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PROGRESS RELATING TO CIVILIAN APPLICATIONS DURING JULY, 1958

by

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A. DEVELOPMENTS FOR ZIRCONIUM-CLAD FUEL ELEMENTS

F. R. Shober

Thermal-conductivity measurements of uranium and UO_2 are being made as part of two programs to determine the effect of irradiation on these two materials. Data obtained from creep tests on 15 per cent cold-worked Zircaloy-2 at 290, 345, and 400 C indicate that the 10,000-hr creep strengths at these respective temperatures are greater than 35,000, 25,000, and 15,000 psi. Tests have been in progress approximately 6000 hr.

Corrosion tests in 300 C water and hot-hardness tests of high-strength zirconium alloys containing molybdenum, niobium, and tin have been used as the basis of selection for seven alloys which appear superior to Zircaloy-2 in both respects. A study directed toward development of sink-float density measurements as a method of identifying factors affecting irradiation-induced volume changes in graphite is under way. Research has been initiated on the preparation of molybdenum single crystals for irradiation-damage studies.

Thermal Conductivity of Uranium and UO_2

C. F. Lucks and H. W. Deem

The effect of irradiation on the thermal and electrical conductivities of uranium and on the thermal conductivity of uranium oxide is being studied.

Uranium

Thermal-conductivity measurements on clad specimens are being continued. Data will be reported as soon as comparison measurements are completed.

Uranium Oxide

An apparatus for making thermal-conductivity measurements on UO_2, both before and after irradiation, has been completed. A steady-state absolute method is being used. Briefly, accurately measured power is introduced into the top part of the specimen and the heat flows through the specimen into a heat sink. Compensated thermocouples at known positions measure the thermal gradients. The thermal conductivity of the specimen will be calculated from the heat flow, cross-sectional area, and the thermal gradients. Guarding to prevent stray heat flows has been provided.

Calibrating and establishing the reliability of the apparatus has continued. Results on the titanium-alloy standard are given in Table A-1. Although the experimental results are in reasonable agreement with literature values, the apparatus shows considerable sensitivity to small temperature unbalances between the guard and specimen heater. However, careful control of guard temperature is effective.

TABLE A-1. THERMAL CONDUCTIVITY OF TITANIUM-6 w/o ALUMINUM-4 w/o VANADIUM

Temperature, C	Thermal Conductivity, w/(cm)(C)		Difference, per cent
	Observed	Literature	
135	0.070	0.081	-11
150	0.076	0.083	-8
164	0.085	0.084	+1
184	0.081	0.087	-7
298	0.084	0.102	-18
325	0.098	0.106	-8
409	0.122	0.117	+4
453	0.116	0.123	-6

Mechanical Properties of Zirconium Alloys

F. R. Shober and J. A. VanEcho

The creep strength of 15 per cent cold-worked Zircaloy-2 is being determined and evaluated as a part of a program to provide design data for reactor components in the 290 to 400 C temperature range. Creep data are being obtained from two types of tests: short-time stress-rupture and long-time creep tests (about 10,000 hr). The time for initiation of third-stage creep and the total deformation associated with it are of special interest. The ability of cold-worked zirconium to retain the additional strength achieved by cold working under sustained loads at elevated temperatures is being studied.

Twelve creep units are in use at the present time with the majority being used for long-time creep tests. Seven of these tests have been in progress over 5000 hr, and the results to date indicate that they will require at least 10,000 hr for completion.

Based on data from tests at 290 C, both those completed and those in progress, it appears that 15 per cent cold-worked Zircaloy-2 will sustain stresses of 35,000 psi or less for periods exceeding 10,000 hr. The minimum creep rates obtained for tests in this stress range are less than 0.00001 per cent per hr. Test specimens loaded in the stress range from 40,000 to 45,000 psi at 290 C failed between approximately 1700 and 0.1 hr, respectively. At 345 C, stresses capable of being sustained by the specimen for periods of 10,000 hr are 25,000 psi or less. Loads of 40,000 and 30,000 psi produced failure in 0.5 and 4200 hr, respectively. Insufficient results have been obtained at 400 C to make a similar comparison, but it appears that a 10,000-hr rupture life will be attained by stresses below approximately 15,000 psi.

A-3

Development of High-Strength Corrosion-Resistant Zirconium Alloys

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

The development of a reactor to operate at elevated temperatures is dependent upon the development of cladding and structural materials capable of withstanding these temperatures. Zircaloy-2 is an example of a cladding material developed by such alloy studies. If stronger corrosion-resistant zirconium alloys (stronger than Zircaloy-2) can be developed for use as cladding, the use of metallic uranium fuel elements at higher temperatures will be feasible. The expansion coefficients, nuclear properties, and thermal conductivities of these alloys should be similar to Zircaloy-2.

Nine series of alloys were prepared. They consist of ternary zirconium-base alloys containing 2.0, 3.0, and 4.0 w/o tin plus 0 to 2.0 w/o molybdenum, ternaries containing 2.0, 3.0, and 4.0 w/o tin plus 0 to 3.0 w/o niobium, and quaternary alloys containing 2.0, 3.0, and 4.0 w/o tin plus 0.5 to 2.0 w/o molybdenum and 0 to 3.0 w/o niobium. The above-mentioned alloys were screened by corrosion testing for 1000 hr in high-purity 300 C water and by room-temperature hardness (DPH) measurements. Limiting parameters of a minimum room-temperature hardness of 250 DPH (this gives a 0.2 per cent offset yield strength of approximately 55,000 psi using the strength-hardness relationship) and a maximum weight gain of 30 mg/(dm^2)(1000 hr) were established. Measurements of hot hardness over the temperature range from 200 to 900 C on alloys containing the minimum and maximum alloying additions were made. Several alloys shown in Table A-2, which have the best combinations of properties, have been selected for further investigation.

Specimens were prepared by rolling arc-cast buttons to sheet from an 850 C helium furnace and annealing at 700 C for 4 hr followed by furnace cooling. Pieces 1 by 3/4 by 0.070 in. were prepared in triplicate for corrosion testing by shearing from fabricated sheet. Approximately 0.003 in. was machined from each surface and an additional 0.002 in. was pickled from the surfaces (45 volume per cent HNO_3-5 volume per cent HF-50 volume per cent H_2O pickle solution).

Corrosion testing has been completed. In general, increasing the tin content has improved the corrosion resistance of these alloys. In the alloys containing 4.0 w/o tin, 29 of 32 alloys fell within the maximum corrosion parameter. The addition of molybdenum or niobium does not appear to improve corrosion resistance, nor in many alloys does it appear to be greatly detrimental. It is difficult to separate the relative strengthening effect of the individual alloying elements as reflected by the hot-hardness values. Increases of 2.0 w/o tin, other elements remaining constant, produce a significant increase in hardness of alloys at 300 C. Corresponding increases in total molybdenum and niobium contents also produce significant increases in hardness. Table A-3 shows these effects at 300 C. The effect is evident over the temperature range from 200 to 700 C.

Table A-2 shows the alloys and their properties which this survey indicates should be investigated further. Several other alloys have favorable properties, but these seven have the best combination of all properties. Zircaloy-2 is listed as a reference alloy.

TABLE A-2. ALLOYS SHOWING FAVORABLE PROPERTIES

Nominal Alloy Composition (Balance Zirconium), w/o	DPH		Weight Gain in 300 C Water Corrosion Test, mg/(dm^2)(1000 hr)	Approximate Thermal-Neutron Cross Section, barns per atom
	Room Temperature	300 C		
2.0 Sn-0.5 Mo	290	140	18	0.198
2.0 Sn-2.0 Nb-0.1 Fe-0.05 Ni	307	140[a]	14	0.200
2.0 Sn-3.0 Nb-0.1 Fe-0.05 Ni	297	150[a]	14	0.213
3.0 Sn-0.1 Fe-0.05 Ni	312	160	12	0.189
3.0 Sn-0.5 Mo-0.1 Fe-0.05 Fe	304	165	15	0.199
3.0 Sn-0.5 Mo-1.0 Nb-0.1 Fe-0.05 Ni	319	160[a]	19	0.209
4.0 Sn-0.5 Mo	333	165	21	0.205
Zircaloy-2[b]	230	104	24	0.195
Zircaloy-2[c]	210	104	13	0.195

(a) Estimated hot-hardness values from similar alloys.
(b) Values obtained in this testing program.
(c) Values obtained from literature.

TABLE A-3. RELATIVE EFFECT OF TIN AND MOLYBDENUM AND NIOBIUM ADDITIONS ON HOT HARDNESS AT 300 C

Nominal Alloy Composition (Balance Zirconium), w/o	Hardness at 300 C (DPH)	
	Nominal 2.0 w/o Sn	4.0 w/o Sn
0.1 Fe-0.05 Ni[a]	113	150
0.5 Mo-0.1 Fe-0.05 Ni[a]	140	165
1.0 Mo-1.0 Nb[a]	160	195
1.0 Mo-3.0 Nb[a]	145	228
2.0 Mo-3.0 Nb-0.1 Fe-0.05 Ni[a]	187	277
Zircaloy-2[b]	104	

(a) Tin content listed under hot hardness for comparison purposes.
(b) Reference alloy.

A-5 and A-6

Physical Distortion of Graphite

W. C. Riley, A. J. Roese, and W. H. Duckworth

Research will be directed toward development of sink-float density measurements as a method of identifying factors affecting irradiation-induced volume changes in graphite.

Modification and improvement of the experimental method are under way. Limitations of the method will be determined. Studies will include the effect of variations in particle size on sink-float density measurements.

When the experimental method has been adequately developed, samples of unirradiated reactor-grade graphite will be measured. These samples will be sent to another site for high-temperature irradiation to various exposures. Sink-float density measurements will be repeated after irradiation to determine changes.

Preparation of Molybdenum Single Crystals

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

Single crystals of high-purity molybdenum are to be prepared for irradiation-damage studies.

A modified Andrade furnace has been constructed to grow molybdenum single crystals. Preliminary experiments with several 1/4-in.-diameter molybdenum rods indicated that temperatures sufficiently high to grow single crystals could not be maintained for long periods of time. Smaller diameter (1/8 in.) molybdenum rods have been obtained and are being prepared for testing in the furnace.

The length of time and exact temperature necessary to grow single crystals of molybdenum have not been established. Once these parameters have been established, eight or ten single crystals of 1/8-in.-diameter rod will be prepared. Upon completion of the molybdenum single crystals, an attempt will be made to prepare several zirconium crystals.

B-1

B. DEVELOPMENTS FOR ALUMINUM-CLAD FUEL ELEMENTS

R. J. Carlson

Radiographic examination and sectioning of extrusion billets of aluminum-35 w/o uranium cast by centrifugal techniques revealed essentially sound material. The evaluation of the aluminum-35 w/o uranium alloys containing 3 a/o per cent ternary additions is continuing. Thermal analyses and fabrication studies are being conducted on these alloys.

A series of nickel-plated uranium slugs has been clad with aluminum and shipped to another site for evaluation. The preparation of these specimens completes this program of study.

The program concerned with the improvement of the corrosion resistance of high-uranium fuel alloys has been completed. The results of this study indicate that the corrosion life of uranium can be increased by a factor of 2 to 3 and the life of uranium-2 w/o zirconium can be increased by a factor of 1.5 to 2 by ternary additions.

Continued emphasis has been placed upon the acquisition of information pertaining to the engineering properties and applications of uranium monocarbide. Certain desired data are very limited; therefore, future work will emphasize the application of property data that are obtainable to calculations designed to predict the performance of UC under reactor-operating conditions.

Preparation of Aluminum-Uranium Alloys

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

Interest in aluminum-uranium alloys clad with aluminum for use as fuels in low-temperature water-cooled and -moderated reactors is reflected in present efforts to prepare more satisfactory fuels of this type. Most of the reactors now using these fuels employ alloys containing 20 w/o or less of uranium in the form of flat-plate-type fuel elements clad in aluminum. The technique for the production of these elements is fairly well established. However, it is desirable to increase the fuel loading of these reactors and thereby realize an economy in both fuel handling and neutron utilization. When the uranium content of the alloys is substantially increased, two major difficulties are encountered. These are segregation and porosity. The segregation is due to the density differences between the solid and the liquid during freezing and to the large freezing range of the alloys. The porosity is attributed to shrinkage and to the evolution of hydrogen during solidification.

One fuel element now being considered consists of an aluminum-35 w/o uranium-alloy tube clad inside and out with aluminum. The envisioned technique for the production of these elements requires casting in the form of a hollow right cylinder. After suitable cleanups, the cylinder would then be coextruded with aluminum to form the tubular fuel element. In casting such a cylinder, the usual difficulties which are

encountered in casting simple shapes are magnified. Therefore, a program of study concerned with centrifugally casting suitable extrusion billets is now in progress.

The evaluation of a series of castings containing 35 w/o uranium has been completed. This evaluation included chemical analyses, radiographic examinations, and macroscopic examinations of sections cut from the ingots. The chemical analyses revealed that areas of high uranium concentration were located in those sections of the ingot where the molten metal was introduced. The concentration of the uranium fell off drastically toward the areas farthest away from the point of entry of the melt. The over-all variation in uranium content was from 5 to 8 w/o.

Radiographic examination and sectioning revealed essentially sound material in the major portion of the ingots. The porosity that was found was attributed to shrinkage of the melt. It is believed that much of this will be eliminated as soon as the movable pouring spout is introduced. This mechanism is now completed and will permit the introduction of the molten metal into any portion of the mold that the operator desires. This also is expected to decrease the variation in uranium content along the length of the ingot.

A concurrent program concerned with the improvement of the casting and fabricating characteristics of aluminum-uranium alloys has been concentrated upon those alloys which exhibit the greatest change in compound formation with 3 a/o alloy additions. It is believed that the fabricating characteristics of the binary alloys can be improved greatly by either refining the primary compounds or by reducing the amount of compound present in the as-cast structure. It was found that copper, niobium, and palladium increase the hardness of binary aluminum-uranium alloys on the order of 50 per cent. Metallographic examinations of these alloys revealed very coarse structures in the alloys containing copper and niobium. The structure of the alloy containing palladium was relatively fine, with an apparent decrease in the percentage of primary compound present. Other alloy additions which have produced fine as-cast microstructures are tin and sodium. In an effort to evaluate the effect of particle size on fabricability, a series of alloys has been press forged at 1000 F. The visual examination of these specimens failed to reveal extensive edge cracking in any of the materials tested after approximately 45 per cent reduction in length. However, microcracking may have occurred in any number of these alloys; therefore, the specimens are being examined by metallographic techniques to determine the extent of microcracking present and where these cracks are initiated. The alloys are also being subjected to thermal analyses utilizing a platinum-platinum 10 w/o rhodium thermocouple embedded in the specimens. Both cooling and heating curves are being obtained. These curves will be used in conjunction with the metallographic examinations to determine if the peritectic reaction of UAl_3 with liquid to form UAl_4 occurs in the alloys containing the ternary additions. If there is no evidence of UAl_4 in the microstructure and if there is no evidence of the above reaction occurring, it can be assumed that no UAl_4 is formed in the alloy.

Future studies will be conducted on both phases of the program – casting and ternary additions. The casting studies will use a movable pouring spout and improved protection of the molten metal. The portion of the program concerned with the ternary additions will continue to be concentrated upon the evaluation of the melts that have been made to date. However, an increased effort will be made to determine the mechanism

through which the various additions refine the structure of the binary alloys or inhibit the formation of UAl_4.

Aluminum Cladding

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

The cladding of internally and externally cooled fuel elements consisting of a nickel-plated uranium core clad with aluminum is being investigated. Pressure bonding, a technique which incorporates high-pressure gas and elevated temperature to obtain a metallurgical bond between core and cladding, is being investigated as a method of preparing this type of fuel element.

The additional uranium slugs prepared during June by bonding 1 hr at 950 F and 10,000 psi have been forwarded to another site for final evaluation.

Studies concerned with gas-pressure bonding of nickel-plated uranium elements have been concluded, and a report covering the results of this study is being prepared.

Development of a Natural-Uranium Fuel Alloy With Improved Corrosion Resistance

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

Development of a high-uranium fuel alloy that possesses improved corrosion resistance in 300 C water is being pursued. Alloy composition is limited to an addition of 4 w/o zirconium or its equivalent in terms of the resultant alloy thermal-neutron cross section. Corrosion life greater than that exhibited by the uranium-2 w/o zirconium alloy is being sought.

Four corrosion tests have been conducted on groups of 17 alloys in which the nominal compositions do not exceed the cross-section limit noted above. The tests were carried out on specimens measuring 1/4 by 1 by approximately 0.06 in. Alloys selected for testing were sealed in an autoclave, which was then flushed with argon and evacuated several times. The autoclave was heated to 300 C, and preheated water was admitted. Corrosion proceeded for 15 min; steam was released from the autoclave, and the cooling autoclave was flushed with cold argon. Further cooling was achieved by water quenching the autoclave from 204 C.

Results of the first three corrosion tests are reported in BMI-1273. The fourth and final test is reported in Table B-1. This test was performed to compare the corrosion rates of aged and slowly cooled alloys. These results indicate that the corrosion rates for aged uranium-zirconium alloys are the same or slightly lower than for alloys in the slowly cooled condition. However, the uranium-zirconium alloys with ternary additions of aluminum, niobium, platinum, or ruthenium show higher corrosion rates in the aged condition.

TABLE B-1. CORROSION RESULTS OBTAINED ON URANIUM ALLOYS TESTED SIMULTANEOUSLY 15 MIN IN 300 C WATER

Sample	Nominal Alloy Composition (Balance Uranium), w/o	Corrosion Rate[a] for Alloy With Indicated Heat Treatment, mg/(cm^2)(hr)	
		24 Hr at 880 C, Air Cooled in Vycor	1 Hr at 900 C, 24 Hr at 450 C, F.C.
	Biscuit uranium	4320	--
1	2 Zr	3328	3268
2	4 Zr	>3310[b]	2960
3	5 Zr	3140	2776
5	2 Zr-0.33 Nb	2692	2200
6	2 Zr-0.047 Ni	--	>2890[b]
9	2 Zr-0.05 Pt	1980	2528
13	2 Zr-0.46 Al	2212	2360
14	1 Zr-0.5 Nb	2712	--
15	2 Zr-0.15 Nb-0.05 Mo	2664	--
16	2 Zr-0.16 Ru	2052	2520

(a) Based on original area.

(b) Disintegrated, corrosion rate quoted is based on original thickness of specimen.

This investigation is now complete; experimental data obtained indicate:

(1) Aluminum, niobium, platinum, and ruthenium, when added to uranium-2 w/o zirconium alloys in amounts as determined by cross-section considerations, improve the corrosion resistance more than an additional 2 w/o zirconium.

(2) The corrosion life of uranium can be increased by a factor of 2 to 3 and the life of uranium-2 w/o zirconium can be increased by a factor of 1.5 to 2 by ternary alloying.

(3) The degree of improvement in corrosion life varies with heat treatment.

Literature Survey for the Appraisal of Uranium Monocarbide as a Possible Nuclear Fuel

J. B. Melehan, A. A. Bauer, and R. F. Dickerson

Efforts have continued on gathering information concerned with the science of uranium monocarbide and its engineering properties and applications. Numerous references have been reviewed and, although additional reports remain to be consulted, it is apparent that the major part of past research effort has dealt with the preparation of the carbide phases for the determination of relationships in the uranium-carbon system, and the determination of physical properties. Reports show that UC has little tendency to react either with NaK up to 1300 F or with bismuth at 1100 F. Destructive hydrolysis reactions are known to occur with water even at ambient temperatures, and UC oxidizes rapidly in air at elevated temperatures. Data on fabrication, thermodynamic and chemical properties, and behavior under reactor-operating conditions are very limited.

However, on the basis of crystal structure, thermal conductivity, and mechanical properties, it may be possible to make some predictions concerning behavior of uranium carbide as a fuel. The capacity of a fuel material to accommodate fission products is reported to depend upon the density of interstitial lattice sites and the concentration and distribution of porosity. The moduli of rupture and elasticity establish the limiting internal stresses which may be imposed upon the lattice by accumulating fission products. These two moduli are, in turn, functions of the internal-temperature distribution, which is controlled by the coefficient of thermal conductivity. In addition, the diffusion of fission products from the fuel material will determine the strength properties required by a cladding designed to protect the fuel and contain fission products. These several interrelated factors ultimately determine the maximum operating temperature for the fuel and, hence, the optimum geometry of fuel elements.

In future work, emphasis will be placed on applying such property data as can be obtained to calculations designed to predict UC performance under reactor-operating conditions.

C-1

C. RADIOISOTOPE AND RADIATION APPLICATIONS

Paul Schall

Two studies have been initiated as part of the program of the Office of Industrial Development of the AEC. These studies are on (1) radiation chemistry of organic materials trapped in inclusion compounds, and (2) the use of radioactive tracers for industrial quality control.

Inclusion compounds have been prepared and initial irradiations were started during July. The effort on the tracer study has been devoted to review of pertinent literature to select specific quality-control procedures for investigation.

Radiation Chemistry of Inclusion Compounds

M. J. Oestmann, J. L. McFarling, and W. S. Diethorn

Certain inorganic and organic molecules are trapped in cagelike molecular structures when the cage component is crystallized in the presence of these substances. In these so-called inclusion compounds both the cage component, the "host", and the "guest" component retain their chemical identity. Typical host molecules include urea, thiourea, and the quinones. Guest molecules include a variety of inorganic and inert gases, and the hydrocarbons and their derivatives. For a given host the formation and physical stability of an inclusion compound is determined largely by the size of the guest molecule. Urea and thiourea inclusion compounds are decomposed readily into their components by dissolving the compounds in water. Cage sizes are of molecular dimensions and the trapped guest molecules are restricted in their translational motion and contact with neighboring guest molecules. These latter restrictions suggest that the radiation chemistry of the guest molecules will be different from that for the same molecules in the gaseous or liquid state.

Currently being investigated is the radiation chemistry of inclusion compounds which may have possible application in the area of radiation processing. The objective of this study is to investigate the feasibility of using inclusion compounds as a means of increasing the radiation yields of desirable products from selected hydrocarbons and their derivatives. Possible applications in radiation processing include the formation of branched hydrocarbons from straight-chain hydrocarbons and the synthesis of novel products by radiation-induced reactions between host and guest molecules.

During July, the pertinent literature on inclusion compounds was surveyed and six compounds selected for the initial irradiation experiments using urea and thiourea as the hosts as indicated on the following page.

C-2

Urea	Thiourea
Heptane	Cyclohexane
Cetane	Cumene
Stearic acid	
Amyl acetate	

Several of these compounds have been prepared and are being irradiated in the Battelle cobalt-60 irradiation facility at dose rates from 2 to 4.5 x 10^5 rads per hr. These compounds and others to be prepared next month will be irradiated to a total dose of 10^8 rads. Following these irradiations several of the compounds will be selected for chemical analyses and the nature and yields of the radiolysis products determined. Gas-chromatography methods are being explored for the chemical analyses.

Development of Radioactive-Tracer Quality-Control System

D. N. Sunderman, J. E. Howes, and M. Pobereskin

An investigation has been undertaken to establish the feasibility of the development of radiotracer systems for process and quality control. This may be accomplished by developing and demonstrating the appropriate radiochemical analytical techniques on a laboratory-scale process.

The research program will be executed in the following manner:

(1) A study will be made of ASTM analytical procedures presently in use in various operating processes. One process will be selected for further development on the basis of complexity and cost of the standard analytical techniques.

(2) Analytical procedures based on the use of radiotracers will be developed as substitutes for the procedures established in Step (1).

(3) Using a laboratory-scale process as a source of samples, the radiotracer techniques will be compared with the standard ASTM methods. Comparison will be based on ease of analysis, precision, time, and cost.

The study of ASTM analytical procedures and other pertinent literature has been initiated. It is anticipated that selection of suitable processes will be made and experimental work will be initiated during August.

D-1

D. PROCESSING OF FEED MATERIALS

E. L. Foster

The study of the importance of the factors involved in the solidification of the uranium casting is the objective of this program.

To date, the theoretical considerations have been evaluated, and a mathematical model of the heat flow from the ingot has been formulated. The initial solutions from the mathematical model have appeared reasonable, and an experimental program has been initiated to provide data for comparison with the calculated results.

Solidification of Uranium

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

Initial small-scale casting experiments have been completed. The objective of these experiments was to measure and record temperatures in the casting and mold materials during the cooling period. The temperature data which have been recorded will be compared with data obtained by calculation of the heat flow. Based on these comparisons, evaluation of the various casting factors present during the casting of uranium ingots of production size will be made.

Two ingots, 3 by 20 in. and approximately 100 lb in weight, were cast. Each melt was held for 5 min at 2550 F and subsequently poured into a cold graphite mold under a vacuum of 50×10^{-2} mm of mercury. A pouring time of about 15 sec was observed for the melts.

The procedure used to obtain temperature histories was to place thermocouples in the mold in such a manner as to measure temperatures in the metal at horizontal positions 0.25, 0.75, and 1.50 in. from the ingot surface and vertically on the outside mold wall at intervals of 5 in. One thermocouple was placed flush with the inside wall of the mold. In addition, thermocouples were placed on the inside wall of the furnace jacket and at the bottom of the crucible support. The thermocouples at these positions recorded the temperatures in the immediate surroundings of the mold and ingot.

Preliminary examination of the thermal data taken during the casting operations shows that temperatures as high as 925 F existed at the outer surfaces of the mold walls during the cooling of the ingot. In the metal itself, some loss of information occurred as a result of mechanical failure of the thermocouples positioned at the center of the ingot. Temperatures of 200 and 700 F were noticed at the furnace wall and at the crucible supports, respectively.

Further castings will be prepared by the same technique in order to more definitely establish temperature fields in various areas of the mold and ingot. Each casting will be sectioned to determine the exact location of the thermocouples and to observe the as-cast grain structures.

D-2

With the experimental work now under way, the program for calculation of the heat flow in the casting is being adapted to the experimental-size ingot. To simplify the interpretation of the results obtained, the actual machine calculations are being withheld until the position of the thermocouples is definitely established by sectioning the ingot; this should reduce the amount of interpolation necessary in subsequent computer runs to compare calculated with experimental results.

E-1 and E-2

E. DEVELOPMENTS FOR LMFRE

J. McCallum

The purpose of this study is to investigate the possibilities of applying a thin protective coating of molybdenum on the interior surfaces of the reactor system. Both electroless and vapor-plating techniques are being studied as possible means of attaining the coating.

Electroless Plating

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

During July, four different Grignard reagents were used to reduce molybdenum pentachloride in several organic solvents. The Grignard reagents were methyl magnesium bromide, ethyl magnesium bromide, ethyl magnesium chloride, and n-butyl magnesium chloride, all as 3 M solutions in ether. The solvents used were diethyl ether, dibutyl ether, benzene, toluene, and xylene. Reduction of the molybdenum pentachloride did not proceed to the metallic state but apparently stopped at an intermediate (±2, +3, or +4) valence state with the formation of an insoluble molybdenum compound. Potential measurements in the solutions of Grignard reagents and of molybdenum pentachloride indicate that sufficient driving force exists for the reduction to the metal.

Future work will be with Grignard reagents, using other molybdenum compounds and other solvents.

Vapor Plating

C. F. Powell and I. E. Campbell

Work during July was concerned with the preparation of 2 lb of molybdenum pentachloride, with deposition of molybdenum on Croloy 2-1/4 specimens by thermal decomposition of molybdenum hexacarbonyl at 500 to 600 C, and with molybdenum deposition on Croloy 2-1/4 by hydrogen reduction of molybdenum hexachloride vapor at 750 to 800 C. The molybdenum coatings obtained from the carbonyl were much thinner than desired, but were adherent. Difficulty was experienced in maintaining a uniform specimen temperature of accurately known value with the type of specimen on hand. The coatings obtained from the hexachloride were adherent, except on two specimens coated at the lower end of the range of deposition temperature used, and were 0.5 to 1.0 mil thick. These coatings were slightly thinner than is desired for testing, so heavier coatings (3 to 5 mils thick) will be applied to additional specimens. Work will then be resumed on studying the vapor deposition of molybdenum from the pentachloride and hexacarbonyl at around 550 C.

F-1

F. RESEARCH FOR AEC REACTOR DEVELOPMENT DIVISION PROGRAM

S. J. Paprocki and R. F. Dickerson

REACTOR MATERIALS AND COMPONENTS

R. F. Dickerson

The first phase of the work concerned with valence effects of oxide additions to uranium dioxide is complete and future work will be concerned with obtaining a major reduction in the additive necessary to prevent oxidation, attempts to stabilize $UO_2 \cdot ThO_2$ bodies by other additive oxides, and more detailed studies of oxidation behavior.

Improved pressure equipment will be constructed for use in the synthesis of new materials. The main disadvantage of present equipment has been an exceptionally high thermal gradient. The investigation of hydrides of uranium-zirconium alloys as fueled moderators is continuing. Samples containing 1 to 50 w/o uranium were hydrided in preparation for structural studies by high-temperature X-ray diffraction. One irradiation capsule for operation at 1500 F has been completed and is ready for loading in the MTR. The second capsule for operation at 1100 F will be loaded during August.

The responsibility for, and direction of, an irradiation-damage program to determine the extent of damage to Type 347 stainless steel in fast-neutron fluxes in the core of the ETR has been assumed. The program is designed to determine changes in physical properties caused by continued exposure to fast fluxes after exposure at various flux levels up to 14 to 16 x 10^{22} nvt. Tensile, cyclic-strain fatigue, Charpy V-notch, and subsize Izod impact specimens will be exposed and tested.

Valence Effects of Oxide Addition to Uranium Dioxide

W. B. Wilson and C. M. Schwartz

The purpose of this project is to gain fundamental information on the effects of major additions of divalent and trivalent cation oxides to uranium oxide when the latter is subjected to oxidation. The first phase of the work is nearly complete and a report is being prepared.

Future work will be directed toward three objectives:

(1) Utilizing the information obtained from prior research, efforts will be directed toward a major reduction of additive necessary to prevent oxidation and sublimation of the volatile oxide.

(2) The oxidation characteristics of $UO_2 \cdot ThO_2$ bodies appear capable of considerable improvement through use of other additive oxide. Work in this area will be directed toward the same end as for UO_2 itself

except that $UO_2 \cdot ThO_2$ bodies (which break up upon oxidation and lose uranium oxide) will be used.

(3) More detailed studies of oxidation and its "prevention" will continue in order that principles involved may be applied to other volatile oxide systems.

High-Pressure High-Temperature Solid-State Studies

W. B. Wilson and C. M. Schwartz

This project was initiated to investigate the effects of pressure, as a new degree of freedom not conventionally used, in producing new materials for use as high-temperature reactor components. Experimental studies are continuing to determine the effects of pressure on uranium oxide and uranium oxide with certain additives, such as BeO, La_2O_3, Sc_2O_3, etc.

Improved pressure equipment, to be used in synthesis-type work, will be constructed on the basis of experience with the present equipment. The principal disadvantage of present equipment has been too high a thermal gradient. This led to a relatively large uncertainty in the temperature of the sample. The new design will attempt to minimize this.

Fueled Zirconium Hydride Moderator

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

The investigation of hydrides of uranium-zirconium alloys as fueled moderators for possible use in gas-cooled reactors is continuing. During July, zirconium-1 to 50 w/o uranium samples were hydrided, in preparation for structural studies by high-temperature X-ray diffraction. Capsule BMI-20-1, which contains both fueled and unfueled zirconium hydride specimens for radiation-damage studies, has been completed.

Structure and Pressure-Composition-Temperature Studies

Alloys of zirconium containing 1 and 50 w/o uranium, which were fabricated into 10-mil wire, were hydrided to levels of 10, 30, and 50 a/o hydrogen (based on the zirconium content). Suitably hydrided samples were obtained by reaction with hydrogen at 610 to 620 C for 3 hr. However, it was found necessary to supplement the wires with bars of alloy to get a hydrogen uptake large enough for accurate measurement.

These hydrided alloy wires will now be examined by X-ray diffraction at 500, 600, 700, and 800 C in order to establish the structure of the condensed phases. The determination of hydrogen-absorption isotherms will now be resumed, using zirconium-25 w/o uranium alloy.

F-3

Radiation-Damage Studies

The zirconium hydride specimens were loaded into Capsule BMI-20-1, and the chamber surrounding them was filled with hydrogen and sealed. The outer capsule shell was then assembled and loaded with NaK. Work has been started toward the completion of Capsule BMI-20-2, which is identical with Capsule BMI-20-1. However, the design center-line temperature of the second capsule is 1100 F, as compared with 1500 F for the first.

Irradiation Surveillance Program on Type 347 Stainless Steel

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

The responsibility for, and direction of, an irradiation-damage program to determine the extent of damage to Type 347 stainless steel in fast-neutron fluxes in the core of the ETR has been assumed. The program is to supply data in support of the KAPL-33 loop program by determining changes in physical properties caused by continued exposure to fast fluxes. Present irradiation-damage data on Type 347 stainless steel are not adequate to predict changes in physical properties for materials exposed to flux levels of 14 to 16 x 10^{22} nvt. These levels of irradiation represent approximately a 3-year life for materials in the C33-F-10 position at the ETR. The surveillance program includes exposure of tensile specimens, cyclic-strain fatigue specimens, and Charpy V-notch and subsize Izod impact specimens to increasing amounts of integrated neutron flux and determination of changes in mechanical properties associated with the several levels of irradiation. The levels of irradiation to be investigated are 2.4, 4.8, 7.2, 9.6, 12, and 14.7 x 10^{22} nvt.

The design of the in-pile tube has been based primarily upon the tensile properties of Type 347 stainless steel. Any changes of these properties with increased exposure to ETR process water and at approximately 600 F are important. Tests of irradiated tensile samples will be made at room temperature and at 600 F after exposure at the above-mentioned flux levels to determine whether temperature influences the extent of radiation damage either during or after exposure. It is thought that the thermal stress experienced by the tube in operation will exceed the yield point of the material and, hence, it will be important to know how neutron irradiation affects the low-cycle high-strain plastic fatigue strength of Type 347 stainless steel. The specimens will provide cyclic-strain fatigue curves after irradiations for time increments equivalent to 6 months of operation for the in-pile tube. Comparison of these properties with preirradiated properties should indicate whether irradiation-induced changes in this property make it unsafe for loop operation. Impact specimens will be used to determine if Type 347 stainless steel becomes brittle during irradiation at levels of exposure expected for the KAPL in-pile tube. Safe operation of the loop is of prime importance. The impact specimens will be irradiated to integrated neutron fluxes equivalent to 6 months and 2-1/2 years of the KAPL-33 in-pile-tube operation. Comparison of test data from the irradiated samples with similar preirradiated properties of the material will provide an indication of the structural integrity of the in-pile-tube material.

F-4

The eight "cold" capsules or those to be exposed to ETR process water were inserted in the F-10-NE and F-10-SE positions in June. Nickel disks were attached on top of each capsule and on aluminum-cobalt wire attached in the same general area. These will provide a measure of fast- and thermal-neutron fluxes in the vicinity of the experiment. Loading was made in the F-10 instead of the H-10 position, as planned originally, for several reasons: (1) the H-10 position was not available for the experiment at the present, (2) the experiment in the F-10 position, the position to be occupied by the in-pile tube, will receive neutron fluxes equal to those anticipated for the in-pile tube, and (3) it was thought further delay would impair securing data in advance of insertion and operation of the in-pile tube. All capsules are to be transferred to the H-10 or a comparable position as soon as possible.

The mock-up capsules containing the same weight per unit length of material to be exposed in the ETR are being prepared for the ETRC.

The mechanical properties of the Type 347 stainless steel before irradiation as determined by KAPL have been reviewed. Plans for construction of a unit to perform cyclic-strain fatigue tests are under consideration.

In the present plans for dosimetry to measure the neutron flux, nickel wires will be placed in the center and in one core-face dosimeter hole and exposed simultaneously during one complete ETR cycle in the F-10 position. After discharge of the dosimeter wires, samples will be taken from eight comparable positions on each wire. These will represent two places for each of four capsules in the test holes, and will provide flux data for eight points in two vertical planes and two points in eight horizontal planes. Sulfur may be used in either or both dosimeter holes to substantiate the fast-flux data determined. For succeeding cycles in the F-10 position only minimum dosimetry, defined as a nickel wire in the center dosimeter hole of the core filter piece, will be used. The use of cobalt and cadmium-shielded cobalt wire has been suggested to determine more completely the energy spectrum of the neutron flux.

A lead or instrumented capsule will be used to check out the design of the hot capsules and to correlate gamma flux with temperature of the specimens in the capsule during irradiation. The expected procedure is as follows:

(1) Determination of the gamma profile in either the NW or SW hole of the F-10 position with the reactor at low power, presumably at the start of a cycle.

(2) Insertion of the lead capsule to the peak flux level in the hole on which the gamma profile had been made.

(3) Recording the temperature of the specimens in the capsule at this time during an entire cycle or until reactor shutdown during a cycle. At such time the lead capsule will be moved to a new position toward the top of the test hole.

It is thought that the temperatures at the peak flux level and the top of the hole can be measured during one cycle and the temperature at intermediate points established if there are several reactor shutdowns during one cycle. This procedure will be repeated when the experiment is moved to the H-10 or comparable position. The reported gamma-flux measurement for the F-10-NW and F-10-SW at the start of Cycle 3 and with the reactor power at 80 megawatts was 14 and 13 w per g, respectively. The hot capsules were designed and built on the basis of an expected gamma flux of 25 w per g. This indicates that the hot capsules will run at somewhat lower temperature than the anticipated 600 F in the F-10 position.

Attempts will be made to evaluate the fast flux in the F-10 positions so that time of exposure for the capsules can be determined. The gamma-flux measurements will be made as soon as possible.

STUDIES OF ALLOY FUELS

R. F. Dickerson

The development of gamma-phase uranium alloys formerly reported in this section has been completed, as has been the study of the mechanism of aqueous corrosion. Experimental work on the radiation effects of uranium-zirconium alloys has been temporarily recessed.

Because of the possible use of niobium-uranium alloys as a high-temperature fuel, the program concerned with these alloys has been continued. The primary objectives will be to study the properties of alloys containing 40, 50, 60, 70, 80, and 90 w/o niobium. These will include fabricability, mechanical and physical properties, and corrosion characteristics in several environments. The effect of impurities on these properties will also be evaluated.

A program aimed at developing thorium-uranium alloys with increased irradiation stability and corrosion resistance has been initiated. Effects of impurities, fabrication techniques, and heat treatment on distribution of the alpha uranium will be studied as well as alloying to stabilize the gamma-uranium phase.

Development of Niobium-Uranium Alloys

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

The possible use of niobium-uranium alloys as fuel materials has resulted in an active interest in the properties of the high-niobium portion of the niobium-uranium constitution diagram. Most of the information reported has been concerned with uranium-rich alloys (less than 20 w/o niobium). A survey of the literature has shown that the properties of uranium-niobium alloys are influenced greatly by the purity of the niobium and uranium used in the preparation of alloys. Therefore, the effects of

the major impurities, zirconium and oxygen, upon the properties of high-niobium alloys will be investigated. Those properties to be studied are fabricability, mechanical and physical properties, and corrosion characteristics in several environments.

The alloys selected for study are uranium-40, -50, -60, -70, -80, and -90 w/o niobium. In order to check the effect of contamination on the properties of these alloys, three grades of niobium will be used. These are high-purity niobium containing less than 300 ppm oxygen and less than 200 ppm zirconium, niobium containing approximately 800 ppm oxygen and about 1.0 w/o zirconium, and a third grade of niobium containing 800 ppm oxygen and less than 0.5 w/o zirconium. Each of the above alloy compositions will be prepared using all three grades of niobium as base materials.

Fabrication temperatures are to be estimated by the use of special hot-hardness buttons cast for this purpose. Hot-hardness readings will be obtained up to 900 C.

All alloys other than the hot-hardness specimens are to be prepared by consumable-electrode arc-melting techniques and will weigh about 4 lb. Fabrication methods to be studied will include hot rolling, forging, and press forging. The methods of fabrication may vary from one composition to another, depending upon information obtained from hot hardness, metallography, alloy composition, and prior investigations.

A study of the mechanical and physical properties of these alloys will be made to determine the effects of the impurities and heat treatments upon the alloys. There is little or no information available on the properties of the high-niobium alloys. The final phase of the program will be a corrosion study in sodium, water, carbon dioxide, and air.

Center-cut derby uranium biscuit has been secured and is being prepared for melting. The niobium to be used for melting stock is on order. Casting of the hot-hardness buttons will proceed as soon as materials are available.

Development of Thorium-Uranium Alloys

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

Interest in thorium-uranium alloys as breeder materials has prompted a program aimed at developing thorium-uranium alloys with increased irradiation stability and corrosion resistance. Ternary and quaternary alloying will be considered for this purpose.

Initially, thorium-uranium alloys will be studied comprehensively to determine the effects of impurities, fabrication techniques, and heat treatment upon the distribution of the alpha-uranium phase in the alpha-thorium matrix. Control of the alpha-uranium phase distribution may provide a means for limiting swelling of the alloy which accompanies release of fission gases by this dispersed phase.

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The retention of fission gases by the uranium-rich phase may also be increased by alloying to stabilize the gamma-uranium phase. Ternary additions of niobium and molybdenum, which show little solubility in thorium but which are soluble in and stabilize the gamma-uranium phase, will be used for this purpose.

Thorium-uranium-zirconium alloys will also be examined. Such alloys should be heat treatable, permitting variations in phase distribution to be achieved. In addition, it may be possible to produce matrix variations, either alpha thorium, or a thorium-uranium-zirconium phase, or uranium-zirconium-epsilon phase as the matrix phase. Improved corrosion resistance as well as irradiation stability might be obtained with such alloys.

Techniques to be employed in this investigation will include metallography, X-ray diffraction analysis, and hot-hardness tests. Selected alloys will also be corrosion tested and mechanical-property data will be obtained on promising alloys. On the basis of results obtained, alloys will be selected for irradiation studies.

GENERAL FUEL-ELEMENT DEVELOPMENT

S. J. Paprocki

During the past fiscal year, dispersion-type fuel specimens containing 24 w/o fully enriched UC and UN dispersed in a 18 w/o chromium-14 w/o nickel-2.5 w/o molybdenum-balance iron matrix and clad with Type 318 stainless steel were fabricated for irradiation testing at 1650 F. The irradiation behavior of these materials will be compared to specimens containing an equivalent loading of uranium-235 in the form of UO_2.

The studies concerned with the development of cermet fuel elements are continuing. Emphasis is being directed to the development of cermet fuel elements containing 60 to 90 volume per cent of fuel component dispersed in a metal matrix. It is anticipated that such fuel materials will combine some of the desirable features of both the ceramic and the conventional cermet fuel systems.

Gas-pressure bonding is being investigated as a technique for the cladding and bonding of niobium and molybdenum fuel elements. This bonding process is extremely promising because a solid-state bond is produced and the brittleness encountered during the brazing and welding of these materials is not present. The initial studies will be concerned with the development of an optimum surface treatment and pressure-bonding conditions for molybdenum and niobium.

The initial selection of gas-pressure-bonding parameters of time, temperature, and pressure for the bonding of a material is based on the recrystallization temperature of the material. Using these parameters as a starting point, many tests are then conducted to establish the optimum bonding conditions. A study is being conducted with the objective of relating the creep properties of a material to its stress behavior during pressure bonding. This would permit the prediction of optimum bonding condition for any material for which the creep properties are known.

F-8

Radiation Effects on Dispersion-Type Fuel Elements

D. L. Keller, G. W. Cunningham, and A. W. Hare

The primary purpose of this program is to determine the effects of irradiation at elevated temperatures on dispersion-type fuel elements which contain 24 w/o UC or UN in a matrix of stainless steel and are clad with Type 318 stainless steel. Twelve test specimens have been prepared for the irradiation experiment. The fueled specimens have a core which is about 31 mils thick, 1/2 in. wide, and 1 to 1-1/8 in. long. The cladding thickness is about 7 mils. Six of the specimens will contain UC and the balance will contain UN. Two of each type of specimen will be irradiated at 1650 F in three irradiation capsules to estimated burnups that will equal and exceed the burnup level achieved with similar specimens containing an equivalent amount of uranium-235 in the form of UO_2.

The necessary specimens have been fabricated and given the necessary pre-irradiation physical measurements and density measurements prior to encapsulation.

Four of the specimens have been encapsulated in a thermocoupled capsule and have been sent to the MTR for irradiation. This lead capsule will be used to determine the relative flux necessary to obtain a specimen temperature of 1650 F. After the data from the lead capsule are obtained, the other two capsules will be loaded and shipped to the MTR for irradiation. Progress reports will be discontinued until irradiation results are available.

Fabrication of Cermet Fuel Elements

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

Development has been expanded on cermet fuel elements containing 60 to 90 volume per cent fuel compound dispersed in a continuous metal matrix. Fuels being investigated include UN, UC, and UO_2. Metallic skeletons will consist of chromium, molybdenum, nickel, niobium, and stainless steel.

Initial work is to be concentrated on the production of dense compacts for thermal-conductivity measurements. Thermal conductivity will not be measured on all the possible combinations, but an attempt will be made to obtain enough data to establish the relative degree of improvement which may be expected when a cermet is used rather than a ceramic. A hot-pressing technique will be used to prepare the specimens.

Preliminary fabrication studies have shown that hot pressing is the most feasible method of producing many of these cermets; therefore, a detailed study will be made of the effects of such variables as time, temperature, pressure, and powder particle size. Also, for systems such as stainless steel-UO_2, in which the metal does not wet the ceramic, techniques such as coating the UO_2 with metal by vapor deposition, adding sufficient metal to give the correct composition, cold pressing, and sintering will be

investigated. Infiltration can also be considered in cases where wetting can be promoted. Fabrication methods will be evaluated on the basis of visual and metallographic examinations, mechanical-property tests, electrical-conductivity measurements, thermal-shock tests, and thermal-expansion measurements.

Gas-pressure-bonding techniques will be investigated for producing clad assemblies. Bend tests, transverse tensile tests, and metallographic examinations will be made to determine the core-to-cladding bond strength.

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

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

Niobium and molybdenum are promising materials for inert-gas-cooled and some liquid-metal-cooled high-temperature reactor applications. Their most important property is high-temperature strength. At 2000 F, the tensile strength of niobium is approximately 9,000 psi, while molybdenum has a tensile strength of approximately 10,000 psi. Molybdenum and niobium possess relatively low thermal-neutron-absorption cross sections with no significant resonance in the thermal or epithermal regions. These properties make niobium and molybdenum highly desirable for use as structural components for high-temperature reactors.

The favorable nuclear and high-temperature strength properties of molybdenum and niobium as cladding materials establish a need for an effective process for bonding, cladding, and joining of molybdenum and niobium-clad fuel elements. It is possible to join both of these materials by welding in a closely controlled atmosphere. The niobium is more easily welded because it does not become embrittled as readily during welding. Also, the room-temperature ductility of the molybdenum welds is sometimes difficult to obtain as a result of oxide precipitation along the molybdenum grain boundaries during the welding operation and the relatively high brittle-to-ductile transition of recrystallized molybdenum. This transition can be as high as 575 F for material that has been recrystallized during fusion welding. Thin foil sections of molybdenum and niobium can be joined by ultrasonic spot welding; however, this process has not been proven applicable for welding of claddings to relatively thick frame material. In general, welding of these materials by inert arc welding has been fairly well established if base materials of suitable purity are employed and if atmospheric contamination is avoided during welding. Since both niobium and molybdenum display a high affinity for oxygen and nitrogen, it is necessary to protect them from atmospheric exposure during cladding at elevated temperatures by roll cladding or coextrusion. The temperatures necessary to obtain bonding of these materials to themselves present an additional problem. Oxidation-resistant materials normally employed as outer protective cans during cladding of metals that have a high affinity for oxygen and nitrogen will readily alloy with the molybdenum and niobium at the bonding temperatures. In addition, the lower strength of the protective can materials at elevated temperatures will result in the can reducing much more than the molybdenum and niobium components; consequently, possible rupture of the protective can may occur or

the fabricated fuel element will display poor dimensional properties. In an attempt to eliminate these problems, some work has been previously done on bonding of these materials by solid-state diffusion processes involving the use of die pressures to obtain bonding; however, none of these processes has been proven applicable for cladding fuel elements with molybdenum or niobium.

Many of the problems associated with conventional fabrication techniques for cladding with molybdenum and niobium can be avoided by pressure bonding of these elements with a high inert-gas pressure at elevated temperatures. The gas pressure can be applied directly on an edge-welded fuel element or bonding can be done in a two-step operation. The latter case would consist of obtaining a partial bond by pressure bonding of the elements in a coated protective can at intermediate temperatures subsequent to decanning and final pressure bonding by direct pressure on the cladding at elevated temperatures. The partial bond obtained in the initial stage would have to be leakproof so that a pressure differential could be established during the second bonding operation. The two-step process would be applicable for both fuel elements and complete subassemblies. Since only a minimum amount of deformation is encountered during pressure bonding, it may also be possible to pressure bond in a one-step operation using a coated protective can at elevated temperatures.

Three types of fuel elements are being considered in this investigation. These are flat plate, wafer, and tubular fuel elements which will embody core materials consisting of high-loaded cermets, high-loaded dispersions, or compartmented uranium dioxide cores. Pressure bonding is an attractive method of bonding these brittle materials because a minimum amount of deformation is involved during the bonding operation. In addition, the dispersion characteristics of the core are not altered during the bonding operation.

In the initial phases of this investigation, the bonding of niobium and molybdenum to itself will be studied. The effect of prior fabrication history and surface preparation on the bonds obtained during pressure bonding will be investigated. If niobium and molybdenum will not bond properly except by recrystallization and grain growth during the pressure-bonding cycle, it will be necessary to put the material in the best condition for recrystallization and grain growth to minimize bonding times and temperatures. Because the bond surfaces do not deform enough during pressure bonding to break up surface contaminant films, the preparation of the surface for bonding is especially important. In work with other cladding materials, it has been found that surfaces contaminated by improper pickling, machining, abrading, or degreasing do not bond properly. It will be necessary to determine the most suitable surface preparation for niobium and molybdenum for bonding. It is assumed, because of the desire to obtain recrystallization of the material along the mating interfaces, that methods of surface preparation will include peening, belt abrading, or machining to produce localized areas of high energy by cold work. The latter two methods would include determining a suitable surface roughness to produce high-energy points for bonding.

A series of flat-plate specimens incorporating two flat plates of molybdenum or niobium will be used to study effects of fabrication history and surface preparation on the bonding of the materials to themselves. Similar specimens will be used to investigate pressure-bonding parameters and cladding-to-can or -spacer reaction. All bonds will be evaluated by bend, chisel, and peel tests, and metallographic examination to determine bond integrity.

F-11 and F-12

Materials are being obtained for this investigation, and preliminary flat-plate-type specimens are being assembled to obtain information on surface-preparation and pressure-bonding parameters.

Basic Studies of Pressure Bonding

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

An investigation is in progress with the aim of establishing criteria for choosing pressure-bonding conditions. Pressure bonding as considered here refers to the process of bonding contacting surfaces by the application of gas pressure at elevated temperatures. In this study the emphasis is on determining the conditions which cause sufficient deformation of the contacting surfaces to insure perfect contact.

Analytic Studies of Pressure Bonding

The assumption is made that the surface deformation which occurs is substantially a creep phenomenon. It is necessary to prescribe a model to represent the surface geometry. In principle, at least, the creep of the surface can be predicted in terms of the simple creep law $\dot{\epsilon} = A\sigma^n$. Here $\dot{\epsilon}$ and σ refer to strain rate and stress invariants, and A and n are creep parameters for a given material at a given temperature. The first model used to describe the surface geometry was that of a collection of infinitely thick-walled spheres subjected to hydrostatic pressure. However, this model led to a large overprediction of the conditions necessary to bring the surfaces into intimate contact, even though the creep parameters are not known accurately. An alternative model for the surface geometry is being sought.

An effort was made to determine the creep parameters for 2S aluminum at 500 and 700 F by analyzing data on the collapse of thick-walled cylinders under external pressure. Due to the combined effects of limited information obtainable from each experiment and large scatter, these tests do not appear to be a practical way of obtaining creep parameters in a small number of tests. However, the torsional creep of cylindrical bars may be analyzed in terms of the same fundamental creep law. In addition, an individual torsion test can be used to give a direct comparison between creep rate and stress, since the angular velocity and torque can be measured directly. Thus, a reduction in the number of tests needed to obtain the creep parameters is very likely.

Experimental Studies of Pressure Bonding

A series of torsion tests is being planned in an effort to obtain the parameters associated with the power law for creep. Cylindrical bars of 2S aluminum will be twisted at a constant angular velocity and the torque will be measured. If the assumed creep law is valid, a substantially constant torque will be associated with each angular velocity. The data from these tests may then be used in the analytic studies.

G-1 and G-2

G. FATIGUE STUDIES OF INCONEL AND INOR-8

W. S. Hyler and F. H. Lyon

Fatigue Studies of Inconel

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

During July work continued on improving the specimen-extensometer system and increasing the reliability of the recording system. It appears that by calibrating for load and temperature it may be possible to preset the desired operating load. This would permit change from the rest condition to the testing condition without the intermediate manual load adjustment. The calibration is partially completed.

It is expected that during the next month the details of the test procedure will be further clarified, and the modification of the equipment will be extended to provide facilities for other kinds of operation.

Fatigue Studies of INOR-8

This study has been recessed until further notice.

J-1

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

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

The evaluation of materials of construction for use in the various proposed processes for the recovery of spent reactor fuel elements has continued.

Titanium is still the material of choice for use in the Darex dissolver and feed-adjustment tank. Current experiments show no ill effects from caustic cleaning or from mechanical injury. Scale removal does not seem to constitute a serious problem.

Studies have been continued to seek ways of preventing stress-corrosion cracking of Carpenter 20 Cb in the Sulfex-Thorex system. Both stress-relief treatments and cathodic protection are under investigation. Nionel suffers from intergranular attack in areas of carbide precipitation when exposed to these solutions. A remedy is being sought in stabilized or extra-low-carbon grades of this alloy. Illium R shows promise during scouting experiments in Sulfex and Thorex solutions but remains to be studied in the as-welded condition.

A run is under way in a new molten-salt composition (62 mole per cent NaF-38 mole per cent ZrF_4). Specimens of INOR-1 and INOR-8, Hastelloy B, and Type S-816 are being exposed.

The Darex Process

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

Dissolver Studies With Titanium

Specimens from the 2000-hr exposure in the flowing dissolver are still in the process of being examined. No evidence of any severe corrosive attack has been observed.

A new experiment is under way with the flowing dissolver. Several plain and welded titanium specimens, including some longitudinally welded specimens which have been placed in tension by means of special supports, are being exposed to the conditions of continuous dissolution. At weekly intervals, the acid is drained and the entire dissolution vessel and contents are cleaned by boiling an aqueous solution containing 10 w/o NaOH within the dissolver for 3 hr. Following rinsing, dissolution is continued. Three such cleanings have been made with no apparent bad effects.

During this exposure, some titanium specimens exposed high in the vapor phase above the point of dissolution are being scratched daily to see if the oxidative power of the vapor at this point is sufficiently high that the protective film on titanium will be repaired before corrosive damage occurs.

FAT Studies With Titanium

The various plain, welded, and stressed titanium specimens were removed from the boiling Initial FAT solutions following 4500 hr of exposure. The weight losses measured for these specimens were very low and no stress-corrosion cracking was observed. Representative specimens are being sectioned at vulnerable locations, such as through crevices and through weldments and their heat-affected zones, for metallographic inspection.

The scale which forms on the submerged portion of titanium steam tubes exposed in Final FAT solutions was removed quite easily during one experiment by exposing it to a boiling solution of the Initial FAT composition. This indicates that the scale will probably be removed during each refill of the tank prior to the boil-down operation and, thus, it does not constitute any great problem in relation to the heat-transfer characteristics of the steam tubes.

The Sulfex-Thorex Process

The evaluation of various metals for use as container materials for the dissolution step of a proposed Sulfex-Thorex process has continued. In this particular process, stainless-clad fuel elements of thorium or thoria would be dejacketed by dissolution in sulfuric acid. Following this, the thorium or thoria would be dissolved by a solution of 13.0 M HNO_3, 0.05 M F^-, to give a final solution of about 8.5 M HNO_3, 0.05 M F^-, and 1.0 M $Th(NO_3)_4$. Experience has already pointed out that the addition of 0.2 M Al^{+3} to the Thorex solution will help to prevent excessive corrosion of several materials and will probably be used during actual practice.

Experiments With Carpenter 20 Cb

Experiments in which Carpenter 20 Cb specimens are exposed to boiling 6 M H_2SO_4 while in contact with dissolving stainless steel are in progress. These studies are being run in batch fashion for only as long as it takes to reach a buildup of about 5 g per liter of dissolved stainless steel. Then the acid is discarded and replaced with fresh. This is done to prevent obtaining the corrosion-inhibiting effects already observed when sufficient concentrations of dissolved stainless steel are present. So far, the experiment has not been run enough times to allow a determination to be made as to whether or not this cathodic protection from the stainless anode is preventing cracking of the Carpenter 20 Cb specimens.

Specimens of Carpenter 20 Cb, both 11 and 16 gage, containing weldments, were obtained and given a solution anneal at 1950 F for 15 min, followed by a water quench.

Part of these specimens were then given a stress-relief treatment by holding at 1650 F for 10 min and cooling in the furnace. All of these specimens will be exposed in boiling 6 M H_2SO_4 to see if the stress-relief treatment has been successful in preventing the development of cracking.

Experiments With Nionel

As mentioned in BMI-1273, welded specimens of Nionel developed severe intergranular attack following exposure to Thorex solutions either alone or cyclically with Sulfex solutions. By the time 20 cycles had been reached, following the usual schedule, the attack had become so severe that further exposure seemed unwarranted. This attack occurred in the areas of carbide precipitation in the heat-affected zones next to the weldments in much the same manner as has been noted often with austenitic stainless steel. Presently, either an extra-low-carbon grade or a stabilized grade of Nionel is being sought as a solution to this type of attack.

Scouting Experiments With Other Metals

Scouting experiments were conducted with specimens of Illium R exposed to boiling Sulfex and Thorex solutions. Illium R had reasonably low corrosion rates in all but the Initial Thorex solution. Here rates from 120 to 160 mils per month were recorded. However, these were drastically reduced to around 2 mils per month by the addition of 0.2 M Al^{+3}. More scouting experiments will be made with welded specimens of Illium R to see if it suffers from selective attack in the areas of carbide precipitation.

The Fluoride-Volatility Process

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

At the present time, a run is under way in the 62 mole per cent NaF-38 mole per cent ZrF_4 salt. While the material of primary interest in this run is INOR-8, specimens of INOR-1, Type S-816, and Hastelloy B have also been included. Present plans call for inspection of these specimens following 500 hr of exposure. During the second 500-hr period, the HF flow rate will be increased considerably above that normally used, to determine how important a role this flow rate plays in the corrosion process. Other runs with lithium salts and salts containing uranium to represent final hydrofluorinator conditions are planned.

K-1

K. DEVELOPMENTS FOR SRE, OMRE, AND OMR

J. E. Gates and F. A. Rough

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

EVALUATION OF URANIUM MONOCARBIDE AS A REACTOR FUEL

F. A. Rough

The primary concern of this research is to develop uranium monocarbide as a fuel for SRE. The activities are divided into two major parts: the development of casting and fabrication techniques to produce integral, unclad specimens and irradiation-capsule design, and radiation-effects studies on unclad uranium monocarbide. In the casting-technique phase, six specimens have been prepared for loading into the MTR. A standard arc-melting procedure has been adopted which controls the composition of melted buttons to within ±0.1 w/o of the desired carbon content.

Casting Techniques for the Preparation of Uranium Monocarbide

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

Uranium carbide is one of a number of fuels presently being considered for future reactor use. Portions of this program are concerned with the preparation of as-cast irradiation-test specimens of UC and the determination of some of the physical properties of UC.

Six UC castings are being made using uranium enriched with 8.4 w/o uranium-235. These specimens, 3/8 in. in diameter by 2 in. long, are being prepared to have 4.6, 4.8, and 5.0 w/o carbon. All of these specimens will be loaded into capsules for irradiation in the MTR. Each capsule will contain two specimens of the same nominal composition.

Further thermal-cycle shock tests have been made at 1100 C on specimens containing quantities of carbon above and below the UC stoichiometric composition. Machine-ground specimens with 4.5 and 4.7 w/o carbon, and an as-cast specimen with 5.0 w/o carbon, showed no evidence of fracturing after 100 thermal cycles (7 min in the furnace at 1100 C; 15-min cooling period at room temperature).

K-2

By adopting a standard arc-melting procedure, it has been found that the composition of buttons can be duplicated to within ±0.1 w/o of the desired carbon content. Excellent reproducibility was also achieved on castings made by mixing portions of castings of different composition. In each case the resulting casting showed good uniformity in composition from top to bottom.

Tabulated below are the results of an experiment designed to determine the optimum melting time for minimum carbon pickup in the preparation of 100-g arc-melted buttons:

Number of Melts	Melting Time per Melt, sec	Location of Sample	Composition, w/o carbon
7	20	Edge	4.85
7	20	Center	4.84
6	30	Edge	4.93
6	30	Center	4.95
6	35	Edge	5.04
6	35	Center	5.04

The results indicate that carbon pickup from the graphite electrode tip increases with time and that the buttons are homogeneous. The procedure adopted as standard involves six remelts at 30 sec each.

Irradiation-Capsule Design for Uranium Carbide

R. B. Price, R. H. Barnes, and W. H. Goldthwaite

The first MTR capsule, BMI-23-1, containing uranium-5.0 w/o carbon specimens is being irradiated in MTR Position A-28-NE in a reactor thermal flux of approximately 1×10^{14} nv. Due to the plutonium run being scheduled for Cycles 108 and 109, Capsule BMI-23-1 will be discharged at the end of Cycle 107 in August. Satisfactory specimens of uranium-5.0 w/o carbon are available for Capsule BMI-23-2 and all but one, the 1/4-in. uranium-5.0 w/o carbon specimen, are available for Capsule BMI-23-3. Some uranium-4.8 w/o carbon specimens and one uranium-4.6 w/o carbon specimen have been fabricated and radiographed for Capsules BMI-23-4, BMI-23-5, or BMI-23-6. Metallographs of these specimens are now being made.

As soon as the specimens are approved for encapsulation and physical measurements on the specimens have been completed, Capsules BMI-23-2 and BMI-23-3 will be loaded and shipped to the MTR. The target insertion date for these two capsules is Cycle 110 in September following the plutonium run. All six MTR capsules are thermocoupled.

Typical thermocouple temperatures for Capsule BMI-23-1 during 40-megawatt reactor operation are given in Table K-1.

TABLE K-1. TYPICAL THERMOCOUPLE TEMPERATURE FOR CAPSULE BMI-23-1

Thermocouple[a]	Temperature When Indicated Regulating Rod in Use, F							
	RR1 (7-11)	RR1 (7-14)	RR1 (7-16)	RR2 (7-18)	RR2 (7-21)	RR1 (7-23)	RR1 (7-25)	RR1 (7-29)
1	1330	1260	1225	1190	1145	1615[b]	1545	1440
2	720	720	700	710	700	870	825	835
3	660	645	645	635	610	820	770	760
4	410	405	420	410	385	500	480	465
5	495	490	505	500	485	590	555	555
6	315	300	320	315	305	390	360	355

(a) Position of thermocouple:
- 1 In core center of top specimen.
- 2 and 3 Touching basket at center of top specimen.
- 4 Touching inner capsule wall opposite top specimen.
- 5 Touching basket at center of bottom specimens.
- 6 Touching inner capsule wall opposite center of bottom specimens.

(b) Capsule raised into a higher flux during midcycle refueling.

These readings represent peak values observed periodically by the Battelle representative at the MTR. Readings for thermocouples 2, 3, and 5 must be extrapolated to the specimen surface. For more precise values, studies will be made of the recorded temperature graphs. There is a general tendency toward decreasing temperature with time irradiated.

The long-term NaK-UC-stainless steel compatibility studies at 1100 F are continuing. Since the longest irradiation anticipated is six cycles (about 12 weeks at power), the compatibility studies will be terminated at the end of 3 months at temperature.

The BRR capsule containing specimens for fission-gas-release study is being irradiated in BRR Core Position 56 in a reactor thermal flux of about 1×10^{13} nv. This capsule will be discharged in August. Fission gas released into the capsule during irradiation will be examined and specimen postirradiation measurements will be made. Following this, the fission gas released will be studied on individual postirradiation heat-treated specimens. A preliminary test of apparatus and procedures is being conducted on two uranium-5.0 w/o carbon specimens irradiated for 1 min at 1×10^{11} nv thermal.

POSTIRRADIATION STUDIES OF SRE, OMRE, AND OMR FUEL MATERIALS

J. E. Gates

The postirradiation examination of twelve thorium-11 w/o uranium specimens irradiated in two capsule trains has been essentially completed. Preparations are being made to perform radiochemical burnup analyses and metallographic studies. After these additional data become available, the results can be evaluated.

K-4

Two capsules from the SRE program, NAA-28-1 and NAA-29-1, containing irradiated specimens of UO_2 arrived during July. An examination of these specimens is scheduled for August.

The dummy element containing irradiated specimens of structural materials of interest to the OMR program arrived in July. Examination and testing of these specimens to determine the effects of irradiation and corrosion induced by the organic coolant will be initiated during August or September.

An irradiated OMRE fuel element was received in July. An examination of this, OMRE Element 3, has begun. Preliminary inspections have indicated that it is in good condition.

SRE Fuel Material

G. E. Lamale, J. H. Saling, W. J. Braun, and J. E. Whitney

Thorium-Uranium Specimens

The postirradiation examinations of thorium-11 w/o uranium specimens from Capsules NAA-15-6 and NAA-15-7 have been completed. Specimens have been prepared for radiochemical burnup analyses, metallographic examinations, and measurements of the linear-thermal-expansion coefficients. This work is scheduled to begin in August and to be completed during September. An evaluation of the irradiations will be made after the burnup is determined.

The data obtained from the gas collected from several capsules are still being evaluated. A fission-product analysis of the NaK recovered from two capsules is also being studied.

Uranium-Dioxide Specimens

Preparations are being made to examine the UO_2 specimens irradiated in Capsules NAA-28-1 and NAA-29-1. The examination is scheduled for August.

OMRE Fuel Elements

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

The first fuel element from the OMRE has arrived. The inspection of this element, OMRE Element 3, has begun.

Visually, the element appears to be in good condition. A layer of black carbonaceous material was visible on the outer surfaces of the fuel element. The thickness was

estimated to be approximately 0.5 mil. Most of this material near the fueled area of the element was easily removed with trichloroethylene. The adherent material on the cooler portions of the element near the ends was not easily removed. All of the coolant passages between the plates were free of any obstruction.

The examination of this element will continue. It is planned to examine the outer surfaces of the element for evidence of corrosion, measure over-all distortion, cut off the ends and measure plate spacing, and scan the element to determine the relative radiation intensity at various points. Upon completion of the examination the element will be milled apart. The thickness of individual plates will then be measured and the plates inspected. Burnup analyses will be made on selected areas and a comparison made with the gamma scan data.

OMR Fuel and Structural Materials

R. J. Burian, and J. E. Whitney

The first dummy element containing specimens of structural materials has arrived. The structural materials will be examined after the surveillance examination of the OMRE element and the routine examination of the UO_2 capsules are completed. Since the examination of the structural materials will involve several individual tests, such as tensile and corrosion tests, it is planned to insert this work into the cell schedules in small portions, rather than to do it all at one time. It is believed that this work can be completed during the next 2 to 3 months.

L-1

L. STUDIES OF SODIUM-TANTALUM COMPATIBILITY AT ELEVATED TEMPERATURES

J. H. Stang

The studies of the creep properties (in sodium and in inert atmosphere) and the weldability of tantalum conducted for Los Alamos Scientific Laboratory in connection with the LAMPRE program are nearing termination. During July, attention was given to the initial aspects of a program having the objective of developing core-construction alloys for LAMPRE service. According to present plans, the materials to be investigated will be based on tantalum, with additions of refractory metals such as molybdenum, niobium, and tungsten. The current exploratory studies are concerned with the tantalum-tungsten system.

Tantalum-Sodium Compatibility Studies

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

All experiments in this research, which has been concerned with determining the effects of sodium exposures (in polythermal loop systems at 1200 F) on the creep behavior of tantalum, were terminated during July. Data from two long-time inert-atmosphere creep runs are now on hand and provide baseline creep data directly comparable with those from the sodium runs. The earlier conclusion that 1200 F sodium has very little effect on the creep strength of tantalum remains unchanged. The completed baseline data indicate, however, that the minimum creep rates previously reported have probably been somewhat on the high side. This is based on the observation that in one of the inert-atmosphere runs an externally mounted dial gage such as was used in conjunction with the creep-loop systems indicated a consistently higher minimum creep rate than did a conventional internal gage-section-mounted extensometer. The difference, which is small from the standpoint of actual elongation, apparently stemmed from deformation of the specimen grips and elongation of the pin holes (as reported in BMI-1273, pin-hole distortion during first-stage creep has been an important consideration). With the dial-gage method, these stray deformations are registered along with gage-section deformation, while with the extensometer method they are not.

It was reported in BMI-1273 that the creep rate being exhibited by a sodium-exposed tantalum specimen which had a reduced oxygen content by virtue of a lengthy soak in sodium prior to stressing was somewhat higher than exhibited by comparable specimens loaded immediately after contact with sodium. During the last portion of this experiment, the creep rate of this specimen gradually declined to about 0.0003 per cent per hr, which is comparable with other sodium-exposed-specimen rates. This result would suggest that interstitial oxygen does not contribute greatly to the strength of tantalum, an indication found in prior inert-atmosphere creep experiments reported in BMI-1238.

L-2

High-Temperature Mechanical Properties of Tantalum

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

Final creep tests at 1200 F in a helium atmosphere on 30-mil strip specimens of thermally degassed fine-grained sintered tantalum are in progress. Data now on hand are presented in Table L-1. Results of the long 12,000-psi run indicate that the creep strength of this material is higher than that of commercial annealed sintered material; its strength appears to be about the same as that of the commercial annealed arc-cast product. The runs at 10,000, 14,000, and 16,100 psi will be concluded after about 1000 hr.

TABLE L-1. CREEP DATA ON THERMALLY DEGASSED FINE-GRAINED SINTERED TANTALUM SHEET TESTED AT 1200 F IN A HELIUM ATMOSPHERE

Stress, psi	Time in Progress, hr	Total Deformation, per cent	Minimum Creep Rate, per cent per hr
10,000	91	0.062	Nil
12,000	1148	0.092	Nil
14,000	428	0.138	0.0002
16,000	306	0.166	Nil

The preliminary phase of an alloy-development program, having the objective of tailoring materials to the requirements of the LAMPRE applications, is under way. The requirements include resistance to attack by molten plutonium-base fuels more consistent than possessed by commercial tantalum, fabricability, and an acceptable level of elevated-temperature strength. It is believed that a high purity will be essential to reduce the susceptibility to attack to a degree that can be tolerated. The work now under way is dealing with tantalum and tantalum-3 and -6 w/o tungsten alloys. Small (100 to 150-g) buttons and rods are being arc melted at low pressure in a helium atmosphere to provide material that can be used to evaluate factors such as ingot-purity level, hardness, microstructure, and ease of cold rolling and swaging. Melting stock composed of Fansteel sintered sheet and Temescal electron-beam-melted ingot is being used to prepare the unalloyed tantalum specimens; Fansteel tantalum sheet and tungsten wire are being used for the preparation of the tantalum-tungsten alloys.

Weldability of Tantalum for High-Temperature Systems

S. M. Silverstein and R. P. Sopher

The final activity in this program has been devoted to a study of factors responsible for porosity in arc welds of some lots of tantalum, and, during July, experimental work on this phase was concluded. In the final experiment, a lot of tantalum which was classed as unweldable because of porosity formation was remelted in an arc furnace. In several melts, carbon was added, but in one it was not. It was found that sound welds could be made with all remelted samples. This result for samples to which

carbon was added is in line with past experience concerning the beneficial effects of carbon in reducing porosity. It is noteworthy, however, that sound welds could be made in the sample without a carbon addition. To investigate this, analyses for carbon and oxygen were made on the original material and on this sample. It was found that the former contained 175 ppm oxygen while the latter contained 62 ppm oxygen; both contained 10 ppm of carbon.

These findings along with others that have been accumulated lead to the belief that a carbon-oxygen ratio is an important governing factor in porosity of tantalum welds. If the carbon content is relatively high, the oxygen content can be high without causing a lack of weldability. Similarly, if the carbon content is low, the oxygen level should be low if the weld is to remain free of porosity. The explanation for porosity in materials which do not come under these constituent requirements is not known. It is not unlikely that elements other than carbon and oxygen play a part in the porosity problem.

M-1 and M-2

M. DEVELOPMENTAL STUDIES FOR THE PWR

R. W. Dayton

No experimental work was carried on during the month.

Improved Techniques for Corrosion Detection in Subassemblies and Clusters

G. G. Cocks and C. M. Schwartz

The purpose of this project is to make it easier to interpret the photographs of corrosion-tested fuel-plate assemblies taken with the panoramic camera. No research was performed during the past month. Future work will include testing the corroded samples furnished by the cooperating laboratory and the preparation of the final report.

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

S. J. Paprocki, E. S. Hodge, D. C. Carmichael, and C. C. Simons

Consideration is being given to the types of studies which should be made during further work on this subject.

N-1

N. DEVELOPMENTS FOR THE MGCR

W. H. Goldthwaite

In the MGCR program there is interest in carbon dioxide as the coolant, at temperatures up to 1500 F and pressures to 2000 psi, and graphite as the moderator. The graphite must be clad with a material resistant to corrosion by the CO_2 and excessive carburization by the graphite. The effect of radiation on these reactions is being studied at Battelle.

Investigation of the Effect of Irradiation on Clad Graphite Specimens in a CO_2 Environment

J. C. Smith, R. H. Barnes, and W. H. Diethorn

A program to investigate the effects of reactor radiations on clad graphite specimens is in progress. The experimental objectives of this initial program are to irradiate the specimens in a CO_2 environment at 1000 psi and 1500 F. A capsule containing six specimens is presently being irradiated in the Battelle Research Reactor. Specifically, the specimens are AGOT graphite clad with Type 310 stainless steel, Type 446 stainless steel, or Inconel. There are two specimens with each type of cladding. The results of the postirradiation examinations will be compared with results obtained from duplicate out-of-pile experiments which will be conducted elsewhere. The program objective is to learn the effects of reactor radiations on corrosion by the gaseous environment and reactions at the graphite-cladding interface.

Out-of-Pile Operation Check of In-Pile Capsule

Prior to insertion in the reactor, the operation of the in-pile capsule was checked in out-of-pile tests using argon as an inert gas. The capsule was placed vertically in a pool of water to simulate the situation in the reactor. In the process of attaining the desired experiment conditions, it was found that, despite efforts to design the capsule to minimize convection currents, temperature gradients of 500 F existed in the specimen region when the capsule was pressurized to 1000 psi. By placing the capsule in a water jacket and operating the capsule in a horizontal position, the temperature variation in the specimen region was reduced to 100 F with T_{max} at 1500 F and an argon pressure of 1000 psig. To take advantage of the more uniform temperature pattern, the capsule is being irradiated in a horizontal position adjacent to the reactor core at some sacrifice in flux. The alternative, a redesign of the capsule to reduce the convection currents in a manner that would not compromise the experiment by increasing the surface area exposed to the CO_2, was not possible within the time schedule of this program.

Dosimetry and Reactivity Measurements

A nuclear mock-up of the in-pile capsule was fabricated and provided with neutron dosimeters. Both the thermal- and fast- (above sulfur threshold) neutron fluxes were

measured in the originally intended irradiation position in the reactor core before it was known that a change in position would be necessary. The average values obtained were 4.4×10^{12} and 5×10^{12} n/(cm^2)(sec) for the thermal- and fast-neutron fluxes, respectively. The energy delivered to the graphite specimens by a fission spectrum of fast neutrons was calculated to be 1×10^8 rads per hr based on the flux value given above. From CO_2-graphite ion-chamber measurements made in the position of intended irradiation and corrected for the fast-neutron response, a gamma dose rate of 2×10^8 rads per hr was established. Thus, the graphite specimens would have received a total radiation-energy dose rate (fast neutron plus gamma) of 3×10^8 rads per hr.

In order to operate the capsule in a horizontal position, it was necessary to locate the in-pile capsule along a face of the core. At this position it is estimated that the radiation-energy dose rate to the specimens is reduced by a factor of 4, giving an estimated gamma and fast-neutron dose-rate contribution of 5×10^7 and 3×10^7 rads per hr, respectively. The dose rates given above are considered to apply also for the CO_2 gas in the capsule.

The In-Pile Experiment

Prior to establishing the desired experimental conditions of temperature and pressure, the capsule system was flushed five times with CO_2 and then sampled to check the composition of the initial charge of CO_2. The oxygen content of the initial charge was determined to be 0.06 ± 0.03 volume per cent, which is essentially the oxygen content of the tank CO_2.

The system was pressurized and heated. Modifications to the electric power supply delayed operation at the desired temperature and pressure, and at the time the next gas sample was taken, 29-1/2 hr after the reactor startup, the capsule had experienced only 2 hr at an elevated temperature of only 1200 F. Analysis of this sample yielded 0.04 ± 0.03 volume per cent oxygen. The next gas sample was taken 31-1/2 hr later, after 7-1/2 hr at 1500 F and 24 hr at 1350 F. The analysis showed 0.05 ± 0.02 volume per cent oxygen and 3.6 ± 0.2 volume per cent CO. (The absolute magnitude of these concentrations will have to be corrected by consideration of the total volume of the system sampled versus the fraction of this in the irradiation capsule.) The reduction in temperature was necessitated by the loss of two of the three furnace heaters and a reluctance to overpower the remaining heater.

Subsequent analyses of gas samples have revealed a decreasing CO content with in-pile time. In view of this, it has been decided to operate the capsule at the reduced temperature until the CO content stabilizes before risking the last heater in an attempt to operate at 1500 F. At present the operating conditions are 1250 F (oxidation of furnace walls may be responsible for decrease from 1400 F) and 940 psi.

O-1

O. DEVELOPMENTS FOR NMSR

A. W. Hare and R. F. Dickerson

The research reported in this section is part of the Babcock & Wilcox Company's Nuclear Merchant Ship Reactor Program. The current work is primarily concerned with the development of fabrication techniques for UO_2-containing fuel pins for a dynamic loop test at the MTR. A capsule-irradiation program has also been initiated to run long-term burnup tests and controlled central melting tests on prototype fuel pins.

Fabrication of Urania Fuel Pellets for Loop-Test Studies

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

Work on the preparation of slightly enriched UO_2 specimens for the NMSR program has been temporarily recessed, pending receipt of additional UO_2 powder and changes in the capsule-irradiation program. Fabrication of the UO_2 pellets for the loop program has been completed.

Fabrication of Loop-Test Fuel Pins

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

The primary purpose of this investigation is to provide a form of test for prototype UO_2 fuel pins for the NMSR program under anticipated reactor conditions.

Previous work has resulted in the completion of the short test pins for the first in-pile and out-of-pile loadings in the high-temperature high-pressure irradiation-test loop. These pins are now being examined and inspected to detect any discontinuities or cracks, and checked for any helium leaks at the welded areas. With the completion of measurements of length, diameter, and apparent densities of each specimen, the fuel pins will be assembled and shipped to the MTR.

Due to the high boron content (500 ppm) of the Type 304 stainless steel cladding of the long fuel pins, attempts to make leaktight welds at the end caps were unsuccessful.

These thin-walled tubes which have an outside diameter of 0.3125 in. were welded to Type 304 stainless steel end caps using a butt weld design. Metallographic examination of the cross sections of the weld areas indicated that the weakness in the weld could have been caused by a boride precipitate or a boron eutectic phase at the grain boundaries of the weld structure. On the basis of these tests, it was decided to lower the boron content of the cladding material to 300 ppm and to initiate an investigation to determine a method of satisfactorily welding these fuel pins. In this study, an attempt is being made to establish the relationship between welding conditions, depth of penetration, and cracking associated with the welds. Metallographic techniques will be employed where necessary, in order to identify the deleterious phase, or phases, which

would cause the welding difficulties. X-ray diffraction studies will also be made to aid in the identification of the phases, and also to substantiate any results obtained previously by metallographic techniques.

Sixteen specimens were used in this study. One end of each pin was welded in an open atmosphere with supplementary helium shielding through the torch. All sixteen welds were leak tested and examined for cracks and holes. The welds on all specimens were satisfactory as to quality of weld and in regards to leaks. The other ends of the specimens were then end capped in a helium atmosphere. All sixteen specimens exhibited porosity in the ends last welded.

Since all specimens were successfully welded at one end, and contained holes at the other end, it was felt that the expansion of gases could have blown holes in the weld metal.

In order to counteract the possible effect of the expanding gases on the weld joint, it was decided to initiate a welding procedure which would eliminate the pressure inside the tube during welding. This was done by drilling a 45-mil axial hole through the stainless steel end cap prior to welding the end cap to the tube. A strip of wire was then inserted into the opening, with one end of the wire extending out of the end cap. The extended strip of wire was then melted down into the opening, completely sealing the hole.

Seven specimens were welded using this technique, and an evaluation of the soundness of the welds in these specimens is being made at this time. Following the examination of these welds, all seven specimens will be cycled between 1000 C and room temperature for 25 cycles. After the cycling test, the pins will be given a helium leak test and a metallographic evaluation to detect any failures in the welds which may have been caused by the thermal cycling.

Preirradiation Measurements

R. H. Barnes, R. B. Price, and W. H. Goldthwaite

In-Pile-Loop Program

The work on the loop program was continued during July. Six of the short loop specimens were photographed, and the physical examination of the specimens has been completed.

Fuel-Capsule-Irradiation Program

J. C. Smith, R. H. Barnes, and W. H. Goldthwaite

In work on this program, initial calculations have been made and are continuing. It is expected that the design of the capsules will be completed and fabrication will begin during August.

RWD/CRT:all