium and Zircaloy 2 at 500 C should establish a family of curves at these two temperatures. These isothermal curves, normally sigmoidal in shape can be obtained by plotting the fractional property change as a function of time at temperature, using the mechanical-properties data for the annealed material as a basis. Such an analysis should permit a calculation of the softening rate at 425 C for Zircaloy 2.

Creep Properties

Several additional creep and stress-rupture tests have been completed on transverse sheet specimens of both annealed and cold-worked Zircaloy 2. These results are shown in Table A-3.

TABLE A-3. CREEP PROPERTIES OF ANNEALED AND COLD-WORKED ZIRCALOY 2

Temperature, C	Stress, psi	Elongation, per cent					Time to Failure, hr	Test Time, hr	Creep Rate, per cent per hr
		Load On	100 Hr	500 Hr	1000 Hr	Total			
Annealed Transverse Sheet									
150	30,000	6.80	26.8	--	--	37.6	117.5	117.5	0.30
425	17,500	--	--	--	--	32.0	49.6	49.6	0.194
Cold-Worked Transverse Sheet									
150	54,000	0.46	0.60	0.62	0.63	0.63	--	1003	0.00064
150	67,000	0.71	1.30	--	--	7.2	313.2	313.2	0.0009
150	70,000	0.80	--	--	--	13.2	27.6	27.6	0.0225
150	74,000	1.00	--	--	--	10.4	0.35	0.35	--
230	65,000	1.00	--	--	--	13.2	0.25	0.25	--
230	59,000	0.90	--	--	--	7.6	45.0	45.0	0.010
230	56,000	0.60	0.95	1.12	--	1.21	876.5	876.5	0.0002
345	45,000	0.60	--	--	--	14.0	23.2	23.2	0.062
425	24,000	0.30	1.60	4.70	--	24.8	867.0	876.0	0.0071

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A comparison of these additional creep data from cold-worked material and previously reported data on annealed material indicates the following:

(1) The increased rupture strength resulting from cold working persists at 150, 230, and 345 C for times up to 1000 hr.

(2) Although short-time tensile data indicate only a slight drop in the strength of cold-worked Zircaloy 2 after 500 hr at 425 C, the data on rupture strength indicate that some softening occurs after 100 hr at 425 C. Similarly, at 500 C the short-time tensile data indicated little softening after a 10-hr anneal, but the rupture data indicate significant softening under stress at 500 C in 10 hr.

Testing will continue on these same materials on longitudinal specimens to compare the creep properties of longitudinal and transverse specimens.

Tube Bursting

No additional burst tests have been made during the month. Additional tubing was received and several tubes are being prepared to investigate the effect of shot peening on the welded zirconium tubing.

Zirconium Cladding

H. A. Saller, J. R. Keeler, and L. J. Cuddy

The feasibility of cladding uranium slugs in zirconium cans by heating under hydrostatic pressure is being investigated.

Small-diameter thin-walled zirconium tubing was available and is being used for initial tests. Swaged uranium rod was pickled and then welded in the zirconium tubes under vacuum. One assembly was held 24 hr at 600 C and one at 700 C under a 2500-psi helium pressure. Peel tests on the sample heated at 600 C indicated slight bonding, but the bond was weak. The sample heated at 700 C had oxidized on the surface, indicating that the seal was defective.

Additional tests at 700 and at 800 C are in progress.

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Radiation Damage to Graphite

L. D. Loch, A. E. Austin, G. B. Engle, and M. J. Snyder

The study of irradiation deformation in graphites at temperatures of 300 C and slightly above has been resumed. Physical contractions have been observed in present pile graphite (CSF) and in the four Battelle-prepared experimental graphites that have been irradiated at these elevated temperatures. Such contractions might cause serious difficulties particularly if the effect does not saturate.

The following possible causes of the contraction are suggested by previous research (BMI-1042) on low-temperature radiation stability:

(1) Radiation annealing of defects originally responsible for the presence of micropores, allowing crystals to come closer together

(2) Crystal growth across crystal boundaries, eliminating micropores and/or disordered carbon

(3) Contraction of the graphite layers.

A research program to study these mechanisms and to look for new factors that may be important has been formulated. Four graphites were selected which represent a wide range of crystallite size and microporosity. These are graphites made from resin coke, carbon black, pitch coke, and skeletal graphite.

During the month, specimens of graphitized carbon black and skeletal graphites were shipped to Hanford for high-temperature irradiation. Preparation of resin-coke graphites was completed. X-ray diffraction measurements were made on irradiated samples available from previous work.

In future work, crystal parameters, helium density, and macropore structure will be measured on unirradiated specimens for comparison with similar measurements on specimens after irradiation at elevated temperatures.

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B. DEVELOPMENTS FOR ALUMINUM-CLAD FUEL ELEMENTS

D. C. Martin

Work has stopped on all phases of the aluminum-clad-fuel-element development except internal cladding of tubes and extrusion cladding of flat plates. Equipment has been obtained for further work on both of these projects. This equipment is being assembled and experimental work will start early in January.

Preparation of Aluminum-Uranium Alloys

H. A. Saller, R. F. Dickerson, and E. L. Foster, Jr.

The experimental work on this project has been finished. A topical report is being prepared.

Internal Cladding of Tubes

R. J. Fiorentino, D. B. Roach, and C. J. Slunder

The work on this project has been concerned with the preparation of a few uranium tubes and aluminum billets for internal cladding by the tube-expander method. The new 5-roll tube expander has just been received from the manufacturer and the planned experiments to produce one clad 8-in. section of tube will be carried out in January.

Extrusion Cladding of Flat Plates

R. J. Fiorentino, D. B. Roach, and C. J. Slunder

The construction of the auxiliary equipment to be used with the extrusion-cladding assembly has been completed. These include a device for pulling the core during extrusion cladding and a modified gas burner to provide additional heat to the bolster and die of the extrusion assembly.

The new extrusion container, mandrel tip, and other reworked tools have just arrived and are being checked. The entire extrusion assembly will be set up in the laboratory and checked for temperature variations at several

points, dimensional changes due to thermal expansion, fit of the various parts, and other similar details. When these have been determined and found to be satisfactory, the equipment will be assembled in the press for actual extrusion-cladding experiments.

Undercutting Corrosion

W. E. Berry, J. G. Beach, R. J. Carlson,
and R. S. Peoples

Experimental work on this project has been finished. A topical report is being prepared.

C. PLANT ASSISTANCE TO MCW

J. R. Keeler

Factors affecting the production of active UO_3 and UO_2 are being studied in a program of plant assistance to MCW. Work this month was concerned with the relation of crystallite size to the sinter density of UO_2, the effect of removal of residual nitrate from UO_3 on the sinterability of UO_2, the effect of absorbed nitrogen or oxygen on the X-ray diffraction pattern of UO_2, and the reactivity toward hydration of Hanford continuous-process UO_3.

The electrical properties of UO_2 are also being investigated, and several major differences between the electrical conductivity of "active" UO_2 and "ordinary" UO_2 have been observed.

Additional corrosion tests were completed on selected stainless steels in an atmosphere of cracked ammonia containing water and sulfur.

Identification of Uranium Oxides

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

The reactivity of various modifications of UO_3 is being investigated as an aid to the production of active UO_3 and UO_2. Previous studies have shown that the chemical and sintering reactivity of UO_2 is related to the structure type of the UO_3.

Studies this month were concerned with the relation of crystallite size to sinter density, the effect of removal of residual nitrate from UO_3 upon the sinterability of UO_2, the effect of adsorbed nitrogen or oxygen upon the UO_2 X-ray diffraction pattern, and the reactivity toward hydration of Hanford continuous-process UO_3.

Crystallite Size - Sinter Density Study

As shown in Table C-1, various modifications of UO_3 were reduced at different temperatures and then sintered. The crystallite size of the UO_2 powder was compared to the density of the sintered pellet. It was found that, while the sinter density of the UO_2 is quite dependent upon the type of UO_3 from which it was made, the crystallite size of the UO_2 is of very little significance. It appears that UO_2 made from Type H* UO_3 has the highest

* The various types of oxide, H, G, C, and A, represent repeatedly obtained but as yet unidentified oxide X-ray pattern.

TABLE C-1 RELATION BETWEEN CRYSTALLITE SIZE AND SINTER DENSITY OF UO_2

Sample	UO_3 Type	UO_2 Reduction Temperature, C	UO_2 Crystallite Size, A	UO_2 Sinter Density[a], per cent of theoretical
94	MCW UO_3	545	570	69.4
95	MCW UO_3	650	1030	69.4
93	H	482	320	95.2
91	H	545	400	94.7
92	H	593	830	87.7
89	H	650	560	94.5
90	G	482	430	89.3
87	G	593	880	88.4
88	G	704	1970	86.8
16	Amorphous	620	180	90
22	Amorphous	680	250	87

(a) Pressed at 20,000 psi with camphor binder, sintered at 1650 C in hydrogen for 1 hr.

sinter density, regardless of the reduction temperature or crystallite size of the UO_2.

Residual Nitrate of UO_3 Versus Sinter Density of UO_2

A study was made to determine the effect of any nitrate or nitrogen, remaining in UO_3 after decomposition, upon the sinter density of UO_2. MCW UO_3, as received, was heated at various temperatures before and after washing with water. The resulting materials were reduced at 600 C in hydrogen and then sintered at 1650 C. The results are shown in Table C-2. All treatments except firing to U_3O_8 gave better densities than the untreated material, and heating at moderate temperatures (300 C) appears to give a higher density than heating to a higher temperature (450 C).

Effect of Oxygen and Nitrogen Pickup by UO_2

A UO_3 material was prepared to give a reactive UO_2 on exposure to air. Three portions of the UO_3 were reduced to UO_2 in hydrogen. Oxygen, nitrogen, or air was admitted individually to each portion at room temperature. The UO_2 preparations with oxygen or air added gave very diffuse X-ray diffraction patterns, indicating a chemical reaction with the UO_2. The sample to which nitrogen had been added gave a diffraction pattern with very sharp lines and a lattice parameter of 5.4690 A, indicating that the nitrogen was physically adsorbed only. A study is being made of the effect of

annealing these samples at higher temperatures in their respective gases at low pressures.

Reactivity of UO_3 Toward Hydration

UO_3 preparations from Hanford continuous process were hydrated to equilibrium at 30 C in water-saturated air. The results as given in Table C-3 indicate that for UO_3 with 1000 ppm SO_4, the reactivity decreases with increasing temperature of denitration. For samples containing more or less than 1000 ppm sulfur in the UO_3, the reactivity does not appear to change appreciably with different denitration temperatures.

During the next period the effect of the nitrogen content on the sinterability of UO_2 will be investigated.

The Corrosion Resistance of Selected Stainless Alloys

W. J. Braun, F. W. Fink, and R. S. Peoples

Hot cracked ammonia gas is used by Mallinckrodt to reduce UO_3 to UO_2. Water vapor is a product of this reduction. Sulfur, which catalyzes this reaction, is added to the UO_3 feed as sulfuric acid. In the plant, the screw conveyor for the orange oxide is made of Inconel and Hastelloy C. Local hot spots resulting from the exothermic reaction have caused attack, particularly on the Inconel. Bed temperatures are estimated to approach 1700 F.

It is the purpose of this research to evaluate the effects of temperature and sulfur addition on the corrosion resistance of selected alloys exposed in wet hot cracked ammonia gas. Sulfur was found to have little effect on the scaling resistance and physical properties of special alloys which were previously exposed at 1200 F and 1500 F*.

For the present study, ten of the most resistant alloys were exposed at 1700 F. Two test runs were made. One run was sulfur free and the other contained 100 grains of sulfur as sulfurous acid per 100 ft^3 of cracked ammonia. The results of these tests, given in Table C-4, show that scale formation was up to ten times as great after exposure at 1700 F for 100 hr as it was after 500 hr at 1200 F. The data indicate that the presence of sulfur did not result in increased scale formation at 1700 F. Similarly, sulfur did not result in decreased ductility after exposure for 100 hr at 1700 F.

* Results at 1200 F and at 1500 F are reported in BMI-1035, August, 1955, and BMI-1043, September, 1955.

TABLE C-2. EFFECT OF RESIDUAL NITRATE IN UO_3 UPON THE SINTER DENSITY OF UO_2

Sample	Treatment Before Reduction	Phase Before Reduction	UO_2 Sinter Density(a), per cent of theoretical
97	MCW UO_3 as received	C + A	72.5
104	MCW UO_3 heated 300 C	C	80.3
117	MCW UO_3 heated 450 C	C	73.0
116	MCW UO_3 heated 750 C	U_3O_8	70.0
101	MCW UO_3, washed, heated 50 C	A	82.0
100	MCW UO_3, washed, heated 300 C	Amorphous	82.2
113	MCW UO_3, washed, heated 450 C	C	77.6

(a) Pressed at 20,000 psi with camphor binder, sintered at 1650 C in hydrogen for 1 hr.

TABLE C-3. HYDRATION OF HANFORD CONTINUOUS-PROCESS UO_3

Run	Drum	X-Ray Type	Temperature, C		SO_4, ppm	Type After Hydration	Initial Reactivity	Equilibrium Water Content
			Feed	Discharge				
B-17	60	C	290	300	1000	G + A	0.24	10.65
B-18B	76	J	230	250	1000	G + A	0.23	12.83
B-19	84	C	295	335	3000	G + A	0.21	11.00
B-22	105 + 106	C	260	280	3000	G + A	0.31	12.16
B-24	122	C	260	280	1000	G + A	0.19	8.78
B-25	127	C	260	280	0	G + A	0.21	10.66
B-29	173	C	290	310	0	G + A	0.19	11.28
B-30	179	C	450	460	1000	A + C	0.09	2.53

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TABLE C-4. EFFECTS OF TEMPERATURE AND SULFUR ON THE PHYSICAL PROPERTIES OF STAINLESS ALLOYS EXPOSED TO AN ATMOSPHERE OF CRACKED AMMONIA PLUS 40 PER CENT WATER VAPOR

Alloy	Exposure Time, hr	Exposure Temperature, F	Sulfur, grains per 100 ft³ of cracked ammonia	Tensile Data		Elongation, per cent	Hardness, Rockwell B	Weight Gain, mg per in.²
				0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi			
Type 310	0	--	--	35,500	80,800	53	75	--
	100	1700	None	32,100	81,200	51	73	2.5
	100	1700	100	33,500	82,400	46	77	4.8
	500	1200(a)	80	36,600	83,900	47	81	0.6
Type 330	0	--	--	34,300	83,100	45	70	--
	100	1700	None	31,500	82,900	33	75	4.9
	100	1700	100	34,700	84,800	39	78	4.2
	500	1200	80	48,500	93,600	32	87	0.4
Type 347	0	--	--	48,200	92,300	51	84	--
	100	1700	None	36,100	89,400	54	73	3.1
	100	1700	100	36,900	88,000	50	75	2.6
	500	1200	80	49,700	95,700	49	86	0.4
Carpenter 20 plus niobium	0	--	--	53,600	93,800	39	87	--
	100	1700	None	43,200	90,700	39	83	1.7
	100	1700	100	44,400	91,300	36	86	1.5
	500	1200	80	56,900	97,000	39	90	0.5
Hastelloy C, quench annealed	0	--	--	54,000	121,000	55	91	--
	100	1700	None	61,700	87,400	6.0	103	3.2
	100	1700	100	74,200	145,000	0.0	105	2.7
	500	1200	80	68,100	109,000	10	96	0.3
Haynes Alloy 25	0	--	--	81,400	158,000	47	102	--
	100	1700	None	79,700	138,000	40	109	3.3
	100	1700	100	85,800	158,000	9.5	110	1.8
	500	1200	80	103,000	140,000	9.0	108	0.3
Inconel X	0	--	--	45,900	114,000	56	85	--
	100	1700	None	100,000	161,000	27	105	7.7
	100	1700	100	100,000	162,000	28	106	5.2
	500	1200	80	136,500	189,000	16	111	0.7
Illium R	0	--	--	60,300	121,000	40	94	--
	100	1700	None	61,200	135,000	38	88	3.1
	100	1700	100	52,200	117,000	36	89	3.6
	500	1200	80	60,900	121,000	32	94	0.4
Timken Alloy 588	0	--	--	97,800	123,000	20	102	--
	100	1700	None	63,300	102,000	15	99	3.8
	100	1700	100	73,400	92,400	15	101	2.4
Nimonic 80-A	0	--	--	106,000	164,000	29	108	--
	100	1700	None	85,000	139,000	21	102	8.4
	100	1700	100	83,800	144,000	20	101	4.7

(a) See BMI-1035, and BMI-1043 for a complete tabulation of previous results at 1200 and 1500 F.

C-6

Duplicate sets of samples are being exposed at 1700 F for a 500-hr test period. Results of these tests will be included in a future progress report.

The Electrical Properties of UO_2

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

Electrical-conductivity measurements have been made on both sintered and nonsintered specimens of uranium dioxide, which were hydrostatically pressed at 100,000 psi without a binder. A number of major differences between the electrical conductivity of "active" or ball-milled UO_2, which sinters to high densities, and that of "ordinary" MCW UO_2 have been observed. These differences are apparent in the absolute magnitude of the conductivity, the temperature dependence of the conductivity, the thermoelectric power, and the amount of excess oxygen present when other impurities furnish the charge carriers. Some of these differences may be explained by assuming the presence of an n-type impurity, possibly nitrogen, in the "ordinary" UO_2.

The electrical conductivity of MCW uranium dioxide at 27 C increases with the amount of excess oxygen in the lattice of both sintered and nonsintered samples. The magnitude of the electrical conductivity, especially of the nonsintered materials, is dependent on the previous history of the specimen. Large increases in the conductivity at 27 C are observed following a 16-hr vacuum anneal and a subsequent quench from 200 C. No additional changes are noted if the specimen is then annealed at 300 C and quenched. Anneals at higher temperatures followed by quenching did produce additional changes, some of which were large, but the magnitude of these changes was strongly dependent on the amount of excess oxygen. These conductivity changes can be explained on the basis of a diagram of equilibrium phases similar to the one recently proposed by Gronvald. According to such a diagram, much of the excess oxygen could be taken up by a tetragonal second phase ($UO_{2.33}$) in the temperature region below 200 C, then by a U_4O_9 second phase at intermediate temperatures, and finally becoming single phase at still more elevated temperatures. The temperature at which a given specimen becomes single phase depends on both the amount of excess oxygen and the past history of the material with respect to time and temperature.

When uranium dioxide is heated to 1000 to 1200 C in a reducing atmosphere, the density decreases due to the loss of oxygen and the resulting expansion of the lattice. Under these conditions, specimens began sintering (increasing in density) at temperatures above 1200 C. Samples of uranium dioxide which sinter to the highest densities appear to have the smallest amount of excess oxygen present after sintering under the usual conditions

(hydrogen atmosphere at 1600 C or higher). The opposite was observed when uranium dioxide samples were heated under conditions in which excess oxygen could not be lost; i.e., in a sealed Vycor capsule. Sintering began at much lower temperatures (about 800 C) and the largest increases in density were observed for samples containing greater amounts of excess oxygen.

Studies of the electrical properties of UO_2 after anneals at high temperatures and subsequent quenches to room temperature will be continued. Additional observations will be made on the changes in density which occur during the annealing process.

D-1

D. PROCESSING OF FEED MATERIALS

D. L. Keller

Research studies for National Lead of Ohio (NLO) are concerned with improving uranium production metal and production metal-processing methods.

In the salt-bath development program, various mixtures of carbonates, sulfates, chlorides, and halides are being evaluated by measuring the hydrogen picked up by uranium from the bath, by determining the water picked up by the bath from a controlled atmosphere, and by measuring the weight loss of uranium while submerged in the bath. Early results have shown that sulfates severely attack the uranium and that more hydrogen is picked up from the carbonates than from the halides.

The resistance of magnesia and zirconia refractories to molten-fluoride slags was tested at 3000 F after being screened from a group of seven commercial refractories in tests run at 1800 and 2700 F. At the higher temperature, magnesia brick continues to show some promise, and additional tests at 3000 F with both molten fluorides and uranium metal will be made.

Several charges of high-carbon uranium were melted with a zirconium-uranium master alloy in stabilized zirconia crucibles to determine the scavenging effects of carbon by zirconium. A concentration of both carbon and zirconium was evident in each of the melts.

The identification of uranous dibutyl phosphate by an oxidation-reduction titration has proven unattainable, since no method has been found to dissolve it without hydrolyzing the dibutyl phosphate in the diluent systems. Final experiments directed toward establishing the reproducibility of measuring DBP content of plant solvent by a phosphorus analysis have been completed.

Salts for Use in Uranium Heat-Treating Salt Baths

J. W. Droege, K. A. Sense, M. J. Snyder, and R. B. Filbert, Jr.

An experimental program is under way to find a salt bath more suitable for heat treating uranium than the one currently in use.

D-2

As presently constituted, the experiments are designed to measure (1) the hydrogen pickup by uranium from the salt bath, (2) the water pickup by the salt bath from the humid atmosphere, and (3) the corrosion of uranium by the bath as indicated by the loss in weight.

The experimental procedure is as follows. Crucibles containing fused salts are maintained at 1350 ± 10 F in an atmosphere of controlled humidity for 2 days. Pieces of uranium rod, 1/2 in. long with a diameter of about 1/4 in., previously outgassed to 0.1 ppm hydrogen, are then dropped into the melts. After 1 hr, the uranium is removed and water quenched. The uranium is subsequently analyzed for hydrogen, and the salts analyzed for alkalinity and for water. The uranium samples are weighed before and after the salt-bath treatment to test for corrosion loss.

Tests have been started on various mixtures of the following salts: Li_2CO_3; Na_2CO_3; K_2CO_3; LiCl; NaCl; KCl; $CaCl_2$; $BaCl_2$; LiBr; KBr; Li_2SO_4; and K_2SO_4. The first test was made with a water vapor partial pressure above the salt bath of about 30 mm mercury. The results were not surprising in that the sulfates reacted with the uranium, and the hydrogen pickup was higher from the carbonates than from the halides. The results to date are summarized in Table D-1.

TABLE D-1. HYDROGEN PICKUP IN URANIUM AFTER 1 HR AT 1350 F IN 2-DAY-OLD SALT BATHS

Water vapor pressure: 30[a] mm mercury

Composition of Salt Bath, w/o	Weight of Uranium, g	Loss of Weight of Uranium, g	Hydrogen Content of Uranium, ppm
44 Li_2CO_3 - 56 Na_2CO_3	8.1	0.27	8.8
44 Li_2CO_3 - 56 K_2CO_3	7.8	0.15	9.8
50 KCl - 50 Na_2CO_3	7.6	0.24	4.2
22.5 NaBr - 77.5 LiBr	7.5	0.20	2.5
26.5 K_2SO_4 - 73.5 Li_2SO_4		Total loss	

(a) Possibly less.

Tests have also been made at zero and 20 mm mercury water vapor pressure, but analytical results have not yet been obtained. No water analyses of the salts have been made as yet.

D-3

Refractories for Continuous Reduction of Uranium

A. K. Smalley, C. Hyde, J. F. Quirk, and A. G. Allison

Study of the resistance of commercial refractories to attack by selected molten fluorides, and by molten uranium metal, is being done with the objective of selecting a suitable container material for the continuous reduction of uranium fluoride.

In previous work, Norton Company's magnesia and stabilized-zirconia refractories were selected for further testing. These two were the most resistant to fluoride slags of seven commercial refractories assessed in screening experiments.

During the past month, the two refractories were subjected to repeated heating in contact with the fluorides of aluminum, calcium, magnesium, and sodium. Holes, 1-1/2 in. in diameter and 1-3/4 in. deep, were drilled in brick specimens; each hole was filled with a powdered fluoride; and a refractory lid was cemented over the hole to reduce loss by volatilization of the fluoride. The filled specimens were heated in a gas-fired furnace at 3000 F for 4 hr, and then allowed to cool overnight in the furnace. They were similarly recharged and reheated twice more for a total of three heats. After these treatments, the specimens were sectioned axially with a diamond saw, and examined visually.

After each heat, the slag pockets which were charged with NaF, MgF_2, or CaF_2 were empty. This was attributed to the volatility of the fluorides, and to the permeability of the refractory specimens. A white, powdery residue remained in the slag pockets which were charged with AlF_3. This was thought to be alumina. The actual time of exposure of the refractories to the molten fluorides was unknown.

Visual examination indicated that the magnesia specimens were unaffected by AlF_3 under the conditions of the experiment. They were discolored, but were not disrupted by MgF_2 or CaF_2. NaF caused considerable softening of the magnesia refractory.

The stabilized-zirconia specimens showed evidence of severe attack by all four of the fluorides. The attacks were characterized by cracking, bloating, or softening of the refractories.

Additional slag-pocket tests will be made on the magnesia refractory. The drilled specimens will be refilled periodically during the heat, to insure prolonged contact of the specimens with molten fluoride. Also, small specimens will be immersed in molten fluorides in covered platinum

crucibles. In this manner, the fluorides can be contained in contact with the refractory for longer periods of time. In addition, crucibles machined from Norton's magnesia brick will be heated at 3000 F in contact with molten uranium to evaluate resistance of the material to attack by molten uranium at the proposed operating temperature of the reaction vessel.

Metal-Recovery Studies

H. A. Saller, J. R. Keeler, and L. J. Cuddy

The removal of carbon from melts of ingot-top croppings by the addition of zirconium is being investigated.

Four charges of high-carbon uranium were melted with zirconium-uranium master alloy in stabilized-zirconia crucibles. The melts were stirred to mix the zirconium throughout the melt, and were then held 30 min at 2550 F. Three of the melts were frozen in the crucibles; the fourth was bottom poured into a hot-topped graphite mold 1-1/2 in. in diameter by 7-1/2 in. long. Analyses at the top and bottom of these ingots are given in Table D-2.

TABLE D-2. INGOT ANALYSIS

Type Uranium Used in Charge	Nominal Charge Composition, w/o		Type of Ingot	Composition, w/o			
				Top		Bottom	
	Carbon	Zirconium		Carbon	Zirconium	Carbon	Zirconium
High carbon[a]	0.11	0.87	Frozen	0.28	8.58	0.07	0.09
High carbon[a]	0.11	0.81	Poured	0.08	0.29	0.06	0.14
Very high carbon[a]	0.49	3.22	Frozen	1.33	6.03	0.17	0.80
Top crop[b]	0.12	0.93	Frozen	0.55	4.10	0.04	0.18

(a) Prepared at Battelle.
(b) From FMPC production ingot.

A concentration of both carbon and zirconium is evident in the three melts frozen in the crucible.

The poured ingot contained approximately the same amount of carbon and zirconium at the bottom as did the heats frozen in the crucible. The top contained slightly more carbon and considerably more zirconium than the bottom, but the average of both elements is less than that of the charge.

D-5 and D-6

Metallographic examination of these heats is in progress, and additional samples for chemical analyses have been taken.

Degradation Products of Tributyl Phosphate

A. E. Bearse, R. A. Ewing, and S. J. Kiehl, Jr.

Uranous dibutyl phosphate, which may form in TBP-diluent systems containing dibutyl phosphate, is extremely insoluble. Its earlier identification had been made on the basis primarily of the uranium:phosphorus ratio. The desired confirmation by an oxidation-reduction titration has proven unattainable, since no method has been found to dissolve it without hydrolyzing the dibutyl phosphate.

To conclude the experimental program, samples of three different lots of plant solvent have been stripped, in duplicate, for analysis of their (uranyl) dibutyl phosphate content. Each of these stripped samples was spiked to three levels of dibutyl phosphate (1 per cent, 0.1 per cent, and 0.01 per cent of the tributyl phosphate) and again stripped for analysis. Results of these analyses, when completed, should better establish the accuracy and reproducibility of the proposed method for analysis of dibutyl phosphate.

E-1

E. GENERAL FUEL-ELEMENT DEVELOPMENT

H. A. Saller

A broad program on general fuel-element development is in progress. Individual programs are sponsored by the Reactor Development Division of the Atomic Energy Commission, Westinghouse Atomic Power Division - PWR, and Oak Ridge National Laboratory.

Studies on ceramics as high-temperature fuel-element materials were continued. These included investigations to determine the optimum processing conditions for producing strong dense BeO bodies and methods for minimizing uranium losses from $MoSi_2$ and graphite. Preparation of UO_2 specimens for the thermal-fracture studies is under way. Results obtained so far on the characterization of sinterable oxide powder have depended upon the history of the raw material. Specially prepared lots of BeO are being made from various types of sulfates. Studies of various additives such as silicon, beryllium, and silica to UO_2 have continued.

Thermal-expansion data for a number of uranium-molybdenum alloys are reported. A pronounced expansion occurred at the transformation regardless of alloy content. Thermal cycling of these alloys showed little effect in those containing from 5.0 to 12.0 w/o molybdenum. A 3.5 w/o molybdenum alloy appeared to be breaking up. The evaluation of a number of low-melting uranium alloys is continuing. Both thermal-cycling and thermal-stressing tests are under way. A stainless steel fuel matrix containing UN showed considerable reaction after fabrication at 2000 F. Previous tests at 2000 and 2300 F had not shown this reaction. Incomplete reduction of U_2N_3 to UN may be causing the difficulty.

Tests of intentionally defected zirconium-clad pure uranium pins in a window autoclave indicated some delay before rapid attack begins. The fundamental study of uranium corrosion is concentrating on the initiation of corrosion and identification of the soluble material. Studies of the ignition limits in the hydrogen-oxygen-water system are continuing. Some of the equipment is being modified to obtain more precise results.

E-2

CERAMICS

Refractory Fuel Elements

L. D. Loch, C. Hyde, J. R. Gambino, and J. F. Quirk

Refractory fuel elements based on BeO, $MoSi_2$, and graphite are under development for use at very high temperatures. The optimum processing conditions for producing dense BeO ceramics of maximum strength are being determined. Measurements of uranium losses from heated $MoSi_2$- and graphite-matrix elements are being made.

BeO

In previous laboratory work (BMI-1020), exceptionally strong BeO ceramics were prepared from a readily sinterable BeO powder. Indications were that the high strength resulted because substantial densification occurred at temperatures below those for detrimental crystal growth.

A series of experiments is under way to see whether higher strengths are obtainable. One of the approaches is to develop a more sinterable BeO powder, so that densification will occur at lower temperatures. The sinterability of the BeO powder used had been found to depend on the calcining conditions used to prepare it from commercially obtained $Be(OH)_2$. Accordingly, $Be(OH)_2$ is being calcined at various temperatures for different periods. The resultant BeO powders are then compacted and sintered under standard conditions, and bulk density of the sintered compacts is determined as a measure of powder sinterability.

This month, a more precise determination of the best calcination temperature was attempted. Sinterability was determined for BeO powder made by calcining Brush $Be(OH)_2$ in air at 1740 F. The bulk densities of the sintered compacts made from this powder are given in Table E-1, together with data obtained previously from other calcinations. The results indicate that, between 1650 and 1830 F, calcining conditions are not critical. Observed differences in sinterability in this range are probably within the limits of experimental error. The compacts made from BeO calcined at 1740 F sintered to slightly greater density than was obtained from material calcined at higher or lower temperatures.

On the basis of these data and practical considerations of temperature control during calcinations, 2 hr at 1740 F has been selected as the best calcining treatment. Compacts for determining the effect of sintering conditions on strength will be made from BeO powder prepared by this treatment.

TABLE E-1. EFFECT OF CALCINING CONDITIONS ON BULK DENSITY OF SINTERED BeO COMPACTS[a]

Calcining Conditions		Bulk Density After Sintering 1 Hr at Indicated Temperature, F							
		2200		2300		2400		2600	
Temperature, F	Time, hr	G per Cm^3	Per Cent of Theoretical[b]	G per Cm^3	Per Cent of Theoretical[b]	G per Cm^3	Per Cent of Theoretical[b]	G per Cm^3	Per Cent of Theoretical[b]
1470	1	2.50	82.5	2.89	95.5	3.00	99.0	2.89	95.6
	2	2.49	82.3	2.90	95.8	2.98	98.5	2.95	97.5
	4	2.43	80.2	2.93	96.8	2.96	97.8	2.98	98.5
1650	1	2.57	85.0	2.93	96.8	2.99	98.7	2.98	98.5
	2	2.57	85.0	2.94	97.3	3.00	99.0	2.99	98.7
	4	2.60	85.9	2.96	97.8	2.97	98.1	3.00	99.0
1740	1	2.50	82.7	2.96	97.8	2.99	98.7	2.99	98.7
	2	2.42	80.0	2.98	98.5	2.99	98.7	3.01	99.4
1830	1	2.66	88.0	2.97	98.2	3.00	99.0	3.01	99.4
	2	2.50	82.5	2.93	96.8	3.00	99.0	3.01	99.4
	4	2.56	84.6	2.95	97.4	3.00	99.0	3.01	99.4
2010	1	2.30	76.0	2.76	91.2	2.87	94.8	2.98	98.5
	2	2.21	73.0	2.64	87.2	2.88	95.2	2.98	98.5
	4	2.17	71.7	2.67	88.2	2.85	94.2	2.98	98.5
2190	1	1.91	63.1	2.32	76.6	2.54	79.1	2.85	94.2
	2	1.85	61.2	2.26	74.7	2.46	81.3	2.78	92.0
	4	1.88	62.0	2.25	74.4	2.59	85.8	2.91	96.1

(a) Compacts 1/2 in. in diameter by 1/2 in. high were pressed in a rigid steel die at 20,000 psi with 3 w/o of camphor as a temporary binder.
(b) The theoretical density of BeO was taken as 3.025 g per cm^3.

E-4

$MoSi_2$

Because of its resistance to oxidation and its high-temperature strength, the most likely application for $MoSi_2$ as a fuel-element matrix appears to be in air-cooled reactors operating at temperatures of about 2500 F. Three major problems are foreseen: (1) fuel loss to the coolant, (2) fission-product loss to the coolant, and (3) radiation damage to the $MoSi_2$. Recent experiments indicate that uranium loss in air at 2500 F may not be excessive, unless loss is influenced greatly by the rate of air flow.

The rate of uranium loss from a $MoSi_2$-30 w/o UO_2 compact was determined in a 72-hr test at 2500 F using slowly flowing dry air. The compact was pretreated in air at 2300 F to promote self-glazing.

An average loss rate of 2 μg of uranium per cm^2 per hr was obtained. The loss rate during the first 25 hr was about 50 per cent greater than the average rate for the 72-hr exposure.

The total uranium lost in 72 hr was 0.1 per cent of the original UO_2 loading. This amount of loss is probably tolerable for some proposed reactor applications. However, in this experiment, the Reynolds number of air flowing around the bar-shaped compact was about 1, corresponding to a very low flow rate. Several more experiments, using Reynolds numbers of 100 to 1000, will be made to determine the effect of air-flow rate on uranium loss.

Also, an investigation will be initiated of irradiation damage and fission-product retention in $MoSi_2$-matrix elements. The objectives will be to determine which types of radiation damage are most important at high temperatures and to develop a method of fuel placement that will minimize fission-product damage and fission-product loss.

Graphite

Major problems expected in the use of graphite-matrix elements at high temperature are the retention of fuel and of fission products, presuming they are to be used in nonoxidizing atmospheres.

Recent measurements of uranium loss during vacuum heating of graphite elements fueled with UO_2 or UC_2 showed that:

(1) Retention of fuel is improved by placement of uranium compound in relatively massive form, well below the surface of the element.

(2) Surface coatings can decrease fuel loss by a factor of 10 or more at 3000 F.

(3) Fuel losses from UC_2 loadings are 30 to 50 times greater than from UO_2 loadings.

Work was continued on constructing equipment for measuring uranium losses in flowing argon. Coated and uncoated graphite tubes, axially loaded with UO_2 or UC_2, will be heated at 2500 to 2800 F while loss rates are determined as functions of temperature and Reynolds number.

Thermal Fracture of Ceramic Structures

A. K. Smalley and W. H. Duckworth

This research program is concerned with problems arising from thermal stresses induced in ceramic reactor materials. Specifically, it is aimed at providing a basis for predicting thermal-stress conditions under which ceramic components will fracture and at providing information on the resistance of materials and shapes of interest.

Work during the past month was continued on developing UO_2 specimens for evaluation of this material. Sintering studies on a body similar to the WAPD-PWR fuel-element body indicated that specimens of the desired shape, hollow circular cylinders formed by hydrostatic pressing, sintered to about 84 per cent of the true density of UO_2 when heated for 2 hr at 3200 F in dry hydrogen. These specimens were made from minus 200-mesh MCW UO_2. It is planned, during January, to begin evaluating the thermal-fracture resistance of these cylinders.

In addition to this work, development was continued on specimens of a denser body made from a different form of UO_2 powder. To prepare the powder, MCW UO_3, as received, was washed several times with distilled water, and then dried. The dried UO_3 was dehydrated by heating in air for 24 hr at 300 C, and then rehydrated by ball milling for 1 hr in distilled water. The UO_3 hydrate was dried, and then reduced to UO_2 by heating in a stream of wet hydrogen for 2 hr at 600 C. This process yielded a readily sinterable form of UO_2 powder. Pellets, formed by compression molding at 20,000 psi and then hydrostatic pressing at 100,000 psi, were sintered to bulk densities exceeding 95 per cent of theoretical by heating them for 1 hr at 2600 F in dry hydrogen. Hollow, cylindrical thermal-fracture specimens will be made from the readily sinterable UO_2, and their resistance to thermal fracture will be compared with that of the WAPD-type UO_2 specimens.

E-6

The thermal-fracture characteristics of a high-Al_2O_3 spark-plug porcelain are being evaluated. In the first phase of this evaluation, experiments are being made to determine the temperature dependence of the material factor. Early experiments on hollow cylindrical specimens indicated that the glass phase, comprising about 8 w/o of the body, caused pyroplastic flow before fracture occurred in specimens 2 in. long and 1-in. ID having OD's less than about 1.6 in. Various expedients may be employed to decrease the specimen temperature and to circumvent pyroplastic flow in the specimens. When the temperature-dependence experiments on porcelain specimens are completed, the relative shape factors of circular, square, and triangular specimens will be evaluated. These data will be compared with similar data at hand on ruby and SiC specimens to further establish the validity of the concept of separable shape and material factors.

Characterization of Sinterable Oxide Powders

J. F. Quirk, C. Hyde, and G. B. Engle

Factors affecting the sinterability of refractory oxide powders are being investigated. In previous work (BMI-1020), compacts of BeO powder prepared by calcining commercial $Be(OH)_2$ sintered to essentially theoretical density at temperatures as low as 2400 F. However, similar compacts of BeO powder prepared from laboratory-made $Be(OH)_2$ did not approach theoretical density, even when sintered at 3200 F. The preparation of the starting material, in this case $Be(OH)_2$, was studied this month as an approach to the more general problem of oxide sinterability and characterization.

An effort was made to prepare a high-purity $Be(OH)_2$ that would decompose to a readily sinterable BeO powder. Variations in the solution concentrations, digestion, and washing were investigated in the process of preparing $Be(OH)_2$ by precipitation from a $BeSO_4$ solution with NaOH. None of the BeO samples prepared by calcination of the hydroxides resulting from this investigation proved to be readily sinterable.

In view of these results, investigation will be made to determine whether differences in the starting sulfate affected the sinterability of the processed BeO powder.

Surface areas are being measured by the BET method on BeO powders prepared by calcination of commercial $Be(OH)_2$. The results of determination made thus far are shown in Table E-2. In this table, only the BeO powder resulting from the 2190 F calcination was poorly sinterable.

E-7

TABLE E-1. EFFECT OF CALCINING TEMPERATURE ON BET SURFACE AREA OF BeO[a] POWDER

Calcining Temperature, F	Surface Area, m^2 per g	Equivalent Average Particle Diameter, μ[b]
782	106	0.019
1110	37	0.054
1470	34	0.058
1830	6.4	0.32
2190	1.8	1.1

(a) Brush $Be(OH)_2$, Lot W-1659, was calcined in air for 2 hr at temperatures shown.
(b) Assuming solid spherical particles of theoretical density.

Experiments will be made in January to characterize this BeO ignition series by X-ray and thermogravimetric analyses.

Modified UO_2 Fuel-Element Cores

D. J. Bowers, P. T. Woodberry, and M. J. Snyder

The objective of this work in support of the WAPD-PWR program is to develop UO_2 cores which have high resistance to water logging and thermal cracking. Toward this end, core materials made of MCW UO_2 in combination with up to 20 volume per cent of selected metal and oxide additives are being evaluated. The density, strength, and elastic modulus of sintered compacts are measured as the initial evaluation.

Past work has included the fabrication and testing of compacts containing silicon, silicon nitride, beryllium, or zirconia additives. Most promising results were obtained using zirconia. Beryllium was the least promising additive. The studies of compacts containing zirconia, silicon, and silicon nitride have been continued this month.

In an attempt to improve the density over that obtained earlier with hydrostatically pressed and sintered compacts, mixtures of UO_2-silicon containing 5, 10, or 20 volume per cent silicon were hot pressed in graphite dies at 2600 F and 3500 psi for either 10 or 30 min. The resulting compacts had a density of about 70 per cent of theoretical; a density of as high as 86 per cent was obtained by the technique used previously. Increasing the hot pressing time to 30 minutes did not increase the density of the compacts.

E-8

In the extended study of UO_2-silicon mixtures fabricated by hydrostatic pressing followed by sintering in hydrogen, compacts were sintered at a higher temperature, 3200 F, in an attempt to promote better bonding of UO_2 to silicon through liquid-solid contact. Much of the silicon escaped from the compacts, however, and the compacts were relatively porous.

In the continuation of the work with compacts containing silicon nitride (nitrided in the furnace from silicon in the compact), a higher temperature was used in an attempt to promote greater density. However, the compacts were no more dense than those made previously. Data obtained for both the UO_2-silicon sintered compacts and the nitrided UO_2-silicon compacts are given in Table E-5. Further work with silicon as an additive will be given consideration only after other additives have been investigated.

Work this month also included the evaluation of another additive, berylliа. UO_2 compacts containing 5, 10, or 20 volume per cent BeO were hydrostatically pressed at 100,000 psi and then sintered. Physical-property data obtained from compacts sintered at 2800 and 3200 F are given in Table E-6.

The compacts sintered at 3200 F were appreciably more dense than were those sintered at 2800 F. Tensile strengths of these compacts will be determined before further work with BeO is planned.

The investigation of zirconia as an additive to UO_2 cores had been expanded to include the use of stabilized zirconia and a very pure zirconia along with the continuing study using cp zirconia. Data are not yet available.

Planned work will include screening evaluations of other additives, silica and ceria.

METALLURGY

Uranium Alloys for High-Temperature Application

R. F. Dickerson and W. E. Murr

There is considerable interest in uranium-zirconium and uranium-molybdenum alloys for use as high-temperature-reactor fuels. This program is concerned with the determination of physical and mechanical properties of binary alloys of uranium containing 3.5 through 15.0 w/o molybdenum and 3.0 through 20 w/o zirconium at elevated temperatures.

TABLE E-3. PHYSICAL PROPERTIES OF UO_2 COMPACTS MODIFIED BY SILICON

Composition, volume per cent		Sintering Conditions			Young's Modulus, 10^6, psi	Bulk Density, per cent of theoretical	Theoretical Bulk Density[a], g per cm^3
UO_2	Silicon	Temperature, F	Time, hr	Atmosphere			
100[b]	--	3350	1	Hydrogen	22.1	97.1	10.97
95	5	3350	4	Nitrogen	Broke	74.9	10.54
90	10	3350	4	Nitrogen	5.92	68.6	10.11
80	20	3350	4	Nitrogen	4.69	87.6	8.30
95	5	3350	1	Hydrogen	16.2	97.3	10.54
90	10	3350	1	Hydrogen	--	76.3	10.11
80	20	3350	1	Hydrogen	--	69.7	8.35

(a) Calculated on the following basis: density of UO_2 = 10.97 g per cm^3; density of silicon = 2.40 g per cm^3; density of Si_3N_4 = 3.44 g per cm^3.
(b) Reference material.

TABLE E-4. PHYSICAL PROPERTIES OF UO_2 COMPACTS MODIFIED BY BeO

Composition, volume per cent		Sintering Conditions			Young's Modulus, 10^6, psi	Bulk Density, per cent of theoretical	Theoretical Bulk Density[a], g per cm^3
UO_2	BeO	Temperature, F	Time, hr	Atmosphere			
95	5	3350	1	Hydrogen	--	84.7	10.57
90	10	3350	1	Hydrogen	--	81.0	10.13
80	20	3350	1	Hydrogen	15.3	80.8	8.30
95	5	3350	1	Hydrogen	23.3	91.7	10.57
90	10	3350	1	Hydrogen	23.1	92.1	10.13
80	20	3350	1	Hydrogen	22.7	86.0	8.30

(a) Calculated on the following basis: density of UO_2 = 10.97 g per cm^3; density of BeO = 3.02 g per cm^3.

E-10

Previously, linear-thermal-expansion data of uranium-molybdenum alloys of retained and of partially and fully transformed gamma structures were reported. In this report similar data is given for uranium-zirconium alloys that have been subjected to heat treatments designed to produce small-grain random structures. Table E-5 lists heating-curve information obtained near the transformation zone of each composition. Two heat-treated conditions of each composition are represented in the table. The two were selected for dilation studies from a total of seven screening treatments given each composition. The selection was made from a study of microstructure and grain size of the heat-treated specimens.

Inspection of the table reveals that a pronounced expansion took place in each composition upon being heated through the transformation zone. Uranium-20 w/o zirconium specimens changed less during transformation than those of any other composition.

The uranium-3.5 through 12.0 w/o molybdenum series of alloys was thermal cycled for a number of times between 150 and 725 C. Table E-6 lists data obtained after 60, 250, and 530 thermal cycles. As shown by the table, the uranium-5 through 12 w/o molybdenum alloys changed very little dimensionally due to cycling. However, the uranium-3.5 w/o molybdenum specimen appeared to be breaking down. At the end of 530 cycles, the specimen's surface had roughened and the specimen had distorted. Thermal-cycling information is being obtained on uranium-molybdenum alloys that have been water quenched after 1 hr at 800 C. In addition, uranium-zirconium specimens are undergoing cycling at two temperatures and in several conditions of heat treatment. The objective in studying the cycling behavior of the alloys is for correlation with physical properties to be obtained on the alloys at a later date.

Preparation and Properties of Low-Melting Alloys

H. A. Saller, R. F. Dickerson, and W. E. Murr

The purpose of this investigation is to obtain pertinent information concerning castable uranium alloys. These alloys are of interest as possible reactor fuels. The investigation is concerned with evaluation of alloys by means of stress and thermal cycling and determination of physical and mechanical properties.

During the past several months, a number of alloys have been thermal cycled in the temperature range of 300 to 1275 F. In every case, the alloys deformed less due to cycling than did the reference alloy, uranium-5 w/o chromium. Five alloys showed very good resistance to deformation and

TABLE E-5. TRANSFORMATION-ZONE DATA FOR URANIUM-ZIRCONIUM ALLOYS

Specimen Composition (Balance Uranium), w/o	Temperature at Start of Transformation, C	Mean Linear Expansion at Start of Transformation, 10^{-6} per deg C	Temperature at End of Transformation, C	Mean Linear Expansion at End of Transformation, 10^{-6} per deg C
3.0 Zr[a]	680	19.46	760	24.63
3.0 Zr[b]	685	19.42	720	23.11
5.0 Zr[c]	685	19.00	725	23.48
5.0 Zr[b]	695	19.23	715	24.46
7.0 Zr[c]	690	18.89	720	22.88
7.0 Zr[b]	690	18.49	710	22.65
10.0 Zr[c]	690	19.15	720	23.59
10.0 Zr[d]	685	19.64	725	24.09
15.0 Zr[d]	625	15.88	700	19.58
15.0 Zr[c]	630	15.73	730	19.80
20.0 Zr[e]	625	14.81	710	17.40
20.0 Zr[d]	620	14.08	720	16.74

(a) One hr at 800 C, hold 24 hr at 670 C, furnace cool.
(b) One hr at 800 C, isothermally transform 2 hr at 550 C; 5 min at 780 C, isothermally transform 2 hr at 550 C; repeat last step.
(c) One hr at 800 C, furnace cool to 670 C, hold at 670 C for 24 hr and then furnace cool.
(d) One hr at 800 C, furnace cool to 750 C, furnace cool from 750 C to 570 C at 1/2 C per min.
(e) One hr at 800 C, isothermally transform at 550 C for 2 hr and water quench.

TABLE E-6. THERMAL-CYCLING DATA FOR HEAT-TREATED URANIUM-MOLYBDENUM ALLOYS CYCLED BETWEEN 150 AND 725 C

Specimen Composition (Balance Uranium), w/o	Precycling Dimensions		After 60 Cycles		After 250 Cycles		After 530 Cycles	
	Diameter, in.	Length, in.	Diameter, in.	Length, in.	Diameter, in.	Length, in.	Diameter, in.	Length, in.
3.5 Mo[a]	0.4358	1.0002	0.4346	1.0074	0.4317	1.0265	Unable to obtain accurate measurements	
5.0 Mo[b]	0.4350	1.000	0.4344	1.0040	0.4362	1.0041	0.4363	1.0043
7.0 Mo[c]	0.4370	1.0002	0.4380	1.0030	0.4380	1.0030	0.4380	1.0028
9.0 Mo[d]	0.4358	0.9997	0.4359	1.0013	0.4360	1.0013	0.4358	1.0014
12.0 Mo[e]	0.4363	1.0003	0.4362	1.0003	0.4361	0.9999	0.4360	0.9998

Note: Diameter measured perpendicular to central axis; length measured parallel to central axis.

(a) One hr at 800 C, air cool.

(b) One hr at 800 C, furnace cool to 500 C, hold 24 hr, air cool.

(c) One hr at 800 C, furnace cool to 500 C, hold 100 hr, air cool.

(d) One hr at 800 C, furnace cool to 500 C, hold 200 hr, air cool.

(e) One hr at 800 C, furnace cool to 500 C, hold 2 weeks, air cool.

will be examined more carefully. The five alloys are listed below in order of their decreasing resistance to deformation.

(1) Uranium-5.97 w/o chromium-2.69 w/o thorium

(2) Uranium-5.0 w/o chromium-1.0 w/o molybdenum

(3) Uranium-4.82 w/o chromium-0.72 w/o silicon

(4) Uranium-2.56 w/o chromium-2.26 w/o vanadium

(5) Uranium-5.0 w/o chromium-1.0 w/o germanium.

Work on the stress cycling apparatus is continuing. A vacuum-tight shell has been constructed as a housing for the induction heat coil. This was done to prevent oxidation of the specimens during test. Tests have been run on cylinders of two compositions, but are inconclusive because of the short duration of testing. To obtain maximum information from the equipment, meaningful tests must be developed that will provide a basis for screening out less desirable alloys. As uranium-5 w/o chromium has been a basis for comparison in earlier work, it is planned to continue using this alloy for comparative purposes. After all alloys have been subjected to thermal stressing, an attempt will be made to correlate their behavior with physical and mechanical properties.

Compartmentalized Fuel Elements

H. A. Saller, D. L. Keller, and G. W. Cunningham

The development of improved cermet-type fuel elements for service at moderate to high temperatures is the major objective of this program.

A stainless steel element containing a UN fuel dispersion was hot rolled at 2000 F to a 4 to 1 reduction. Metallographic examination revealed that an extensive reaction occurred between the fuel particles and the matrix material. Some reaction was also noted with the 18-8 stainless steel cladding.

Previous studies have indicated that no reaction occurs between stainless steel and UN at temperatures between 2000 and 2300 F. However, since the uranium compound used in the rolling studies was prepared by the reduction of U_2N_3 to UN, the powder is being examined to determine if the reduction to UN was complete. The phases present in the rolled specimen will also be determined.

E-14

Several cold-pressed compacts of UC dispersed in a Zircaloy 2 matrix have been prepared and are being fabricated into sealed packs suitable for hot rolling into clad sheet.

CORROSION

The Reaction of Fuel-Element Cores With Elevated-Temperature Water

D. R. Grieser and E. M. Simons

A study of the reaction of fuel-element cores with 600 F water is being made as a part of the general fuel-element development program.

Two zirconium-clad pure uranium pin specimens, with a drilled pinhole fault through the unbonded cladding of each, were subjected suddenly to 600 F water in the windowed autoclave. A corrosion rate for the first of these tests was given in BMI-1057 as 4850 mg/(cm^2)(hr). This was calculated from the pressure record of evolved hydrogen before the motion pictures of the test were developed. Actually, the pictures showed that it took longer for complete oxidation of the core than was estimated from the pressure record. Using data from the pictures, a revised corrosion rate of 3350 mg/(cm^2)(hr) at 610 F was obtained. On the same basis, the second test, which was a duplicate of the first, yielded a corrosion rate of 3400 mg/(cm^2)(hr) at 605 F.

There is some evidence that the reaction does not begin immediately upon contact of the specimen with the hot water. In some instances, there have been noticeable delays before the appearance of hydrogen bubbles. This situation will be examined more closely.

Mechanism of Uranium Corrosion

J. B. Schroeder, C. M. Schwartz, and D. A. Vaughan

Corrosion tests are being carried out in boiled, deionized water to determine the mechanism of aqueous uranium corrosion. Previously reported work has shown: (1) that the products of corrosion are $UO_3 \cdot xH_2O$, $UO_{2.88}$ (cubic structure), and a soluble phase which has not been identified, (2) that uranium hydride reacts slowly with water, and (3) that the corrosion rate is not a linear function of time.

E-15

Work done this month has been concerned with the initiation of corrosion and identification of the soluble material. To determine when corrosion starts, one sample was run in apparatus designed to collect all of the gaseous corrosion product. Figure E-1 shows the results of this test. These data show that no gas (hydrogen) was evolved during the first day, within the limits of accuracy of the equipment (0.2 cm^3). To determine the effect of an air-oxidized film on the length of this initial period of very slow corrosion, two samples were run together. The first was electropolished and heated to 85 C in air for 1/2 hr before testing. The air exposure of the second was kept to a minimum. After 5 days the weight loss was the same for both samples, indicating that either the air-oxidized film is not important, or that insufficient precautions were taken to avoid the formation of this film.

The material obtained by evaporating the clear water from the corrosion test has been found to give four different diffraction patterns. None of these compounds have been identified. Chemical analysis has shown one of these compounds to contain 12 w/o water of hydration and 16 w/o uranium. Spectrographic analysis has not been completed.

Future work is planned to investigate the role of uranium hydride in corrosion and to continue the work already in progress.

Correlation of Microstructure With Corrosion Behavior of Uranium Alloys

W. K. Boyd, W. E. Berry, and R. S. Peoples

A study of the relationship between microstructure and corrosion behavior of uranium alloys is being made as a part of the general fuel-element development program.

Present studies are concerned with following the progress of corrosion in boiling water of uranium-12 w/o molybdenum alloys. Metallographically prepared and etched samples are being examined after periodic exposure to boiling water. To date, no significant changes in microstructure have been observed. These studies are being continued and expanded to include other alloy compositions.

E-16

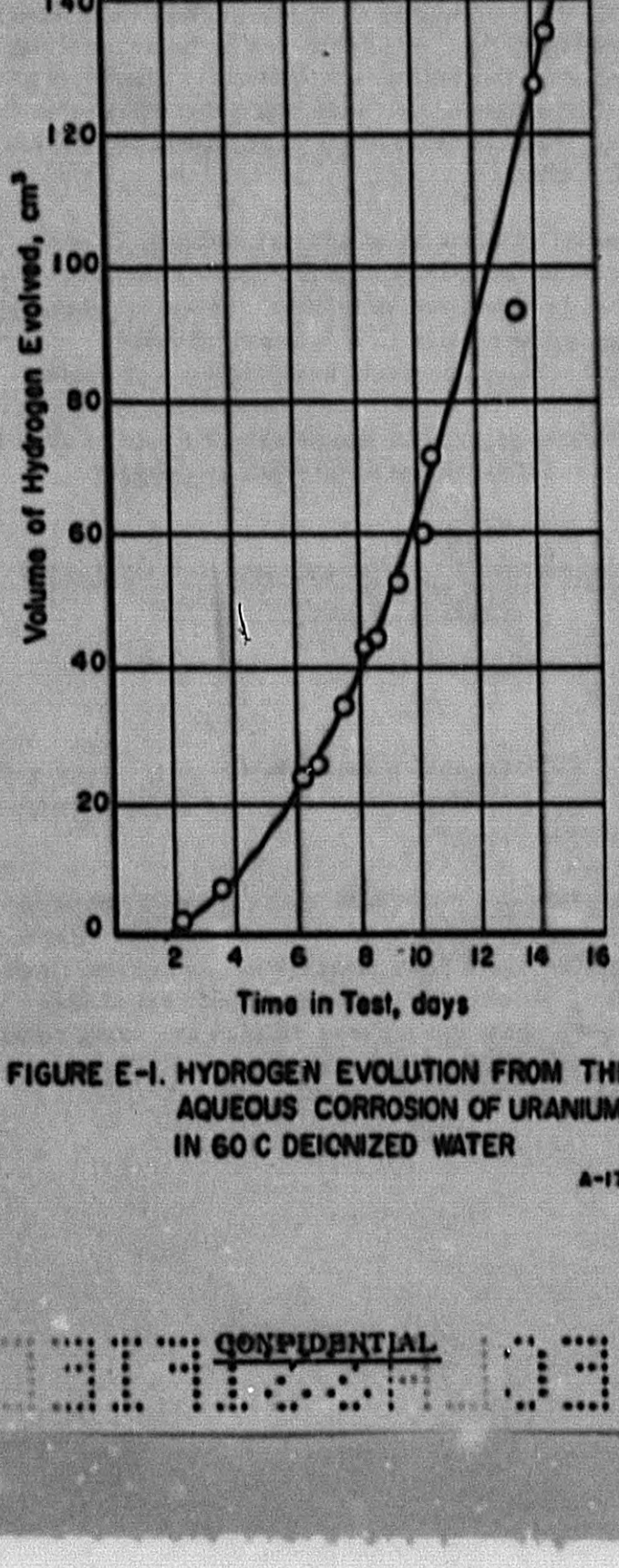

FIGURE E-1. HYDROGEN EVOLUTION FROM THE AQUEOUS CORROSION OF URANIUM IN 60 C DEIONIZED WATER

A-17116

E-17 and E-18

Ignition Reaction Limits in the Hydrogen-Oxygen-Water System at Elevated Temperatures

E. F. Stephan, N. S. Hatfield, and R. S. Peoples

Studies for ORNL of the reaction limits of hydrogen-oxygen mixtures in saturated water vapor at elevated temperatures were continued.

Repeated tests in 3-in. -diameter autoclaves at temperatures between 425 and 600 F with both 1:1 and 2:1 hydrogen:oxygen mixtures in saturated water vapor show that the reaction limits are below 20±5 psia partial pressure of the mixture. It is not practical to determine mixture partial pressures below 20 psia at these temperatures by pressure measurement; therefore, it will be necessary to modify the present equipment so the vapor phase in the reaction vessel can be analyzed in order to determine reaction limits.

Because there is such a great difference in the limits between a 1.5-in.- and a 3-in. -diameter autoclave, future work will be concerned principally with the larger vessel.

F-1

F. STUDIES OF ZIRCONIUM-URANIUM ALLOYS

H. A. Saller

The Reactor Development Division of the Atomic Energy Commission is sponsoring a broad program on zirconium-uranium alloys containing up to 70 w/o uranium.

Results on the various separate programs on constitutional diagram, kinetics, heat treatment, corrosion, and radiation stability are reported in the following sections. No major conclusion can be drawn at this time.

Constitution of Zirconium-Uranium Alloys

F. A. Rough, A. A. Bauer, and J. R. Lulay

Although specimen preparation is continuing, no further results are available at present. It is expected that measurements will be made and data will be taken during the month of January.

Transformation Kinetics and Hardening Reaction of Zirconium Alloys Containing up to 20 w/o Uranium

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

The time-temperature-transformation relations are being determined in an effort to evaluate heat treatments and their effect upon mechanical properties in zirconium-uranium alloys containing up to 20 w/o uranium. The hardening reaction of quenched alloys is being studied as a basis for the improvement of zirconium-uranium alloy properties.

Quenched Alloys

Vacuum-fusion oxygen analyses were made on three samples described in report BMI-1057. The effect of oxygen on the hardness can be seen by a comparison with quenched alloys made from iodide crystal-bar zirconium. Hardness data and oxygen analyses are presented in Table F-1.

TABLE F-1. COMPARISON OF SPONGE-BASE AND CRYSTAL-BAR ALLOYS QUENCHED FROM 1000 C

Sponge-Base Alloys					Crystal-Bar Alloys		
Nominal Uranium, w/o	Original Oxygen, w/o	Final Oxygen, w/o	Vickers Hardness Number	Structure (X-ray)	Nominal Uranium, w/o	Vickers Hardness Number	Vickers Hardness Number Increase by Quenching	Structure (X-ray)
7	0.094	--	274	α'	7	200	38	
10	0.094	0.359	314	$\beta + \alpha'$	10	229	51	$\alpha' + \omega^{(a)}$
15	0.094	--	433	$\alpha + \omega$	15	273	51	$\alpha + \beta + \omega$
					20	290	50	

(a) Slight trace of ω phase.

F-3

No data are available on the oxygen content of the crystal-bar alloys. The oxygen content of a 10 w/o uranium alloy held at 1000 C for 30 min, and water quenched was 0.179 per cent. The value for a similar alloy (held at 1000 C for 60 min) was 0.359 per cent. A 10 w/o alloy slow cooled from 1000 C (2-1/2 F/min) analyzed 0.428 per cent oxygen. These data indicate oxygen pickup by some process other than residual gas contamination or diffusion through the Vycor-capsule wall. Reduction of SiO_2 by zirconium at 1000 C might be one possibility. A second source might be residual oxide left from improper sample preparation.

Preliminary runs with the quenching dilatometer give an M_s value of about 360 ± 10 C for the 10 w/o uranium alloy (oxygen content: 0.094 w/o).

Slow-Cooled Alloys

The oxygen content, 0.428 w/o, of the slow-cooled 10 w/o alloy explains the high Knoop hardness values obtained in microhardness tests. The alpha-phase hardness was 332 Knoop. Oxygen shows a strong preference for zirconium over uranium or uranium-rich phases. X-ray patterns revealed a lattice expansion of the alpha phase. Thus, it is reasonable to expect nearly all the oxygen to partition itself in the α and exert a marked hardening effect.

Aging Studies

Quenched crystal-bar alloys were aged at 400 C for 1 hr and 4 hr. Aging for 1 hr resulted in a marked increase in hardness, but 4 hr caused a slight softening. Additional times will be selected in order to determine the tempering and/or the transition-phase kinetics. Available data are tabulated below.

Uranium, w/o	Vickers Hardness, kg/mm^2		
	Quenched	After 1 Hr at 400 C	After 4 Hr at 400 C
7	200	193	191
10	229	292	287
15	273	341	323
20	290	342	327

X-ray results were completed for the 10 and 15 w/o uranium alloys. Data are presented in the following tabulation.

Uranium, w/o	Phases Present[a] Aged 1 Hr	Aged 4 Hr
10	α'(s) + ω(vf)	α'(s) + ω(vf)
15	ω(s) + β(s) + α(trace)	ω(s) + β(s) + α(f)

(a) Phase X-ray intensity: (s) strong, (f) faint, (vf) very faint.

Transformation Kinetics of Zirconium Alloys Containing 20 to 70 w/o Uranium

F. A. Rough and A. A. Bauer

The kinetics of the beta-zirconium-to-epsilon decomposition is being investigated in alloys containing 20 to 70 w/o uranium by following hardness and structural changes and, in the 50 w/o alloy, electrical resistivity changes, during transformation.

Resistivity measurements have been made during transformation of a 50 w/o uranium alloy which had been quenched to retain the beta-zirconium phase. These measurements indicate that the transformation of retained beta requires at least 10 min at 500 C, as evidenced by a continual resistance increase during this period after the specimen had reached the temperature of transformation. Specimens are being prepared for additional measurements at this and lower temperatures. These measurements are to be correlated with hardness and structural changes.

Hardness measurements have been taken on a series of specimens containing 22 to 70 w/o uranium after isothermal transformation at 400, 500, and 575 C for various periods of time. All these alloys show an aging peak after short times at 500 and 575 C. This hardness peak, which is believed to be associated with the beta-zirconium-to-epsilon decomposition, occurs very rapidly in the 22 to 30 w/o uranium alloys, making it doubtful that gamma can be fully retained in these two alloys except in very small sections and then only under ideal quenching conditions.

The 22 and 30 w/o alloys also show a slight secondary hardness peak, while the 70 w/o uranium alloys show a pronounced secondary hardness peak. It appears that this peak may be related to precipitation aging. Further examination of these samples is planned to determine the nature of this aging.

F-5

Heat Treatment of Zirconium-Uranium Alloys

F. A. Rough and W. Chubb

Zirconium-uranium alloys have shown considerable promise as fuel-element materials, but because of the extreme sensitivity of some zirconium-uranium alloys to heat-treatment variables, the alloys have proven very difficult to handle. It is the purpose of this investigation to define the heat treatment behavior of these alloys. Alloys containing up to 70 w/o uranium and up to 3000 ppm oxygen have been melted and fabricated.

End-quench tests on all the alloys prepared have now been completed. These tests show that oxygen contamination has a pronounced hardening effect on all alloys containing up to 50 w/o uranium. Oxygen contamination showed little effect on the hardenability of alloys containing 55 and 60 w/o uranium. An alloy containing 70 w/o uranium showed a slight tendency toward lower hardness in end-quench bars containing increasing amounts of oxygen. This effect has been interpreted to mean that oxygen shifts the eutectoid composition toward uranium. Since maximum hardness occurs in the binary system at 80 w/o uranium, a shift of the eutectoid toward 80 w/o would tend to result in lower hardness.

Alloys containing 7, 10, and 15 w/o uranium show no severe hardening effects in end-quench tests. Hardness levels below 300 DPH were observed in alloys containing zirconium plus 7 w/o uranium and 0.02 w/o oxygen, zirconium plus 7 w/o uranium and 0.09 w/o oxygen, zirconium plus 7 w/o uranium and 0.3 w/o oxygen, zirconium plus 10 w/o uranium and 0.02 w/o oxygen, zirconium plus 10 w/o uranium and 0.09 w/o oxygen, and zirconium plus 15 w/o uranium and 0.02 w/o oxygen.

Alloys containing 20 to 70 w/o uranium show severe hardening effects in end-quench tests. Hardness levels in most cases ranged from 200 to 500 DPH with higher hardnesses occurring with increasing alloy content.

The data indicate that beta zirconium cannot be fully retained at the highest quenching rates when it contains only 20 to 30 w/o uranium. It follows that these alloys must be held at temperature for long periods and cooled slowly if the product is to be workable. Rapid cooling of these alloys results in hardnesses ranging from 300 to 450 DPH, and quench-cracking may occur.

Beta zirconium can be fully retained in alloys containing 40 to 70 w/o uranium. In this range of compositions and regardless of oxygen content, fully-retained beta zirconium appears to have a uniform hardness of about

200 to 250 DPH. Sections up to 1/8 in. thick can be satisfactorily quenched to a low hardness level by heating to 750 or 800 C for 1 hr and water quenching. Higher temperatures and thicker sections are to be avoided, although sections up to 1/4 in. thick may be satisfactorily treated in some cases.

When alloys containing 40 to 70 w/o uranium are cooled at intermediate rates, hardnesses between 300 and 500 DPH result. Quench cracking has been known to occur in alloys in this composition range when cooled at some critical rate.

Low hardness has also been developed in the 40 to 70 w/o uranium alloys by annealing for 4 to 12 hr at 550 to 600 C. A hardness of 240 to 270 DPH is achieved in alloys of this group containing less than 0.03 w/o oxygen.

Two complete sets of alloys have been heat treated for corrosion testing. One set was heated at 650 C for 12 hr and water quenched, thereby producing fine-grained alpha zirconium plus retained beta or just retained-beta structures. The other set was heated at 650 C for 12 hr, cooled to 550 C for 4 hr, and water quenched, thereby producing alpha zirconium plus epsilon or just epsilon structures. Corrosion tests are expected to show if there is any fundamental difference in the corrosion behavior of retained beta and epsilon.

It has been found possible to produce a network of alpha zirconium in beta-grain boundaries by quenching alloys containing 20 to 40 w/o uranium from the beta phase and then reheating into the alpha plus beta field. Attempts to control the amount of alpha zirconium in the grain boundaries have as yet been unsuccessful. Experiments designed to pinpoint the variables are in progress.

Corrosion of Zirconium-Uranium Alloys

W. E. Berry, E. L. White, and R. S. Peoples

The corrosion behavior of zirconium-uranium alloys is being studied as part of the over-all program to study the properties of zirconium-uranium alloys.

Corrosion tests are being conducted in 680 F degassed water. As reported in BMI-1057, the microstructure on the surface of a number of variously heat-treated zirconium-22 w/o uranium specimens was different from that of the bulk of the sample. Duplicate samples have been machined below this surface layer and have been corrosion tested. After 3 weeks'

exposure, corrosion rates range from plus 0.03 to minus 0.09 mg/(cm^2)(hr). Thus, the corrosion behavior can be affected through heat treatment. Further study is necessary to determine whether this effect is due to the retention of a phase (such as beta) or the result of the dispersion of one phase in another, such as epsilon in alpha.

The effect of heat treatment and zirconium melting stock on corrosion behavior is being studied. Alloys containing 7 through 50 w/o uranium with crystal bar, sponge, and high-oxygen (up to 0.3 w/o) sponge zirconium and which have been water quenched from 650 C have been exposed 2 weeks in 680 F water. Oxygen appears to increase corrosion rate of the 50 w/o alloy. Corrosion rates are minus 0.11, minus 0.18, and minus 0.20 mg/(cm^2)(hr), respectively, for those alloys prepared from crystal bar, sponge, and sponge zirconium with 0.15 w/o oxygen. These studies are being continued.

Radiation Stability of Zirconium-Uranium Alloys

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

The radiation-effects program on the uranium-zirconium alloys is being continued. The initial work will be concerned with zirconium-22 and -40 w/o uranium alloys which were irradiated by KAPL and zirconium-22 w/o uranium alloys which will be irradiated by Battelle under controlled conditions.

As this program is essentially a fundamental investigation, an attempt will be made to determine the mechanics of fission-product diffusion, rate of fission-gas release, and the amount of fission-product release. Post-irradiation heat treatments and thermal-cycling treatments will be attempted to determine their similarity with in-pile thermal conditions.

Postirradiation test equipment will be designed to permit effective and efficient use of hot-lab personnel and to obtain the maximum amount of technical information from any given test specimen. Whenever practical, long-term test apparatus will be so designed to permit the experiment to operate outside of the hot cells proper, thus making more hot-cell space available for other experiments.

G-1

G. CORROSION STUDIES OF ZIRCONIUM

R. S. Peoples

This section of the report covers programs concerned with the corrosion behavior of zirconium-base cladding materials for the AEC Reactor Development Division and ORNL-supported studies of electrocladding zirconium for improved corrosion resistance.

Results obtained this month continue to indicate that Zircaloy 2 is an excellent cladding material. Considerable amounts of hydrogen, nitrogen, and oxygen can be tolerated before corrosion properties are impaired. Investigations have also been continued which support the Zirconium Corrosion Committee's program to develop new zirconium-base alloys.

A microbalance technique is being used to study the reaction of zirconium with water vapor at subatmospheric pressures.

As an aid to understanding the mechanism of zirconium corrosion, a study is being made of the reaction rates of several binary zirconium alloys with hydrogen at a temperature of about 750 F.

In studies for ORNL, platinum has been plated directly on zirconium without the use of an intermediate nickel layer. The platinum coating appears to be adherent; however, more extensive tests are necessary to establish this observation. Data were also obtained which indicate that zirconium does not pick up hydrogen during chemical polishing, while electroplating adds up to 20 ppm of hydrogen.

Corrosion of Zirconium and Zirconium Alloys

W. K. Boyd and R. S. Peoples

Corrosion studies to determine the effect of impurity and alloy additions on the corrosion properties of zirconium alloys are being continued in 750 F steam (1500 psi) and 680 and 600 F degassed water.

Samples of Zircaloy 2 representing seven different melts continue to show excellent resistance to 600 F degassed water. After more than 2-1/2 years of exposure, no indication of breakaway in corrosion rates has been noted. The samples evince a dark tarnish film and low weight gains [5 mg/(dm^2)(mo)].

G-2

The effect of hydrogen, nitrogen, and oxygen on the corrosion behavior of Zircaloy 2 is also being investigated. Alloy melts were prepared by gas impregnation and from master alloys added during melting. Corrosion data based on up to 238 days of exposure in 680 F water indicate the trends noted below.

(1) No effect of hydrogen additions in the range of from 500 to 1500 ppm

(2) Oxygen additions of more than 6000 ppm destroy corrosion resistance of Zircaloy 2

(3) Nitrogen additions above 200 ppm result in an increased rate of attack, while additions above 390 ppm destroy corrosion resistance.

Zirconium Corrosion Committee Program

The corrosion behavior of ternary and quaternary sponge-base alloys containing 0.25 to 0.50 w/o tin plus 0.04 to 0.45 w/o iron and/or nickel is being studied in 750 F steam (1500 psi) and 680 F degassed water. Alloys are being evaluated in each of two conditions of heat treatment, namely, (1) vacuum annealed 1 hr at 775 C and furnace cooled, and (2) held 1 hr at 900 C in vacuum and water quenched.

Corrosion results are summarized in Table G-1. These data, based on 172 days of exposure in 750 F steam and up to 224 days of exposure in 680 F water, indicate no significant effect of the above heat treatment on corrosion behavior. In general, the tin-nickel alloys exhibit somewhat better resistance to 750 F steam than do the tin alloys containing similar additions of iron. These studies are being continued.

Corrosion studies have also been continued on the series of ternary and quaternary alloys prepared at the BuMines for Zircaloy 3 evaluation. Each alloy was evaluated in both the vacuum-annealed and water-quenched conditions. A summary of the corrosion data obtained to date are presented in Table G-2. The quenched samples exposed to 680 F water continue to exhibit a tarnish film, while quenched samples exposed in 750 F steam and the vacuum-annealed samples (both 750 F steam and 680 F water) evince a light oxide pattern. In general, alloys containing 275 ppm aluminum are not so resistant as alloys containing lesser amounts of aluminum.

TABLE G-1. CORROSION OF ARC-MELTED (ROCKING-HEARTH METHOD) SPONGE ZIRCONIUM IN 750 F STEAM AND 680 F DEGASSED WATER

		Heated 1 Hr at 775 C in Vacuum and Furnace Cooled						Heated 1 Hr at 900 C in Vacuum and Water Quenched					
		750 F Steam			680 F Water			750 F Steam			680 F Water		
Heat	Alloy Content, w/o	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]
						Tin and Iron							
462	Unalloyed	1-1/2	768	W	210	211	W,F	10	1680	W	28	Lost weight	W,F
463	Unalloyed	1-1/2	839	W	210	210	W,F	10	1325	W	28	66	W,F
464	0.22Sn-0.28Fe	106	108	M	210	39	G	136	109	M	224	39	G
465	0.23Sn-0.30Fe	106	103	M	210	52	G	136	109	M	224	35	LM
466	0.22Sn-0.38Fe	134	100	M	210	40	G	136	104	M	224	53	LM
467	0.22Sn-0.39Fe	134	100	M	210	39	G	136	100	M	224	33	G
469	0.25Sn-0.27Fe	112	102	M	210	26	G	160	110	M	224	27	G
468	0.25Sn-0.28Fe	112	105	M	210	35	G	136	100	M	224	39	G
470	0.25Sn-0.33Fe	134	102	M	210	34	G	148	100	M	224	36	G
471	0.25Sn-0.34Fe	112	107	M	210	35	G	136	105	M	224	30	M
473	0.27Sn-0.25Fe	106	105	M	210	33	G	160	113	M	224	37	G
472	0.29Sn-0.25Fe	106	115	M	210	28	G	142	102	M	224	31	G
475	0.27Sn-0.28Fe	148	109	M	210	33	G	148	102	M	224	31	G
474	0.28Sn-0.34Fe	106	104	M	210	33	G	136	100	M	224	46	G
477	0.47Sn-0.44Fe	112	112	M	210	40	G	148	117	M	224	46	M
478	0.51Sn-0.47Fe	142	109	M	210	41	G	136	113	M	224	35	G
481	0.52Sn-0.47Fe	134	106	M	210	36	G	136	105	M	224	44	G
479	0.50Sn-0.50Fe	134	94	M	210	43	G	136	110	M	224	50	LM
476	0.49Sn-0.51Fe	124	100	M	210	39	G	148	100	M	224	45	M
480	0.51Sn-0.53Fe	124	102	M	210	36	G	136	104	M	224	46	LM
						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	172	84	M, S, F	210	49	G	170	83	M	196	51	G
490	0.18Sn-0.04Ni	172	86	M	210	46	G	170	70	M	196	51	G

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TABLE G-1. (Continued)

Heat	Alloy Content, w/o	Heated 1 Hr at 775 C in Vacuum and Furnace Cooled						Heated 1 Hr at 900 C in Vacuum and Water Quenched					
		750 F Steam			680 F Water			750 F Steam			680 F Water		
		Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]
						Tin and Nickel (Continued)							
492	0.13Sn-0.08Ni	172	75	M	210	39	G	158	Lost weight	S,F	196	33	G
493	0.18Sn-0.08Ni	148	68	M	210	36	G	170	73	M	196	35	G
495	0.17Sn-0.19Ni	172	76	M	210	36	G	170	68	M	196	38	G
494	0.19Sn-0.19Ni	172	72	M	210	33	G	170	75	M	196	32	G
496	0.21Sn-0.04Ni	172	67	M	210	49	G	170	73	M	196	33	G
497	0.22Sn-0.04Ni	172	86	M	210	48	G	170	73	M	196	29	G
498	0.22Sn-0.07Ni	172	79	M	210	44	G	158	Lost weight	S,F	196	38	G
499	0.22Sn-0.08Ni	172	78	M	210	43	G	170	74	M	196	27	G
501	0.21Sn-0.20Ni	172	84	M	210	46	G	170	96	M	196	32	G
500	0.22Sn-0.41Ni	172	76	M	210	36	G	170	92	M	196	35	G
502	0.27Sn-0.03Ni	172	76	M	210	54	G	158	Lost weight	S,F	196	32	G
503	0.27Sn-0.04Ni	172	87	M	210	49	G	170	83	S,F	196	39	G
505	0.27Sn-0.08Ni	172	86	M	210	51	G	170	45	S,F	196	35	G
504	0.28Sn-0.08Ni	172	87	M	210	49	G	170	81	M	196	33	G
506	0.26Sn-0.19Ni	172	89	M	210	47	G	170	81	M	196	39	G
507	0.25Sn-0.20Ni	172	109	M	210	36	G	170	86	M	196	48	G
508	0.56Sn-0.04Ni	172	67	M	210	52	G	170	90	M	196	28	G
509	0.56Sn-0.04Ni	172	82	M	210	42	G	170	87	M	196	33	G
510	0.54Sn-0.07Ni	172	82	M	210	39	G	170	86	M	196	44	G
511	0.56Sn-0.07Ni	172	94	M	210	41	G	170	89	M	196	34	G
513	0.51Sn-0.18Ni	172	93	M	210	47	G	170	101	M	196	35	G
512	0.54Sn-0.20Ni	172	92	M	210	43	G	170	87	M	196	36	G
						Tin, Iron, and Nickel							
483	0.51Sn-0.16Fe-0.090Ni	124	104	M	210	55	G	136	148	M	224	88	M
482	0.49Sn-0.23Fe-0.097Ni	148	110	M	210	42	G	136	105	M	224	39	G

TABLE G-1 (Continued)

Heat	Alloy Content, w/o	Heated 1 Hr at 775 C in Vacuum and Furnace Cooled						Heated 1 Hr at 900 C in Vacuum and Water Quenched					
		750 F Steam			680 F Water			750 F Steam			680 F Water		
		Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]
						Tin, Iron, and Nickel (Continued)							
485	0.47Sn-0.27Fe-0.13Ni	134	102	M	210	34	G	136	107	M	224	65	LM
484	0.48Sn-0.31Fe-0.13Ni	134	102	M	210	31	G	136	104	M	224	42	G
486	0.48Sn-0.38Fe-0.16Ni	148	106	M	210	39	G	130	107	M	224	51	G
487	0.48Sn-0.44Fe-0.18Ni	148	116	M	210	34	G	130	107	M	224	55	LM

(a) Key to remarks

G = Black tarnish film (good material)
M = Milky
LM = Light milky
W = White oxide
F = Flaking (heavy oxide)
S = Segregation (areas of good and poor material).

G-6

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

Heat	Alloy Content					Vacuum Annealed 2 Hr at 750 F Steam		
	Sn, w/o	Fe, w/o	Ni, w/o	N, ppm	Al, ppm	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]
996	0.24	0.25		44	58	141	62	M,WP
974	0.25	0.25		48	63	141	65	M,WP
997	0.26	0.25		33	46	141	66	M,WP
984	0.26	0.25		47	68	141	63	M,WP
1001	0.26	0.27		55	62	141	103	M,WP
1003	0.48	0.41		219	43	135	120	M,WP
975	0.49	0.36		57	45	141	69	M,WP
1000	0.49	0.38		43	50	141	78	M,WP
983	0.50	0.39		53	70	141	69	M,WP
998	0.51	0.38		48	47	141	77	M,WP
990	0.48	0.25	0.23	47	56	141	70	M,WP
985	0.49	0.20	--	47	64	141	66	M,WP
1007	0.50	0.21	0.19	50	51	135	110	M,WP
999	0.53	0.20	0.18	64	46	141	76	M,WP
995	0.65	0.22	0.19	38	57	141	78	M,WP
1024	0.25	0.25		63	68	141	89	M,WP
1026	0.25	0.25		63	67	141	53	M,WP
1030	0.50	0.44		58	47	141	98	M,WP
1028	0.49	0.38		71	38	141	88	M,WP
1014	0.25	0.25		60	145	141	110	M,WP
1012	0.25	0.25		50	179	141	109	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	141	114	M,WP
1011	0.50	0.39		62	275	135	123	M,WP
1025	0.50	0.21	0.19	65	62	141	73	M,WP
1023	0.53	0.22	0.20	63	60	141	87	M,WP
1017	0.49	0.21	0.19	53	135	141	97	M,WP
1016	0.51	0.21	0.18	48	154	135	107	M,WP
1015	0.50	0.21	0.19	51	265	141	142	M,WP

(a) Key to remarks:

G = Black tarnish film (good material)
M = Milky
WP = White oxide pattern
F = Flaking (heavy oxide)
S = Slight segregation (areas of good and poor material).

G-7

AT THE BUREAU OF MINES FOR ZIRCALOY 3 EVALUATION

775 C and Furnace Cooled			Heated 2 Hr at 900 C in Vacuum and Water Quenched					
680 F Water			750 F Steam			680 F Water		
Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]	Exposure Time, days	Weight Gain, mg/dm^2	Remarks[a]
182	43	G, WP	129	76	M, WP	168	35	G
182	47	G, WP	129	77	M, WP	168	33	G
182	45	W, P	129	73	M, WP	168	33	G
182	46	G, WP	129	78	M, WP	168	36	G
182	56	G, WP	129	70	M, WP	168	37	G
182	74	M, WP	129	127	M, WP, S	168	46	G
182	44	G, WP	129	74	M, WP	168	31	G
182	45	G, WP	129	81	M, WP	168	38	G
182	47	G, WP	129	78	M, WP	168	32	G
182	42	G, WP	129	73	M, WP	168	33	G
182	45	G, WP	129	74	M, WP	168	35	G
182	44	G, WP	129	72	M, WP	168	41	G
182	86	M, WP	129	103	M, WP	168	43	G
182	43	G, WP	129	80	M, WP	168	37	G
182	43	G, WP	129	73	M, WP	168	35	G
182	58	M, WP	129	97	M, WP, S	168	45	G
182	47	M, WP	129	83	M, WP, S	168	35	G
182	49	M, WP	129	86	M, WP	168	39	S
182	46	M, WP	129	86	M, WP	168	34	G
182	59	G, WP	129	96	M, WP	168	37	G
182	64	G, WP	123	112	M, WP	168	34	G
182	Lost weight	W, F	105	108	M, WP	168	52	G
182	81	M, WP	105	107	M, WP	168	35	G
182	63	M, WP	105	112	M, WP	168	30	G
182	77	M, WP	93	115	M, WP	168	37	G
182	47	G, WP	129	97	M, WP	168	33	G
182	47	G, WP	129	105	M, WP	168	36	G
182	57	G, WP	105	105	M, WP	168	39	G
182	61	G, WP	93	98	M, WP	168	39	G
182	79	M, WP	93	108	M, WP	168	56	G

G-8

Zirconium-Boron Alloys

Zircaloy 2 powder compacts containing 1.35, 6.50, and 8.32 w/o boron are being evaluated in 680 F degassed water. After 3 days of exposure, the base and 6.50 and 8.32 w/o boron compacts were covered with powdery oxide coating, while the compact containing 1.33 w/o enriched boron exhibited only a light oxide pattern. Addition powder compacts are being evaluated.

Compositional Factors Affecting Corrosion Properties of Zirconium

W. K. Boyd and D. J. Maykuth

The corrosion behavior of sponge-base zirconium-tin-iron-nickel alloys containing additions of from 70 to 300 ppm nitrogen or 200 ppm aluminum, or 100 ppm titanium is being determined in 750 F steam and 680 F degassed water. Corrosion data based on 65 and 81 days of exposure to 750 F steam and 680 F water, respectively, are summarized in Table G-3. Although no failures (white corrosion product) have been noted to date, raising the nitrogen content from 70 to 300 ppm results in an increased rate of attack. Alloys containing 200 ppm aluminum also show a higher rate of attack in 750 F steam than do alloys containing no aluminum additions.

Effect of Heat Treatment

Extended exposure data (170 days in 750 F steam) continue to show that the size and distribution of the intermetallic compound particles affect the corrosion behavior of zirconium-iron and zirconium-nickel alloys. A structure containing a fine dispersion of compound particles in an alpha matrix is more resistant than one exhibiting large colonies of particles separated by platelets of alpha zirconium.

Mechanism of Zirconium Corrosion

C. M. Schwartz and D. A. Vaughan

As an aid to the understanding of the mechanism of zirconium corrosion, an investigation of the role of hydrogen in high-temperature water corrosion is being made. Previous studies have indicated that corrosion rate varies with hydrogen reaction rate for samples of the same nominal

TABLE G-3. CORROSION OF SPONGE-BASE ZIRCONIUM ALLOYS IN 750 F STEAM AND 680 F DEGASSED WATER

Heat	Alloy Content, w/o	750 F Steam: Exposure Time, days	750 F Steam: Weight Gain, mg/dm^2	750 F Steam: Remarks[a]	680 F Water: Exposure Time, days	680 F Water: Weight Gain, mg/dm^2	680 F Water: Remarks[a]
	Vacuum Annealed 1 Hr at 775 C and Furnace Cooled						
556	0.05Fe-0.002Ni-0.007N	65	55	M	81	24	G
585	0.05Fe-0.002Ni-0.007N	65	59	M	81	20	G
557	0.05Fe-0.15Ni-0.007N	65	42	G	81	26	G
558	0.05Fe-0.15Ni-0.015N	65	33	G	81	30	G
559	0.05Fe-0.15Ni-0.0225N	65	42	G	81	38	G
560	0.05Fe-0.15Ni-0.030N	65	54	G	81	47	G
563	0.05Fe-0.15Ni-0.5Sn-0.007N	65	35	G	81	24	G
564	0.05Fe-0.15Ni-0.5Sn-0.015N	65	36	G	81	26	G
565	0.05Fe-0.15Ni-0.5Sn-0.0225N	65	48	G	81	21	G
566	0.05Fe-0.15N-0.5Sn-0.030N	65	49	G	81	21	G
567	0.25Fe-0.25Sn-0.007N	65	31	G	81	18	G
568	0.25Fe-0.25Sn-0.015N	65	33	G	81	20	G
569	0.25Fe-0.25Sn-0.225N	65	40	G	81	23	G
570	0.25Fe-0.25Sn-0.030N	65	44	G	81	35	G
573	0.40Fe-0.50Sn-0.007N	65	37	G	81	20	G
574	0.40Fe-0.50Sn-0.015N	65	33	G	81	22	G
575	0.40Fe-0.50Sn-0.0225N	65	51	G	81	23	G
576	0.40Fe-0.50Sn-0.030N	65	55	G	81	20	G
579	0.20Fe-0.20Ni-0.50Sn-0.007N	65	37	G	81	22	G
580	0.20Fe-0.20Ni-0.50Sn-0.015N	65	40	G	81	20	G
581	0.20Fe-0.20Ni-0.50Sn-0.0225N	65	52	G	81	25	G
582	0.20Fe-0.20Ni-0.50Sn-0.030N	65	56	G	81	24	G
561	0.05Fe-0.15Ni-0.007N-0.02Al	65	41	G	81	35	G
571	0.25Fe-0.25Sn-0.007N-0.02Al	65	53	G	81	37	G
577	0.40Fe-0.50Sn-0.007N-0.02Al	65	61	G	81	23	G
583	0.20Fe-0.20Ni-0.50Sn-0.007N-0.02Al	65	44	G	81	19	G
562	0.05Fe-0.15Ni-0.007N-0.01Ti	65	33	G	81	23	G
572	0.25Fe-0.25Sn-0.007N-0.01Ti	65	50	G	81	27	G
578	0.40Fe-0.50Sn-0.007N-0.01Ti	65	36	G	81	21	G
584	0.20Fe-0.20Ni-0.50Sn-0.007N-0.01Ti	65	40	G	81	18	G

(a) Key to remarks:
G - Black tarnish film (good material)
M - Milky

composition, and that when hydrogen reacts with zirconium the surface of the metal is damaged, causing an increase in corrosion rate.

During the past month, a number of binary alloys of zirconium were selected for further study of their reaction rates with hydrogen at or slightly above 750 F. Two of the alloys selected (2-1/2 w/o tin and Zircaloy 2) are available. The others (0.1 iron, 0.02 nitrogen, 0.05 titanium, and sponge zirconium) will need to be fabricated into the shape desirable for reaction-rate studies. The reaction-rate studies on the two available alloys will be started during the next period.

Zirconium-Gas Reactions

M. W. Mallett and W. M. Albrecht

Zirconium-Water Vapor

As part of a program on the water corrosion of zirconium, a study is being made of the reaction of zirconium with water vapor at subatmospheric pressures.

A microbalance technique for the continuous measurement of weight gain will be used to determine reaction rates. The vacuum microbalance for use in this study was received during the month and is currently being incorporated with necessary auxiliary equipment. As soon as the apparatus is complete and the operation of the microbalance proves satisfactory, initial rate data will be obtained for the reaction of zirconium with water vapor.

Electrocladding of Zirconium With Platinum Metals

A. B. Tripler, Jr., J. G. Beach, and C. L. Faust

In studies for ORNL, platinum has been plated directly on zirconium without the use of an intermediate nickel layer. The same fluoride activation treatment that is required for successful nickel plating on zirconium worked well for platinum plating. The platinum coating appears to be adherent, but more exhaustive tests, such as a thermal cycling test, will be made. A zirconium specimen coated directly with approximately 0.5 mil platinum showed negligible weight loss after 5 days of exposure to a fluoride solution at 70 F.

G-11 and G-12

The platinum-over-nickel coatings on zirconium remained adherent during thermal cycling between room temperature and 300 C for 100 cycles.

Zirconium specimens, treated in various ways, were analyzed for hydrogen content. In each case, the starting material was centerless-ground zirconium rod prepared from crystal-bar stock. Table G-4 gives the results of the analyses.

It is evident from the above data that there is about 30 ppm hydrogen in the zirconium specimens to begin with. Chemical polishing does not add to the amount. Plating adds up to 20 ppm, depending on the combination of treatments. It is interesting to note that where platinum was deposited directly on zirconium (Specimen 60A), less hydrogen was added than where nickel was used as an undercoat.

Both loop and bomb specimens are being readied for in-pile testing. These specimens will all have the nickel undercoating because there has not been sufficient preliminary testing, as yet, on specimens coated with platinum alone.

TABLE G-4. RESULTS OF HYDROGEN ANALYSES ON ZIRCONIUM SPECIMENS WITH VARIOUS SURFACE TREATMENTS AND/OR COATINGS

Specimen	Treatment	Hydrogen Content, ppm
50A-1	C. G.[a]	31.4
50A-2	C. G.[a], surface abraded to remove any extraneous material	29.0
50B	C. G.[a], chemically polished	30.6
46A	C. G.[a], chemically polished, 3-min activation (100 F), 1 mil nickel, degassed at 400 F, bonded at 1300 F, 0.5 mil platinum	43.9
49A	Same as specimen 46A except no bonding treatment	50.8
60A	C. G.[a], 5-min activation (90 F), 0.5 mil platinum	39.4

(a) C. G. = centerless ground.

H-1

H. REACTOR MATERIALS DEVELOPMENT

H. A. Saller

A number of separate programs on the development of materials for general reactor application are sponsored by the Reactor Development Division of the Atomic Energy Commission.

The study of transformation kinetics and age-hardening characteristics of several zirconium alloys has continued.

Further studies on enamels for uranium showed cracking to be a problem. Apparently, an enamel with an extremely high coefficient of expansion is required. The enameling of zirconium and niobium was continued.

Sandwich-type diffusion samples of UO_2 and zirconium are still under test at temperatures from 950-1200 F. Examination of a sample heated at 1600 F for 68 hr showed a two-phase region (alpha uranium, alpha zirconium) adjacent to the UO_2. Another two-phase region (alpha zirconium, epsilon) occurred further into the zirconium.

Studies of Zirconium Heat Treatment

H. A. Robinson, M. W. Mote, and P. D. Frost

The current investigation of zirconium-base alloys is to evaluate heat treatability and transformation kinetics. Alloys containing enough beta-stabilizing elements so that some beta can be retained on quenching have been age hardened to strength[illegible] about 200,000 psi. However, elongation values of about 1 per cent at t[illegible] strength level are unacceptably low. Although the elongation values seemed to be dependent upon the alloy addition, other factors, e.g., hydrogen contamination and the choice of heat treatment, may have prevented the alloys studied to date from developing optimum properties. The present research studies should determine the effects of hydrogen content and of variations in heat treatment on the high-strength properties of zirconium-base alloys.

During the month of December, the preliminary steps for all newly outlined phases of the research were continued. Aging studies of the two large ingots of zirconium-2 w/o tin plus 5 w/o molybdenum or niobium alloys have been started. Hardness curves, however, have not been completed, so heat treatments for tensile specimens of these two alloys cannot yet be selected. Additional material from these two alloys, to be used for

the study of the effects of hydrogen, has been vacuum annealed or hydrogenated and subsequently rolled to sheet. Selection of heat treatments for this material will also be made when the aging study is completed. Melting and rolling of the experimental alloy buttons of the ternary alloys have been completed. These buttons cover a range of alloy additions as noted below.

Alloying Element, w/o
Aluminum, 0 to 3
Tin, 0 to 4
Molybdenum, 1 to 5
Niobium, 3 to 11

The age-hardening study for these alloys will be similar to the study being made on the alloys from the large ingots; the conditions will include three solution temperatures, three aging temperatures, and aging times up to at least 100 hr.

Enameling of Special Metals

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

Porcelain-enamel coatings for various metals of special interest to the AEC are being investigated. Such coatings might protect the metals against corrosion, function as heat or metal-diffusion barriers, or serve to retain fission fragments.

In the enameling of uranium, the major problem encountered has been cracking of the coating during cooling, apparently due to differences in thermal expansion between the metal and the glass coating. This month, a series of enamels having calculated linear-expansion coefficients ranging from 15.4 to 19.3 x 10^{-6} per deg C was applied to uranium plates. All of the enamels cracked on cooling. However, as the expansion coefficient increased, the amount of cracking decreased. This test indicated that an enamel having an extremely high expansion coefficient might solve the cracking problem.

Last month, it was noted that a thin, hard, adherent coating of oxide formed on zirconium when the metal was heated for a long period in the presence of a small amount of oxygen. This month, an attempt was made to form such a coating on zirconium which had been polished by fine emery cloth. A nonuniform oxide coating formed. The oxide appeared to form more rapidly at scratch marks left by the emery than in other areas. To improve the uniformity of the coating, a higher degree of polish appears to be required. An attempt will be made to produce such a coating by starting with highly polished zirconium.

H-3

Bismuth-uranium alloys containing 0.5, 0.75, and 1.00 w/o uranium were prepared for use as corrodents in testing the corrosion resistance of enameled zirconium at 600 C.

An enamel having the ability to resist molten fluoride salts at elevated temperature might be extremely useful. Ordinarily, glass has poor resistance to fluorides. A glass saturated with fluorine, however, might have improved resistance to fluorides. Therefore, an effort will be made to produce an enamel coating for zirconium which is saturated with fluorine. This glass will then be tested for its resistance to a mixture consisting of the LiF-NaF-KF eutectic, at about 600 C.

Preparatory to the development of refractory coatings for niobium, a literature search was made for information on high-temperature enamels, particularly those which have been applied to molybdenum. Molybdenum has an expansion coefficient close to that of niobium. Hence, molybdenum-enamel compositions are of interest for use as a basis for the formulation of enamels for niobium.

Kinetics of the Zirconium-Uranium Dioxide Reaction

M. W. Mallett, J. W. Droege, A. F. Gerds, and A. W. Lemmon

A study of the solid-solid reaction between zirconium and uranium dioxide is being made. Information obtained can be used to estimate the compatibility of these materials in reactors.

The reaction is being studied by use of sandwich-type elements. In these elements, a plane surface of zirconium metal is in intimate contact with massive high-fired UO_2. Elements currently are being heated at 950, 1100, and 1200 F in autoclaves at a 5000-psi pressure. The elements being heated at 950 F have now been on test for a total of 62 days, those at 1100 F for 35 days, and those at 1200 F for 51 days. High-pressure leaks in the autoclave seals, which have been repaired, account for the lesser times at the two higher temperatures. It is planned to examine one of the 1200 F elements metallographically during January. The elements being heated at the two lower temperatures will be heated for at least 2 additional months before examining metallographically.

The cross section of an element, examined metallographically after heating under a 2400-psi dead load at 1600 F for 68 hr in argon and water quenching, contained a two-phase region in the zirconium adjacent to the UO_2. This region, about 0.007 cm thick, consisted of alpha uranium in an alpha zirconium matrix. Adjacent to this region and deeper into the zirconium was a second two-phase region about 0.006 cm thick, apparently consisting of the epsilon intermetallic phase and alpha zirconium. The phase

identifications were made on the basis of the appearance of the several phases under the microscope. In another program, phases of similar appearance previously were identified for reacted specimens by X-ray diffraction methods. Hardness measurements indicated that oxygen had penetrated into the zirconium from the UO_2 to a depth of about 0.04 to 0.05 cm (0.015 to 0.020 in.). A large sandwich-type element, heated in the same assembly, will be machined into layers, each 0.001 in. thick, beginning at the zirconium-UO_2 interface. Oxygen and uranium analyses will be obtained on each layer to determine the transfer of these elements from the UO_2 to the zirconium.

A thermodynamic analysis of the results of previous studies on this same system, as reported in BMI-1023 and BMI-1028, shows that there are minor discrepancies between the phase-diagram and reaction-rate work. The calculations indicate that at 1300 F the limiting solubility of oxygen in alpha zirconium should be about 13.2 a/o rather than at 28 a/o as predicted from the phase studies. Work to resolve these differences will continue.

I-1

I. PHYSICAL METALLURGY

H. A. Sailer

The Research Division of the Atomic Energy Commission supports a number of programs in the fields of metallurgy, ceramics, and gas-metal studies.

Studies of uranium compounds have continued. Both USi_2 and U_3Si appear to have favorable properties for reactor fuel. Studies of U_2Ti and UNi_2 are continuing.

The program on internal friction in uranium has been completed and a report will be prepared.

The study of intermediate or delta-phase alloys is now concerned with the uranium-titanium-molybdenum system.

A series of zirconium samples having three different grain sizes has been made up and will be tested in impact as part of the investigation of embrittlement.

High-temperature X-ray studies on iodide thorium containing some thorium oxide showed a change at 1425 C. Above 1500 C, both thorium oxide and face-centered thorium had disappeared. The high-temperature phase is being identified.

All the binary niobium alloys have been tested for oxidation. None has shown a drastic change in the oxidation mechanism.

The other programs show progress, but have no major results to report.

Properties of Uranium Compounds

L. D. Loch, G. B. Engle, and M. J. Snyder

Research is being done on some of the less-familiar uranium compounds that are potential nuclear fuels. The investigation is limited to compounds with relatively high uranium contents and melting points. Current work consists of the preparation of compounds and the preliminary evaluation of their general chemical and thermal stability.

1-2

Preliminary chemical- and thermal-stability tests on U_3Si and UNi_2 were completed and reported previously. This month, USi_2 was prepared by arc melting, and preliminary tests were completed on U_2Ti and USi_2. Table 1-1 summarizes data obtained on the intermetallic compounds tested to date. Preliminary tests on US and UN are incomplete because of difficulty in fabricating dense samples.

TABLE 1-1. SUMMARY OF DATA ON URANIUM INTERMETALLIC COMPOUNDS

Compound	Uranium Content[a], g per cm³	Approximate Melting Point, C	Thermal-Stability Tests[b], weight change in per cent		
			Vacuum	Air	H_2
U_3Si	14.39	930[c]	-1.6	+18.0	+0.04
Alpha USi_2	7.27	1700	0.00	+4.0 +2.5 (620 C)	+0.01
U_2Ti	-	890[c]	0.00	+22.0	16.0
UNi_2	7.91	985[c]	-0.10	+18.0	+1.0

(a) Based on X-ray density.
(b) One-hr tests at 500 C except where noted.
(c) Decomposes.

All of the compounds listed in Table 1-1 reacted to some degree with concentrated HCl, HNO_3, and H_2SO_4. None reacted with 1 N NaOH, or showed any weight change after 1 hr in boiling water.

The results of the preliminary tests indicate that U_3Si and USi_2 may make good nuclear fuels. Although USi_2 has a lower uranium content than some of the other compounds originally considered, its resistance in air at 620 C should make it of value where oxidation resistance is needed. Both U_2Ti and UNi_2 are still of some interest because of their corrosion resistance to boiling water.

Future work will be concentrated on U_3Si and USi_2. Samples will be prepared for more severe corrosion tests in common reactor coolants. The density of U_2Ti will be determined to permit calculation of its uranium content per unit volume. Work will be continued on making dense US and UN samples, and preliminary chemical and thermal tests will be run on them.

1-3

Development of Uranium-Titanium Alloys

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

The potential utility of uranium-titanium alloys is being studied by an evaluation of several mechanical properties. Of four compositions tested, the 50 a/o titanium (16.8 w/o titanium) fully annealed alloy has been found to be inherently too brittle for machining and tensile testing. Further work will thus be confined to compositions of 5, 15, and 25 a/o titanium (1.65, 3.3, and 6.3 w/o titanium, respectively).

Hot-Hardness Tests

Elevated-temperature tests were run in vacuo on water-quenched, air-cooled, and slow-cooled alloys.

The 15, 25, and 50 a/o titanium alloys cracked and broke upon quenching. Hardness tests were run on the larger pieces. Only the 5 and 15 a/o titanium alloys yielded sufficient data to enable any conclusions to be drawn. These data are listed in Table 1-2. Both alloys have low softening rates up to the equicohesive temperature, 460 C for the 5 a/o titanium alloy and greater than 500 C for the 15 a/o titanium alloy.

TABLE 1-2. HOT-HARDNESS DATA FOR HEAT-TREATED URANIUM-TITANIUM ALLOYS

Temperature, C	Hardness[a], kg/mm²								
	5 a/o Titanium			15 a/o Titanium			25 a/o Titanium		
	W.Q.	A.C.	S.C.	W.Q.	A.C.	S.C.	W.Q.	A.C.	S.C.
25	372	401	350	536	449	309	422	419	319
146	279	303	264	484	-	292	-	421	305
200	261	316	215	374	-	246	469	370	273
300	250	268	175	400	-	215	-	260	236
400	161	193	122	366	-	186	-	316	119
500	153	137	57	321	371	128	365	257	152
600	20	73	27.4	-	217	77	212	146	67.3
700	5.7	5.3	5.5	-	38.5	35.4	73	44	27.4

(a) W.Q. = Water quenched,
A.C. = Air cooled,
S.C. = Slow cooled (5 C per hr),

The air-cooled samples showed a greater rate of hardness decrease with temperature. Equicohesive temperature values are 360 C for 5 a/o titanium and 480 C for the 25 a/o titanium alloy. The 15 a/o alloy oxidized slightly, and reliable hardness data were not obtained.

Results of tests performed on slow-cooled alloys (5 C per hr) showed a higher rate of softening than either air-cooled or water-quenched alloys. Values of the equicohesive temperatures were 370, 440, and 460 C for 5, 15, and 25 a/o titanium, respectively.

Tensile Tests

During the attempted machining of tensile bars of the 50 a/o alloy, 16 out of 18 specimens fractured. One of the remaining two was tested at 25 C and failed prematurely in the grips at a stress of 36,000 psi. However, a reliable value of 26.4×10^6 psi for the elastic modulus was obtained. The second specimen will be tested at 500 C.

Future work will include tensile tests at temperatures above and below T_c in order to determine ductility, strength, and Young's modulus.

The effect of microstructure will be evaluated along with mechanical property tests.

Investigation of the Tensile Transition in Alpha Uranium

L. L. Marsh and G. K. Manning

The tensile transition in alpha uranium is being studied to learn more about the phenomena involved in producing the changes in mechanical properties in the vicinity of room temperature.

The study of metallographic specimens strained in tension to various plastic strains at minus 40 F, 75 F, and 200 F is being continued. Both optical and electron microscopy techniques are being employed to study the surface deformation markings. Twinning appears to be the primary mechanism for plastic deformation at minus 40 F and 75 F. At 75 F, many areas of the specimen contain regions which are suggestive of kinking, particularly at relatively large values of plastic strain. Since the kinking process is considered normally to arise from a slip process, specimens were prepared for examination with the electron microscope. Surface tracings have been observed with the electron microscope which have been identified as fine slip, not visible with light-microscopy techniques.

The surface deformation markings observed after straining at 200 F consisted of some twinning, regions which have been tentatively identified as heavily kinked, and considerable slip. However, because of the uncertainty in the identification of kinking as opposed to twinning, large grains of uranium will be obtained by annealing in the gamma-phase region at 1800 F. X-ray diffraction techniques will then be employed to determine if more positive evidence of kinking can be obtained.

Internal Friction of Uranium

R. E. Maringer, L. L. Marsh, and G. K. Manning

The internal friction of uranium is being studied to learn more about the phenomena which have been ascribed to the stress-induced motion of twin boundaries.

The internal friction of alpha- and beta-annealed biscuit uranium has been observed as a function of time after the rapid application or removal of an elastic load. In all cases, the internal friction decreases linearly with the logarithm of time, indicating a reversible relaxation process.

The internal friction of beta-annealed biscuit uranium during elastic tensile elongation has been studied at different frequencies and the damping has been found to increase as the period of the oscillation increases. This is interpreted as the result of a greater amount of twin-boundary relaxation which occurs per cycle as the period increases and proves that the damping is not strictly anelastic in nature.

Twin-boundary motion has been observed under the microscope and has been photographed. Experimental work on the project has thus been completed and a topical report is being prepared which will describe the results in detail.

Intermediate or Delta-Phase Alloys

F. A. Rough, A. A. Bauer, J. R. Lulay, and J. R. Doig

The system uranium-titanium-molybdenum is under investigation to determine the relationships between the intermediate phases and the extent of the solubility of these phases.

Work was initiated this month. A study of the kinetics of transformation of gamma uranium to alpha uranium plus delta as a function of titanium

content has been started. Samples were prepared and annealed at 900 C for two hours, then water quenched. Further preparations are now complete for transforming these samples at 500 C for various periods of time.

X-ray results on samples treated at 700 C in the uranium-titanium-molybdenum system indicate an extensive three-phase region exists consisting of gamma uranium, molybdenum, and U_2Ti. Additional data will be obtained to further define this phase region.

Study of Bonding Fundamentals

J. E. Reynolds, J. B. Melehan, H. R. Ogden, and R. I. Jaffee

The simulated single-asperity bonding studies on gold and the nucleation-and-growth studies of the bonding operation using pure silver specimens pressed in closed dies have been continued.

The torsional balance has now been installed in the bell-jar vacuum system. Magnetic coupling through the bell-jar wall is being used to activate the torsional member for bonding and breaking. Thirty needle-point bonding tests are to be conducted during the next period to check reproducibility.

The electron microscope is being utilized in an effort to identify bonded and unbonded areas in a silver specimen from the nucleation-and-growth studies. The surface and profiles of the gold needles also are being studied with the electron microscope.

A Fundamental Investigation of Embrittlement in Zirconium

C. M. Schwartz and A. P. Young

The objective of this investigation is to study the slip-line and twinning structure of deformed zirconium in order to determine why hydrogen lowers the impact strength of slow-cooled samples as compared with that of quenched samples.

The major difference in the deformation structure between tensile samples strained at slow velocity and at impact velocity is the much greater incidence of twinning in the samples strained at impact velocity. During this investigation two sets of samples with quite different grain sizes were inadvertently prepared. The incidence of twinning in two samples of different grain size strained at impact velocity was considerably lower in the finer grain sample. It was expected then that the hydrogen

embrittlement might be affected by grain size. To test this theory, a third set of samples with larger grain size was prepared. The effect of hydrogen on the unnotched tensile-impact properties for two different grain sizes is shown in Table 1-3. In the quenched samples, the hydrogen is retained in solid solution. In the slow-cooled samples the hydrogen is precipitated in the form of hydride platelets. The effect of grain size on the embrittlement

TABLE 1-3. UNNOTCHED TENSILE-IMPACT PROPERTIES OF HIGH-PURITY ZIRCONIUM AND ZIRCONIUM-HYDROGEN ALLOYS AT ROOM TEMPERATURE

Grain Size	Condition	Elongation in 1/2 in., per cent	Reduction in Area, per cent	Breaking Energy, ft-lb
0.05 mm	Vacuum degassed, quenched from 550 C	62	61	26.5
0.05 mm	100 ppm hydrogen, quenched from 500 C	50	30	25.0
0.05 mm	100 ppm hydrogen, furnace cooled from 550 C	46	26	20.0
0.30 mm	Vacuum degassed, quenched from 550 C	72	40	26.5
0.30 mm	100 ppm hydrogen, quenched from 550 C	50	39	22.0
0.30 mm	100 ppm hydrogen, furnace cooled from 550 C	31	19	11.5

in the slow-cooled samples, which contain the hydride, is evident from this data. The samples used in these tests were flat samples prepared from rolled sheet. The samples had 1/2-in. gage length and 0.250 by 0.100-in. cross section. The incidence of twinning versus slip may be affected in the flat samples by a preferred orientation of the grains in the annealed sheet. For this reason, and also because the elongation and reduction in area can be measured more accurately in round samples, the effect of grain size on hydrogen embrittlement will probably be investigated in round samples.

Examination of fractured samples under the light microscope reveals cracks along hydride needles, particularly where lens-shaped twins terminate on hydride needles. Photographs have been taken to document the evidence. A careful examination will be made in the electron microscope of the deformed surfaces with particular attention being paid to areas adjacent to the tips of lens-shaped twins to try to determine whether

there is some mode of stress relief which is inoperative when twins terminate on hydride needles.

Solid-State Studies of Thorium and Uranium Alloys

A. E. Austin and C. M. Schwartz

The thorium-rich end of the thorium-uranium phase diagram is being studied. The effect of oxygen contamination is being sought.

High-temperature X-ray diffraction studies were started with a sample of iodide thorium. The specimen had appreciable thorium oxide in addition to face-centered cubic thorium. Above a 1425 C apparent temperature, a new phase started to form. Above 1500 C, both thorium oxide and face-centered thorium had disappeared. The new phase has not been identified. Its stability at lower temperatures is being studied. It is planned to examine thorium-uranium alloys up to 5 w/o uranium. Emissivity measurements will be made to determine true specimen temperatures.

Oxidation-Resistant Niobium Alloys

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

A study is being conducted to determine the effectiveness of various alloying additions in increasing the oxidation and contamination resistance of niobium-base alloys.

Air-oxidation rates have been determined on alloys of niobium containing tungsten and vanadium in addition to the rates already established for alloys with titanium, zirconium, molybdenum, and chromium. The oxidation rates for the new alloys are given in Table I-4. Tungsten additions of 5 and 10 a/o were slightly beneficial, but the rate increased for 25 a/o tungsten. Additions of vanadium, particularly 5 and 10 a/o, increased the oxidation resistance of niobium. The degree of improvement was of the same order as that afforded by the addition of 5 a/o molybdenum. Thus, of the eight alloying systems studied so far, four alloys (titanium, vanadium, molybdenum, and chromium) have been found to improve the oxidation resistance of niobium. Titanium is the most beneficial of the four.

TABLE I-4. AIR-OXIDATION RATES FOR NIOBIUM-TUNGSTEN AND NIOBIUM-VANADIUM ALLOYS

Composition (Balance Niobium), w/o	VHN, kg/mm^2	Oxidation Rate, mg/(cm^2)/(hr), at Temperature, C		
		600	800	1000
1W	127	6.1	41	59
5W	165	3.1	12.3	54
10W	196	2.9	17	26
25W	299	8.3	31	94
1V	114	3.6	6.3	13.8
5V	160	0.9	1.4	4.6
10V	172	0.05	0.7	3.5
25V	194	0.2	6.1	193
100Nb	-	6.6	34	19.8

As shown in Table I-4, additional data have been obtained for the air oxidation of pure niobium exposed as control specimens with the alloys. The oxidation rate appears to be less at 1000 C than at 800 C; a series of tests has confirmed this result and the effect is being studied further. Oxidation tests on specimens of a different geometry will be conducted to establish a more accurate control rate.

Preparation of the 46 binary alloys by arc melting has been completed. A series of ternary alloys is now being prepared in the same manner as the series of binary alloys. The compositions of these alloys are tabulated below.

Niobium-12.5 a/o titanium-12.5 a/o chromium, zirconium, tantalum
Niobium-20 a/o titanium-5 a/o molybdenum, tungsten, vanadium
Niobium-23 a/o titanium-2 a/o iron, cobalt, nickel
Niobium-24 a/o titanium-1 a/o aluminum, silicon

Niobium-12.5 a/o chromium-12.5 a/o zirconium, tantalum
Niobium-20 a/o chromium-5 a/o molybdenum, tungsten, vanadium
Niobium-24 a/o chromium-1 a/o aluminum, silicon

Niobium-5 a/o molybdenum-5 a/o tungsten, vanadium
Niobium-5 a/o molybdenum-12.5 a/o tantalum
Niobium-5 a/o molybdenum-1 a/o aluminum, silicon.

1.10

In addition, three new binary alloys containing 35 a/o titanium, zirconium, or chromium are being prepared to extend the composition range for these systems. All of these alloys will be exposed to air-oxidation tests.

Two more gas-metal reactions have been conducted for pure niobium in the modified Sieverts apparatus. The oxidation rate of niobium in pure dry oxygen at 1100 C was determined as 0.839 ml/(cm^2)(min)[71.9 mg/(cm^2)(hr)]. This rate falls very close to the straight curve established by the 600, 800, and 1000 C data on an Arrhenius-type plot of the oxidation rate versus the inverse absolute temperature. The mode of oxidation may undergo a change between 1100 and 1200 C, since the 1200 C rate is much higher than expected from the 600 to 1100 C data.

The oxidation of pure niobium in dry air at 800 C was also measured in the Sieverts apparatus. The gas-solid reaction occurred in a parabolic manner according to the equation $x^2 = 0.446t$*. This is significantly different behavior from the dry oxygen-niobium reaction at 800 C, which occurs in a linear manner. This difference will be studied and evaluated more completely in the future.

Alloy oxidation tests in air are currently being conducted on binary alloys of niobium with tantalum, aluminum, and iron. These studies with other binaries and the ternary alloys listed above will continue. Studies of the reactions between pure niobium and oxygen and air will also continue.

Uranium-Hydrogen Reactions

M. W. Mallett and W. M. Albrecht

The reactions of uranium and uranium alloys with hydrogen are being investigated. The reactions are being made under the conditions that $p-p_o$ is a constant, where p is the system pressure and p_o is the dissociation pressure of the uranium hydride.

Equilibrium studies were made of a uranium-5 w/o chromium alloy with hydrogen in the range 240 to 430 C to determine the dissociation pressure, p_o, of the hydride. The data can be expressed by the equation:

$$\log p_o \text{ mm} = -\frac{3830}{T} + 8.24 \quad . \qquad (1)$$

This equation is being used to calculate values for p_o at lower temperatures where equilibrium is very slowly attained.

*x = ml per cm^2; t = minutes.

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Reaction rates of the alloy with hydrogen are given in Table I-5. All the data showed excellent agreement with the linear rate law after initial deviations. Additional rate data are being obtained.

TABLE I-5. REACTION RATES OF URANIUM-5 w/o CHROMIUM WITH HYDROGEN

Temperature, C	Linear Reaction Rate, ml STP hydrogen consumed per cm^2 of surface per sec		
	$P^{-}P_{H_2}$ = 70 mm	$P^{-}P_{H_2}$ = 150 mm	$P^{-}P_{H_2}$ = 430 mm
100	-	8.0×10^{-3}	-
150	-	9.9×10^{-3}	-
200	6.9×10^{-3}	-	-
275	-	-	1.3×10^{-2}
300	3.3×10^{-3}	-	-
350	-	1.9×10^{-3}	5.8×10^{-3}

Study of Hydrides

M. W. Mallett and M. J. Trzeciak

A fundamental study of hydrides is in progress and factors affecting the thermal stability of metal hydrides used in moderator and shielding applications are being evaluated. At present, variations in high-temperature N_H values of different zirconium alloy hydrides are being measured as a function of the various alloying constituents. (N_H = number of hydrogen atoms per $cm^3 \times 10^{-22}$.)

Determinations of N_H values of four zirconium alloy hydrides have been made in the temperature range 1700 to 2200 F. N_H values at 1000 to 1600 F for these alloys had been reported earlier.

The N_H values from 1000 to 2200 F for the four zirconium alloys are listed in Table I-6. N_H values for pure zirconium are also listed in the table for comparison. The data show that the addition of niobium to zirconium results in unusual behavior. That is, niobium, a monohydride former at room temperature, when added to zirconium causes a marked decrease in N_H above 1600 F. On the other hand, molybdenum, manganese, and chromium, which

are nonhydride formers at room temperature, have little or no adverse effect on N_H above 1600 F when added to zirconium.

TABLE I-6. ELEVATED-TEMPERATURE N_H VALUES OF HYDRIDED ZIRCONIUM ALLOYS

Temperature, F	N_H Values of Alloys of Zirconium Indicated Elements				
	Iodide Zirconium	2.3 a/o Niobium	2.3 a/o Molybdenum	2.3 a/o Chromium	2.3 a/o Manganese
1000	6.9	7.4	5.7	6.9	7.1
1200	6.5	6.9	5.5	6.5	6.7
1400	6.0	6.4	5.0	6.0	6.1
1500	5.8	6.0	4.8	5.8	5.4
1600	4.6	4.3	4.4	4.3	5.0
1700	3.8	2.9	4.2	3.9	3.4
1800	3.0	2.2	3.4	3.0	2.6
2000	1.8	1.2	1.9	1.4	1.4
2200	1.2	0.9	1.2	1.1	1.0

The zirconium-molybdenum system appears interesting in that below 1600 F the N_H value is substantially lower than that for pure zirconium. From about 1650 F to about 1950 F, the N_H value of the alloy exceeds that of pure zirconium. Above 1950 F the N_H values for the alloy are essentially the same as those for pure zirconium. This interesting system warrants further study. Consequently, another alloy of different concentration is being prepared for hydriding at the elevated temperatures.

RWD:CRT/all