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PROGRESS RELATING TO CIVILIAN APPLICATIONS DURING DECEMBER, 1958

by

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A-1

A. DEVELOPMENTS FOR ZIRCONIUM-CLAD FUEL ELEMENTS

F. R. Shober

The thermal conductivity and electrical resistivity of uranium and UO_2 are being determined before and after irradiation to investigate the effect of irradiation on these physical properties. Presently, preirradiation properties are being determined for the UO_2.

Six creep samples of 15 per cent cold-worked Zircaloy-2 have been on test more than 10,000 hr. The creep rates calculated from deformation during the later 9000 hr were 0.00001 per cent per hr or less. Creep tests in which the test temperature is cycled to simulate service conditions have been initiated.

The density distribution of crushed graphite is being investigated by the sink-float technique. A change in geometry of the centrifuge tubes reduced the time required for each density separation and improved the reproducibility of the method. Samples of various density fractions will be prepared for irradiation.

Thermal Conductivity of Uranium and UO_2

C. F. Lucks and H. W. Deem

Uranium

Apparatus changes necessary for measuring the thermal conductivity of unclad uranium specimens have been made but measurements have not been started.

Uranium Oxide

The apparatus used in measuring the thermal conductivity of UO_2 has previously been described. This apparatus has been repaired after a heater failure. The thermocouples were modified for remote manipulation during the repair work. After the changes the apparatus was satisfactorily checked using a titanium-alloy standard.

During December, thermal-conductivity measurements were completed from 100 to 600 C on UO_2 Specimen 1000. Data for Specimen 1000 and for a sample evaluated earlier, Specimen 70, are given in Tables A-1 and A-2. A different fabrication method was used to prepare Specimen 1000 than was used for other UO_2 specimens of the series. Although the density of Specimen 1000 is close to that of Specimen 70, the thermal conductivity is roughly 10 per cent higher. This indicates that the method of fabrication may have an effect on the thermal conductivity of UO_2.

While at the highest temperature, with a temperature gradient of 720 to 450 C over 3 in. of the specimen, the vacuum system developed a major leak and the specimen was oxidized. Upon cooling the specimen was found to have fractured.

A-2

During January, the vacuum system will be repaired and measurements begun on a medium-density specimen.

TABLE A-1. INTERPOLATED THERMAL CONDUCTIVITY OF UO_2 SPECIMENS 1000 AND 70 AT SELECTED TEMPERATURES

Temperature, C	Thermal Conductivity, w/(cm)(C)	
	Specimen 1000	Specimen 70
100	0.079	0.075
200	0.065	0.060
300	0.056	0.050
400	0.048	0.043
500	0.043	0.038
600	0.038	0.035

TABLE A-2. URANIUM OXIDE DENSITY DATA

Specimen	Mass, g	Diameter, in.	Length, in.	Density, g per cm^3	Density, per cent of theoretical
1000	25.2492	0.248	3.120	10.27	93.7
70	25.5435	0.251	3.015	10.45	95.3

Mechanical Properties of Zirconium Alloys

F. R. Shober and J. A. VanEcho

An investigation of the creep properties of annealed and of 15 per cent cold-worked Zircaloy-2 in the 290 to 400 C temperature range is being conducted. The greater part of the program has been directed toward long-time creep tests (greater than 10,000 hr) of the cold-worked material. A somewhat lesser part has been concerned with the determination of the creep properties of Zircaloy-2 under a cyclic-temperature condition similar to those expected during service.

Six samples have been on test more than 10,000 hr, four at 290 C and two at 345 C. A comparison of total elongations after 10,000 hr indicates that nearly equivalent total elongations were obtained by loads of 30,000 psi at 290 C and 20,000 psi at 345 C. The creep rates for these same specimens were 0.00001 per cent per hr or less. These samples will be continued on test.

Four cyclic-temperature tests have been started, three cycling from 290 C to 27 C and one cycling from 345 C to 27 C. The specimen is held at the higher temperature for 6 days and then reduced to the lower temperature for 1 day. This sequence is

repeated until specimens have been on test approximately 1000 hr. A comparison made between elongations from specimens tested with and without cyclic-temperature conditions indicates creep behavior is quite similar. Additional constant-temperature tests will be started using annealed specimens at 290, 345, and 400 C for comparison with the 15 per cent cold-worked material.

Physical Distortion of Graphite

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

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

Conical centrifuge tubes were substituted for the round-bottomed tubes previously used. The use of conical tubes resulted in better packing of the sink material and, therefore, reduced the amount of graphite that went into suspension during postcentrifugation handling of the tubes. Also, the time required for each density separation was reduced and the reproducibility of the method was improved.

Measurement of the sink-float density distribution of two different samples of minus 270 plus 325-mesh TS-GBF graphite was made. Results indicate good reproducibility in the experimental method. In addition, a nearly identical density distribution was obtained from measurement of a sample of minus 325-mesh TS-GBF graphite. The results are summarized in Table A-3.

The sink-float density distributions of samples of minus 40 plus 60-mesh, minus 100 plus 140-mesh, and minus 40 plus 140-mesh TS-GBF graphite were measured. Results indicated that the sample with the large particle size contained a higher percentage of low-density material than did the sample with the small particle size. An intermediate percentage of low-density material was found in the composite minus 40 plus 140-mesh sample. The results are summarized in Table A-4. It appears that the composite samples of minus 40 plus 140 mesh can be handled with present experimental technique. No breakdown of minus 40 plus 140-mesh material into small particle-size ranges will be made.

The density distribution of the intermediate size ranges will be measured to determine the minimum number of runs for each bar sample. Two bars were crushed and the density distributions will be measured to determine the variation from bar to bar. Samples of various density fractions will be prepared for irradiation.

Preparation of Molybdenum Single Crystals

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

Materials on order have not arrived. No work was done.

TABLE A-3. DENSITY DISTRIBUTION OF MINUS 270 PLUS 325-MESH AND MINUS 326-MESH TS-GBF GRAPHITE

Minus 270 Plus 325 Mesh						Minus 325 Mesh		
Specific Gravity of Benzene-Bromoform Solution	Amount Floated, w/o	Cumulative Amount Floated, w/o	Specific Gravity of Benzene-Bromoform Solution	Amount Floated, w/o	Cumulative Amount Floated, w/o	Specific Gravity of Benzene-Bromoform Solution	Amount Floated, w/o	Cumulative Amount Floated, w/o
1.643	0.32	0.32	1.566	0.14	0.14	1.877	1.31	1.31
1.892	0.29	0.61	1.953	0.23	0.37	1.955	0.17	1.48
2.035	0.27	0.88	2.083	0.81	1.18	2.073	0.58	2.06
2.100	1.22	2.10	2.124	1.08	2.26	2.126	5.61	7.67
2.146	6.85	8.95	2.164	15.65	17.91	2.183	24.05	31.72
2.178	20.09	29.04	2.211	46.99	64.90	2.232	52.90	84.62
2.244	67.01	96.05	2.285	35.10	100.00	2.280	15.38	100.00
2.278	3.95	100.00	--	--	--	--	--	--

TABLE A-4. DENSITY DISTRIBUTION OF MINUS 40 PLUS 140-MESH COMPOSITE TS-GBF GRAPHITE SAMPLE

Minus 40 Plus 60-Mesh Material			Minus 100 Plus 140-Mesh Material			Composite Sample, Minus 40 Plus 140 Mesh		
Specific Gravity of Benzene-Bromoform Solution	Amount Floated, w/o	Cumulative Amount Floated, w/o	Specific Gravity of Benzene-Bromoform Solution	Amount Floated, w/o	Cumulative Amount Floated, w/o	Specific Gravity of Benzene-Bromoform Solution	Amount Floated, w/o	Cumulative Amount Floated, w/o
1.982	0.80	0.80	1.660	0.08	0.08	1.767	0.24	0.24
2.043	1.73	2.53	1.914	0.08	0.16	1.970	0.16	0.40
2.130	21.52	24.05	1.994	0.12	0.28	2.073	0.86	1.26
2.175	21.32	45.37	2.107	1.49	1.77	2.108	2.96	4.22
2.215	47.83	93.20	2.128	7.54	9.31	2.121	4.44	8.66
2.280	6.80	100.00	2.202	69.39	78.70	2.178	51.00	59.66
--	--	--	2.280	21.30	100.00	2.232	38.24	97.90
--	--	--	--	--	--	2.275	2.10	100.00

A-5 and A-6

11 and 12

B-1 and B-2

B. DEVELOPMENTS FOR ALUMINUM-CLAD FUEL ELEMENTS

Preparation of Aluminum-Uranium Alloys

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

An increase in the uranium content of aluminum-uranium alloys is desirable as a means of increasing the fuel loading of reactors utilizing these fuels. For alloys containing more than 20 w/o uranium, it is necessary to develop casting techniques and to improve fabricability.

Present casting studies are directed toward development of methods for production of extrusion billets of aluminum-35 w/o uranium by centrifugal-casting techniques. The utilization of bottom-pouring techniques in conjunction with a horizontal steel mold has yielded ingots 26 in. long with improved interior wall surfaces. However, attempts to produce billets 36 in. long have proved unsuccessful.

The portion of the program concerned with the investigation of selected ternary additions which might improve the castability and fabricability of the high-uranium alloys has been concerned with the indentification of the phases present in those alloys that exhibited good fabricability. Metallographic and X-ray diffraction studies have shown that the predominant compound is UAl_3. There is considerable variation in the UAl_3 lattice constants and even greater changes in the lattice constants of the aluminum of the eutectic phase.

Future research will be concerned with the continued investigation of centrifugal-casting techniques and of the effects of the ternary additions to the binary aluminum-35 w/o uranium alloy. The effects of casting size on compound formation in the alloys will also be studied.

C-1

C. RADIOISOTOPE AND RADIATION APPLICATIONS

P. Schall

Research on radioisotopes in quality control has resulted in development of a quick and accurate method for the analysis of magnesium in cement. The use of ion-exchange separation of magnesium from other cement constituents is very promising as a rapid method of preparing samples for analysis.

A program on the use of intrinsic radioactive tracers in process control was initiated recently. The effort thus far has been devoted to a study of the technical and economic aspects of various processes to guide the selection of specific systems for experimental evaluation.

In the program on the radiation chemistry of inclusion compounds, the major effort was directed toward separation and identification of the products formed in several urea-hydrocarbon complexes.

Development of Radioactive-Tracer Quality-Control Systems

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

The development of the radiometric method for the analysis of magnesium in cement has been completed. Briefly, this method consists of adding a slight excess of standard phosphorus-32-labeled ammonium phosphate to a magnesium chloride solution and measuring the specific activity of the solution. Magnesium ammonium phosphate is precipitated by addition of ammonium hydroxide and the specific activity of the supernatant solution is measured. From the difference in the specific activity before and after precipitation, the quantity of $(NH_4)_2HPO_4$ required to completely precipitate the magnesium is determined. From the stoichiometry of the reaction, the amount of magnesium in the sample is calculated.

A series of analyses was performed on a standard magnesium sample and the method was found accurate to ±1 per cent. The standard deviation of a series of six analyses was ±0.31 per cent. The time required to run the analyses is approximately 45 min.

Recent experiments also show promise for development of an ion-exchange method for separating magnesium from the other elements in cement. Cement samples dissolved in dilute hydrochloric acid were passed through Dowex 50 cation resin and magnesium was separated quantitatively.

There are no new data on the activation analysis of cement. Another cement irradiation is planned which will aid in further evaluating the feasibility of this technique.

C-2

Use of Intrinsic Radioactive Tracers for Process Control

D. N. Sunderman, T. S. Elleman, J. L. McFarling,
R. H. Barnes, and M. Pobereskin

As part of the effort by the AEC's Office of Isotopes Development to promote the use of radioisotopes and radiation in industry, a research program is being conducted to investigate and develop methods for the use of intrinsic radioactive tracers for process control. The objective of this program is to select a promising process for the application of radiotracer control techniques, and to demonstrate and evaluate these techniques using a complete operational bench model of the process.

The work currently in progress involves a technological and economic review of present-day manufacturing processes to select one with particular promise for radiotracer control. A review of the unit operations and unit processes has been conducted to determine possible general radiotracer applications. A study of existing and past uses of radioisotopes for process control and an economic survey of industry in the United States have been completed.

Emphasis is now directed toward a review of the production processes that are used in the ten top-value-producing industries in the United States. It is anticipated that this phase of the program should be completed and the development of the radiotracer control system for a selected process initiated in the near future.

Radiation Chemistry of Inclusion Compounds

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

The analysis of the irradiated hydrocarbons and urea complexes reported last month was continued during December.

Most of the effort this month was directed toward the refinement of techniques for the separation and identification of radiolytic products from urea complexes of octane, decane, dodecane, and cetane. Considerable progress has been made on resolving most of the analytical difficulties. Results are still being evaluated and will be reported as soon as they become available.

D-1

D. PROCESSING OF FEED MATERIALS

E. L. Foster

National Lead of Ohio (NLO), as the operator of the Feed Materials Production Center, is concerned with the quality of uranium fuel slugs. One area of interest in controlling and maintaining this quality is the vacuum-remelt and casting operation. As part of its work in this area, NLO is sponsoring a program to study the mode of solidification of the uranium in the mold. The application of the results of this program to future casting needs is planned.

Solidification of Uranium

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

A study of the solidification of uranium castings in cylindrical graphite molds is being conducted. The program is embracing two phases of study: a theoretical study of the transfer of heat from the metal and mold, and its relation to the solidification patterns of the casting; and an experimental phase to study the physical properties, the validity of assumptions used in the theoretical phase, and the correlation that exists between the theoretical work and the data recorded during the casting of small-scale ingots.

To study the heat flow in the metal-mold system, a mathematical model has been formulated. The model, consisting of 75 cells, represents a pie-shaped slice of the ingot and mold. The mathematical model provides for the linear variation of the thermal conductivity of the ingot and mold materials. Consideration is also given to the change from conduction to radiation heat transfer as the outer surface of the casting solidifies. Physical properties such as the emissivities of the various surfaces at which radiation is occurring are also being considered.

Hemispherical total-emissivity measurements have been taken on graphite coated with a $MgO \cdot ZrO_2$ mold coating. As reported previously, the initial tests gave emissivity values of 0.71 and 0.46 at 800 and 1350 C, respectively. Because the apparatus failed before the tests were completely finished, further measurements were taken on a second coated specimen. The results of these tests are being evaluated and will be reported as soon as the analysis is complete. Specific heats and liquid thermal-conductivity properties will also be determined on uranium. These experiments will include measurements of these properties at temperatures as high as 1250 or 1300 C. The specific-heat tests will also provide data on the three solid phases of uranium.

The purpose of the theoretical study is to determine the transient heat flow in the mold, casting, and immediate surroundings. Therefore, it becomes necessary to know the physical events occurring in the mold as well as to know the thermal properties of the materials. One of these events to be studied is the mold wall-ingot gap formation caused by shrinkage. Experiments have been started to measure the formation of

the gap between a coated mold and casting. The aim here is to determine when the separation occurs at the mold-metal interface and thereby to indicate when the change from conductive to radiative heat transfer occurs.

Ingots 3 in. in diameter by 20 in. long are being employed for the mold-ingot gap experiments. The melts are being cast into coated graphite molds by vacuum induction-melting techniques. A pouring temperature of 1400 C and a pressure of 1×10^{-1} mm of mercury absolute are being employed. Measurements of the gap are being made with an electrical-probe network. The probes consist of pairs of wires clad with stainless steel. The wires are insulated from each other and the steel sheath with packed MgO powder. The probes are placed in the mold wall 2 in. apart along the length of the casting. One side of each probe is made common at each pickup point. One casting has been made with the probes installed in the mold. Two more castings will be made in this series of tests.

Following the gap-formation studies, additional temperature measurements in the 100-lb ingots will be made. As before, each mold will contain thermocouples arranged to measure temperatures of the metal at the ingot-mold interface, and at the mold surfaces. In addition, thermocouples will be placed on the inside furnace jacket above and below the ingot to obtain environmental temperatures. These thermal data along with gap measurements will be incorporated into the mathematical model and heat-flow calculations will be made.

E-1 and E-2

E. DEVELOPMENTS FOR LMFRE

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

Experimental work was terminated with recommendations to use an electroless or an electrolytic molybdenum oxide coating on the internal surfaces of the LMFRE. Final attempts to obtain molybdenum metal involved: (1) hydrogen reduction of thin coatings of electrolytic molybdenum oxide, and (2) thermal decomposition of electrophoretically deposited molybdenum hexacarbonyl. Both approaches gave negative results. The electroless molybdenum oxide coating was analyzed by X-ray and electron diffractions. The necessary cleaning steps were determined. Operating characteristics of the electroless process were also determined.

F-1

F. RESEARCH FOR AEC REACTOR DEVELOPMENT DIVISION PROGRAM

S. J. Paprocki and R. F. Dickerson

REACTOR MATERIALS AND COMPONENTS

R. F. Dickerson

Preliminary data indicate that the stabilization of uranium oxide can be accomplished by substituting divalent CaO in part for La_2O_3 or Y_2O_3. Additions of 2 w/o La_2O_3 to UO_2 result in greatly increased thermal conductivity. La_2O_3 in the amounts needed to stabilize the urania has the opposite effect on electrical conductivity. Calibration of the high-temperature high-pressure die of advanced design indicates successful performance to pressures just beyond 60,000 atm and temperatures have been recorded up to the melting point of Chromel-Alumel thermocouples. The die will now be water cooled and tested in a larger press at higher pressures.

Boundary limits of the single-phase and two-phase regions have been obtained for the zirconium-25 w/o uranium-hydrogen alloy and X-ray analysis is in progress. The two irradiation capsules containing zirconium hydride fueled with 2 w/o uranium have received approximately one-half the burnup required for the experiment. Despite relocation in the test hole, temperatures are running a little below those for which the experiment was designed.

Eight capsules containing AISI Type 347 stainless steel are being irradiated at ETR process-water temperature. These capsules have received a total integrated fast flux of about 1.8×10^{21} nvt. It is planned to discharge two of these capsules in March. Five dosimeter assemblies for monitoring the fast-neutron flux have been inserted in the test hole. These consist of nickel wires and crystals of ammonium sulfate. A non-lead gamma heat capsule has been inserted in the test position for the purpose of checking the conditions which caused excessive heating of a previous capsule. Hot-cell examination will be performed as soon as the capsule is discharged.

A study concerned with the development of niobium-base alloys as cladding material for advanced EBR designs has been initiated. Since high strength at elevated temperatures is a major requirement, past experience indicates that six alloys should be initially examined: niobium-4.5 w/o zirconium, niobium-1 w/o chromium, niobium-2 w/o chromium, niobium-40 w/o titanium-10 w/o aluminum, niobium-20 w/o titanium-1.5 w/o chromium, and niobium-10 w/o tantalum-2 w/o chromium. The necessary materials have been obtained and the consumable electrodes are being prepared.

F-2

Valence Effects of Oxide Additions to Uranium Dioxide

W. B. Wilson and C. M. Schwartz

Experimental work was continued to reduce the amount of additive oxide required to achieve stabilization of uranium oxides. The present approach, assuming validity of the valence-compensation hypothesis, has been to substitute divalent CaO in part for the sesquioxides of La_2O_3 and Y_2O_3. Compositions of uranium oxide with La_2O_3 + CaO and Y_2O_3 + CaO have been prepared. These materials have not as yet been evaluated in direct air oxidation. The first composition prepared, namely, that of UO_2 + 30 w/o La_2O_3 + 5 w/o CaO, has, however, been tested and indicated a 1.71 per cent weight increase after air oxidation for 20 hr at 3000 F. Negligible fuel losses from this material indicated considerable promise when compared with UO_2 which lost 63 per cent by weight in the same treatment. Temperatures beyond 3000 F appear necessary to evaluate the stabilized materials adequately.

Electrical-measurement studies of uranium oxide with small additions of La_2O_3 have been continued to obtain a more detailed picture of characteristics of mixed oxides with compositions intermediate between UO_2 and the solid solutions containing 40, 50, and 60 w/o of La_2O_3 as additive. These electrical measurements are being performed for correlation with the oxidation characteristics. Materials with nominal compositions of 2, 10, and 23 w/o La_2O_3 have been prepared. The 2 w/o specimen exhibited very high electrical conductivity. The 10 and 23 w/o specimens are being examined. Both the 10 and 23 w/o materials exhibit tremendous affinity for oxygen, and, although attempts were made to prevent oxidation, some of the bodies prepared were converted to powder.

Work will continue on preparation and evaluation of the quaternary materials.

High-Pressure High-Temperature Solid-State Studies

W. B. Wilson and C. M. Schwartz

Experimentation with an advanced die design was continued during the month. This unit was devised to provide higher pressures at temperature than were possible with the previous right-circular cylinder die design. The new unit appears to meet all of the requirements for work currently contemplated. Pressure- and temperature-calibration experiments have been conducted to determine die characteristics.

Pressure calibration, utilizing known transitions, has indicated successful performance to just beyond 60,000 atm. Higher pressures have not been obtained because the calibration work was performed in a small press. The ease of insulation makes direct insertion of thermocouples possible for temperature determination. Temperatures have been recorded to the melting point of the Chromel-Alumel thermocouples used. Modifications are currently being made to the die to provide water cooling and to permit its use in a 750-ton press at higher pressures.

F-3

The interesting behavior of U_3O_8 under pressure, previously discussed, appears to warrant a more detailed investigation; therefore, experimental work in a Bridgman anvil unit has been initiated. If higher temperatures are required, the new die will be utilized to extend the temperature range of the investigation. Work on synthesis will be continued as soon as the die modifications are completed.

Fueled Zirconium Hydride Moderator

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

Additional hydrogen-absorption isotherms were obtained for the zirconium-25 w/o uranium alloy, and samples of this alloy were prepared for high-temperature X-ray diffraction studies. Analysis of the diffraction patterns of the 1 w/o and the 50 w/o alloys is in progress. The capsules containing specimens for radiation-damage studies are now in their fourth cycle in the MTR.

Structure and Pressure-Composition-Temperature Studies

Isotherms for the zirconium-25 w/o uranium alloy were determined at 716 C and at 730 C, and the boundary limits of the single-phase and two-phase regions were obtained from Sieverts plots. Data indicating the phase boundaries are given in Table F-1. These values, together with those obtained previously at 690 C, have resolved what appeared to be anomalies in the 710 C isotherm.

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

	716 C		730 C	
Phase Boundary	Hydrogen Absorption, cm^3 per g	Pressure, cm of mercury	Hydrogen Absorption, cm^3 per g	Pressure, cm of mercury
1	34.3	0.634	33.9	0.84
2	56.1	0.634	53.7	0.84
3	84.5	1.48	84.3	2.19
4	129.6	1.52	129.6	2.26

Three sets of 10-mil specimens of the 25 w/o alloy were hydrided to 10, 30, and 50 a/o hydrogen, based on the zirconium content. The specimens were sealed in quartz tubes under an argon atmosphere and homogenized at 1000 C. They will be used for the X-ray diffraction studies. Analysis of the data previously obtained for the 1 and 50 w/o alloys is in progress.

F-4

Radiation-Damage Studies

The nucleation capsules containing zirconium hydride and fueled specimens of zirconium-2 w/o uranium alloy hydride have been in the MTR approximately four cycles or 60 irradiation days. At present, typical readings from the thermocouples which monitor the specimen temperatures are as follows:

Capsule	Thermocouple 1	Thermocouple 5
BMI-20-1	1230 F	1290 F
BMI-20-2	980 F	1190 F

During the initial irradiation cycles, typical temperatures for Capsule BMI-20-1 were as follows: Thermocouple 1, 985 F, Thermocouple 5, 1370 F. At the end of the third irradiation cycle, the capsule was relocated and the temperatures tabulated above were established. Temperatures for Capsule BMI-20-2 originally were in the 1200-1300 F range; these have steadily declined to the levels tabulated above. If the present capsule temperatures continue to show a decrease, appropriate relocation measures will be taken.

At the end of the present cycle, MTR 115, the specimens will have achieved a uranium-235 burnup of approximately 10 w/o, which is half that required.

Irradiation Surveillance Program on Type 347 Stainless Steel

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

An irradiation surveillance program is being conducted to determine the effect of fast neutrons on the mechanical properties of AISI Type 347 stainless steel in support of the KAPL-33 loop which is to be installed in the ETR. It is planned to investigate the tensile, cyclic-strain fatigue, and impact properties after exposure to a neutron flux at about 120 F and 600 F. These properties will be determined after a time representing a 6-month period of service in the F-10 position and after successive 6-month intervals for up to 3 years of total exposure. Total integrated fast fluxes associated with these times in the F-10 position are approximately 2.4, 3.6, 4.8, 6.0, and 7.2×10^{20} nvt.

Eight capsules containing tensile, cyclic-strain fatigue, and impact specimens are being irradiated in the F-10-NE and F-10-SE position at process-water temperature (about 120 F). The capsules located in the position of highest flux now have received about 1.80×10^{21} nvt total integrated fast flux (neutrons having energies greater than 1 Mev). Previous plans to discharge two capsules in January have been changed and the tentative discharge date will be in March.

The dosimeter assemblies for monitoring the fast-neutron flux in the F-10 position of the ETR were inserted at shutdown for Cycle 10 during the week of December 8. The five assemblies prepared consist of thin-walled 3/16-in. aluminum tubing containing short nickel wires and crystals of ammonium sulfate in individual quartz vials. One

assembly contains eight nickel and eight ammonium sulfate dosimeters. One contains eight nickel dosimeters and the remaining three assemblies contain a nickel dosimeter each. The assembly with the eight nickel dosimeters, spaced to cover the entire length of a test position, was inserted in the east hole of the F-10 core-filler piece. The assemblies with one nickel dosimeter each were inserted in the north, south, and west holes. The one dosimeter was placed in these assemblies such that the flux at the mid-plane of the F-10 position will be measured. The assembly containing the eight nickel and eight ammonium sulfate dosimeters was to have been inserted in the center hole of the core-filler piece. However, the center dosimeter hole in the core-filler piece is directly below the lifting pin and access is attained only through the hole in the lifting pin. This hole cannot accommodate the dosimeter assembly so it is now planned to irradiate the assembly in the east hole during Cycle 11. A new lifting pin is being fabricated to permit future dosimetry in the center hole.

Capsule BMI-24-1, a nonlead capsule, was inserted in F-10-NW just below an ETR gamma heat probe for irradiation during Cycle 10. Except that it has no thermocouple, this capsule is similar in design to BMI-24-17, which attained temperatures in the 1000 to 1200 F range instead of the 600 F for which it was designed. The loading of the nonlead capsule was made to determine whether conditions that caused the excessive temperatures in BMI-24-17 continue to exist in the F-10 position. After exposure of one cycle it is to be returned to the Battelle Hot-Cell Facility where the temperature monitors and the aluminum insert surrounding the stainless steel specimens will be examined to determine whether or not melting occurred. This in turn will give some indication of the temperature obtained in the capsule during irradiation. It had also been expected that data from the ETR gamma probe might be correlated with these postirradiation observations, but the gamma probe did not function properly and data for comparison are not available. Loading of the remaining six nonlead capsules to be irradiated at 600 F will depend upon the results of this examination.

A third lead capsule, a duplicate in design of BMI-24-17 except for the thermocouple leads, is being constructed and is expected to be finished for insertion in the ETR during February. It will be used for temperature monitoring in the test position.

STUDIES OF ALLOY FUELS

R. F. Dickerson

The study concerned with the properties of niobium-uranium alloys has been continued. Hot-hardness data show that binary alloys containing between 10 and 60 w/o uranium soften very little up to 900 C. The elevated-temperature hardness is not affected by the impurity levels in the niobium used for this study. The results of fabrication tests indicate that all of the alloys studied can be cold rolled if the cast structure can be broken up. Based on these tests it seems that a fabrication temperature of 2550 F is satisfactory for alloys containing 10 and 20 w/o uranium, but it is estimated that temperatures on the order of 2700 F will be required for the other alloys. The alloys are protected from oxidation during the initial breakdown by welded molybdenum packs.

F-6

Although additions of zirconium to thorium-uranium alloys caused a marked improvement in corrosion resistance to 200 C water for short times, after 504 hr the corrosion rates reverted to about that of unalloyed thorium. Cerium, praseodymium, and yttrium are being considered as additions. It is postulated that these additions might produce a protective film. Increasing the uranium content in binary alloys above 5 w/o has no significant effect on hot hardness. Alloys prepared from Ames thorium are harder than similar alloys prepared from crystal-bar material. Specimens are being heat treated to determine effect of microstructure, and recrystallization studies and transformation are in process.

Development of Niobium-Base Alloys

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

The need for alternate cladding materials capable of withstanding high operating temperatures in advanced reactor designs of the EBR type motivates a consideration of alloys of the refractory elements. Niobium and niobium-base alloys are potentially suitable materials for cladding purposes in fast reactors. Based on the results of prior and recent investigation and the interest of ANL, several binary and ternary niobium-base alloys are being further developed with respect to application in the EBR. The following alloys have been selected for study: niobium-4.5 w/o zirconium, - 1 w/o chromium, -2 w/o chromium, -40 w/o titanium-10 w/o aluminum, -20 w/o titanium-1.5 w/o chromium, and -10 w/o tantalum-2 w/o chromium. Niobium and a vanadium-10 w/o titanium-1 w/o niobium alloy will be studied concurrently for comparison purposes.

The alloys will be prepared as 1-kg heats by consumable-electrode arc-melting techniques. The hot (1000-1400 C) and cold (room temperature) fabricability of each cast alloy will be studied along with cold fabricability following hot working. In order to determine the feasibility of single end-cap can closures, a sheet-to-sheet weldability test of each alloy will be performed. The thermal conductivity of each fabricable alloy will be measured from room temperature to approximately 800 C. The mechanical strength of all fabricable alloys will be evaluated by duplicate tensile tests at approximately 650 and 800 C. Three corrosion blanks of each alloy will be fabricated for sodium corrosion tests at ANL.

The niobium and alloying elements are being gathered and electrodes are being prepared for arc melting. Molybdenum capsules are being fabricated for canning the niobium alloys during hot-fabrication studies.

Development of Niobium-Uranium Alloys

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

Niobium-rich uranium alloys have potential as reactor fuels but very little data are available concerning their fabricability, mechanical properties, physical properties, and corrosion behavior. Determination of these properties and the effect of the major

impurities on them will yield data necessary for the proper evaluation of these alloys as possible reactor fuels.

The effect of impurities is being investigated by using three grades of niobium as base materials: niobium containing less than 0.17 w/o zirconium and 700 ppm oxygen, niobium containing 0.7 w/o zirconium and 600 ppm oxygen, and niobium containing 100 ppm zirconium and less than 300 ppm oxygen. The alloys of interest are uranium-40, -50, -60, -70, -80, and -90 w/o niobium, each of which has been prepared with all three grades of niobium, making a total of 18 alloys.

Hot-hardness data show that very little softening of these alloys occurs up to 900 C. The hardness values at 900 C range from 176 DPHN for a uranium-40 w/o niobium alloy to 95.1 DPHN for a uranium-90 w/o niobium alloy. These hot-hardness values are for alloys prepared from niobium containing less than 300 ppm oxygen and less than 100 ppm zirconium. Although there is a difference in room-temperature hardness between the low-oxygen low-zirconium niobium and the other two types, this difference does not exist at elevated temperatures. The elevated temperature hardness of the uranium-niobium alloys does not seem to be affected by the impurity levels present in the niobium used for this study.

When hardness versus temperature is plotted on semi-log graph paper a straight line is produced with a slight slope showing decreased hardness as the temperature is increased. In most alloy systems, there exists a temperature at which the alloy begins to soften rapidly. In the uranium-niobium alloys this "break" in the hot-hardness curves does not occur below 900 C.

All melting and casting of alloys has been completed and full effort has been turned to investigating fabrication characteristics of these alloys. Buttons of each composition prepared from niobium containing less than 300 ppm oxygen and less than 100 ppm zirconium were wrapped in 0.005-in. molybdenum sheet, placed in a Type 304 stainless steel pack, and rolled at 2400 F. Metallographic examinations of the uranium-40, -50, -60, and -70 w/o niobium alloys indicate that these alloys alloyed with the stainless steel pack. The uranium 80 and 90 w/o niobium alloys were hot rolled approximately 50 per cent without cracking. The as-fabricated uranium-90 w/o niobium alloy showed excellent room-temperature ductility. If use is to be made of stainless steel packs then thicker molybdenum sheet or lower fabrication temperatures must be employed.

Table F-2 shows the results of fabrication tests of uranium-50, -60, -70, -80, and -90 w/o niobium buttons. These buttons were prepared from niobium containing less than 300 ppm oxygen and less than 100 ppm zirconium. The as-cast 1/4 by 1/2 by 1-in. buttons had been heated to 900 C and furnace cooled in a hot-hardness machine. The results of these tests indicate that if the cast structure is broken up in some manner (it appears that a 50 per cent reduction will do this) then these alloys can be cold rolled.

Table F-3 shows the results of fabrication of six molybdenum packs containing uranium-70, -80, and -90 w/o niobium alloys. The packs were prepared by reaming a thick-walled 2-1/2-in.-OD molybdenum tube until an ID of 2 in. was obtained. Cover plates for 3/8-in. lengths of this tube were cut from 3/16-in. molybdenum sheet and welded in place. Wafers of uranium-niobium alloy 3/8 in. thick were cut and placed in

TABLE F-2. RESULTS FROM ROLLING BARE URANIUM-NIOBIUM ALLOYS

Nominal Composition (Balance Uranium), w/o	Temperature, F	Reduction[a], per cent	Remarks
90 niobium	Room	90	Severe cracking until 60 per cent reduction was obtained; after this, no further cracking was noted; material appears to be quite ductile
90 niobium	572	50	Edge cracking less severe than for above cold-rolled material
	Room	40	No new cracks, existing cracks opened; some sound material obtained
80 niobium	572	50	Severe edge cracking; air cooled
	Room	40	No new cracks; existing cracks opened extensively; material has fair ductility
80 niobium	932	50	Edge cracks not as severe as for above material at 572 F
	Room	40	Some new cracks appeared; fair ductility
70 niobium	932	50	Quite severe cracking
	Room	40	New cracks noted; however, these were not extensive; ductility only fair
60 niobium	1472	50	Extremely severe edge cracking
	Room	20	Many new cracks noted; alloy possessed better ductility than expected
50 niobium	1472	40	Severe fracture; no cold rolling attempted with this alloy

(a) Reductions given represent only the reductions occurring at each temperature. These are subsequent operations, so that a 50 per cent reduction followed by a 40 per cent reduction produces a total reduction of 70 per cent.

the packs prior to closure. The molybdenum packs were welded in an inert atmosphere. The packs were then fabricated as indicated in Table F-3 at 2550 F. The surfaces of the alloys containing uranium-70 w/o niobium have the appearance of cold-worked material. Metallography is being done on these alloys to confirm this.

TABLE F-3. FABRICATION OF MOLYBDENUM-CLAD PACKS AT 2550 F IN AN ARGON ATMOSPHERE

Nominal Composition (Balance Uranium), w/o	*Method of* Fabrication	Total Reduction of Alloy, per cent	Remarks
90 niobium[a]	Rolling	75	Sound, 0.095-in. sheet obtained
90 niobium[b]	Rolling	53	Molybdenum pack broke and specimen was removed from test and quenched when oxidation became apparent
80 niobium[a]	Rolling	48	*Ditto*
80 niobium[b]	Forged and rolled	78	Pack forged from 3/4-in. to 1/4-in. thickness and then rolled; sound sheet was obtained
70 niobium[a]	Forged and rolled	75	Pack forged from 3/4-in. to 1/4-in. thickness and then rolled; cracks present in sheet
70 niobium[b]	Forged and rolled	77	Pack forged from 3/4-in. to 1/4-in. thickness and then rolled; cracks present in sheet

(a) Niobium used contains 700 ppm oxygen 0.17 w/o zirconium.
(b) Niobium used contains 600 ppm oxygen 0.7 w/o zirconium.

It is suspected that temperatures in the neighborhood of 2700 F will be necessary for hot working of most of these alloys. The uranium-80 and -90 w/o niobium alloys were successfully fabricated at 2550 F and yielded sound sheet material. The sheet obtained from all six alloys is being sectioned into strips which will be warm rolled at temperatures ranging from 752 to 1832 F to study fabrication characteristics. Hardness measurements and metallography will be used extensively to assure that maximum information is obtained from each test.

Wafers 0.100 in. thick of the uranium-40, -50, and -60 w/o niobium alloys are being cut for fabrication studies in the as-cast condition. These wafers will be rolled at varying temperatures (752 to 1832 F) to determine if ductile-brittle transition temperatures exist for these alloys.

Larger scale fabrication tests of the uranium-80 and -90 w/o niobium alloys in molybdenum jackets have been started and material for corrosion and mechanical testing should be available soon.

Development of Thorium-Uranium Alloys

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

A program is being conducted to investigate methods for improving the irradiation behavior and corrosion properties of thorium-uranium alloys. For this purpose the

effects of processing variables and alloying on thorium-uranium alloys are being studied. Alloying additions being investigated include zirconium, niobium, and molybdenum, the zirconium being intended primarily to alloy with the thorium, and the niobium and molybdenum being added to alloy with the uranium phase.

Alloys containing up to 25 w/o zirconium and 25 w/o uranium and similar alloys with small quaternary additions of niobium have been subjected to corrosion tests in 200 C water. Specimens were tested in both the water-quenched and furnace-cooled conditions of heat treatment. For short times on test, zirconium additions appeared to produce a marked improvement in corrosion resistance, with specimens water quenched from 1000 C showing the greatest improvement. However, after 504 hr on test, corrosion rates varying from 0.79 to 2.2 mg/(cm^2)(hr) have been obtained on all specimens. Since these rates do not represent any significant improvement over those for pure thorium at this temperature, the specimens have been removed from test. Additions of cerium, praseodymium, or yttrium are now being considered for the possibility of producing a protective oxide film.

Hot-hardness measurements have been obtained on binary thorium alloys containing 5 to 20 w/o uranium prepared from both iodide and Ames thorium. Increasing the uranium content above 5 w/o has no significant effect on hardness. Alloys prepared from Ames thorium exhibit hardnesses 26 to 39 DPHN higher than those prepared from iodide thorium; a higher impurity content in Ames thorium is probably responsible. No significant differences between the hardnesses of arc-melted and induction-melted materials were observed.

Below 500 C, ternary alloys containing either molybdenum or niobium had hardnesses which were 16 to 34 DPHN lower than corresponding binary alloys. This suggests that these additions may either change the uranium distribution in the alloys, perhaps decreasing the amount of uranium contained in the thorium, or possibly gettering impurities in the thorium phase.

Specimens are now being heat treated to determine the effect on microstructure. Recrystallization studies of cold-worked binary alloys are also being performed as are transformation studies of the gamma-uranium phase in the alloys containing molybdenum and niobium additions.

FISSION-GAS RELEASE FROM REFRACTORY FUELS

J. B. Melehan and F. A. Rough

The objective of this program is to study and understand the in-pile release of fission gas from refractory fuels. The most important such problem at present is the release of fission gas from uranium dioxide, and it is this specific problem that is to be studied.

Numerous experiments have been performed analyzing the release of fission gas from uranium dioxide, and reasonably consistent results have been obtained within a given series of experiments. However, widely varying results have been obtained

between different experiments. The data are usually interpreted in terms of a diffusional model, which assumes that the uranium dioxide can be represented by little spheres of uniform size. Release is assumed to be instantaneous when diffusion has occurred from the individual spheres.

An important step in this program is the selection of the appropriate grades of uranium dioxide and the characterization of its microstructure and crystal structure. This type of background coupled with in-pile experimentation under controlled but flexible conditions is expected to provide improved insight into the variables affecting the release of the gases. In addition to the in-pile experiments, various postirradiation studies will be required, and supplementary experiments as needed will be planned.

At present, the principal effort is directed toward the design and construction of suitable apparatus for continuous measurements of fission-gas release during in-pile operation.

GENERAL FUEL-ELEMENT DEVELOPMENT

S. J. Paprocki

An investigation is being made of cermet fuel materials consisting of from 60 to 90 volume per cent of UO_2, UN, or UC dispersed in stainless steel. These are promising fuel materials as they contain a relatively large volume loading of uranium and in comparison to ceramic materials they possess higher strength, improved thermal conductivity, and resistance to thermal shock. Densities exceeding 90 per cent of theoretical have been obtained by a combined swaging-gas pressure bonding process and also by direct gas-pressure bonding of green compacts.

The gas-pressure-bonding process is being investigated for the cladding and joining of niobium and molybdenum fuel elements and assemblies. Fuel elements consisting of a niobium cladding and uranium dioxide core have been bonded at 2200 F for 3 hr at 10,000 psi of helium. The niobium-niobium bonds exhibited excellent bond strength, although metallographic examination revealed areas along the interface where complete grain growth was not achieved.

Fuel specimens consisting of dispersions of fully enriched UN and UC in stainless steel clad with stainless steel are being irradiated. The irradiation resistance of these fuel specimens will be compared with that of stainless-UO_2 fuel specimens containing an equivalent uranium-235 content that have been irradiated under similar conditions. It is anticipated that the specimens containing UN and UC will be more resistant to irradiation because these compounds are of higher density and require a lower volume loading for an equivalent uranium-235 content.

Studies are in progress to determine the factors affecting solid-state bonding after sufficient pressure has been exerted to obtain intimate contact between the bonding surfaces. The studies are being conducted utilizing both copper and zirconium diffusion couples.

F-12

Fabrication of Cermet Fuel Elements

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

Techniques of fabricating cermets containing 60 to 90 volume per cent fuel to densities of 90 per cent of theoretical or higher include pressure bonding and hot pressing. Mechanical and physical measurements are being made on fabricated cermets to evaluate these fabrication techniques.

Green cores 0.5 in. in diameter by 1.75 in. long were pressed at 30 tsi. The cores contained mixtures of 70 volume per cent UO_2 dispersed in Type 302B stainless steel, 70 volume per cent UO_2 dispersed in molybdenum, and 80 volume per cent UO_2 dispersed in Type 302B stainless steel. The respective green core densities were 67, 69, and 68 per cent of theoretical. The first and second cores were sealed in stainless steel tubes and swaged at 800 C to obtain an 18 per cent reduction in cross section subsequent to pressure bonding for 3 hr at 2300 F under a helium gas pressure of 10,000 psi. The third core, 80 volume per cent UO_2, was sealed in an evacuated stainless steel tube and pressure bonded with the first and second cores. Densities after pressure bonding of the first, second, and third cores were, respectively, 95, 93, and 94 per cent of theoretical. The cladding was removed from the third core, and its longitudinal electrical resistivity is being measured.

The transverse and longitudinal electrical resistivities have been measured on a 74 volume per cent UO_2-26 volume per cent stainless steel hot-press-forged cermet having a density 85 per cent of theoretical. A value of 1790 microhm-cm was obtained in the longitudinal direction (perpendicular to the direction of pressing) as compared with 8040 microhm-cm in the transverse direction. Additional measurements of this type are being made on hot-press-forged cermets of various compositions.

Modulus-of-rupture values of 8850 and 7000 psi, respectively, have been obtained on UO_2 with densities of 95 and 93 per cent of theoretical as compared with 22,200 psi on a hot-press-forged cermet of 80 volume per cent UO_2-molybdenum having a density of 94 per cent of theoretical.

A core of approximately 80 w/o UO_2-niobium was compacted at 30 tsi and sintered 2 hr at 2750 F in a vacuum. The UO_2 powder used was coated with niobium by a vapor-deposition method to a thickness necessary to obtain 20 w/o metal. Sintering resulted in no increase in density nor change in dimensions; however, the strength of the compact was increased. Metallographic examination showed bonding of the niobium coatings where contact was made during cold pressing. The sintered core is now being prepared for pressure-bonding studies.

In addition, green and hot-press-forged cores of 80 volume per cent UN and UO_2 dispersed in various matrix materials are being prepared for pressure-bonding studies as well as to evaluate various particle sizes between the range of minus 100 plus 325 mesh.

F-13

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

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

Because molybdenum and niobium retain their strength at elevated temperatures and possess favorable nuclear properties, they are potential cladding and structural materials for high-temperature-reactor application. In inert-gas-cooled or some liquid-metal-cooled reactors, the niobium and molybdenum can be utilized in bare form. The gas-pressure-bonding process is being investigated as a method of fabricating fuel elements and assemblies clad with these materials. In this process, the components are fabricated to final size, then edge welded or assembled in a leaktight container, and subjected to gas pressure at an elevated temperature. The gas pressure brings the components into the intimate contact that is necessary for diffusion to occur and form a solid-state metallurgical bond.

The studies concerned with the determination of optimum conditions for the self-bonding of niobium have been continued. Specimens with machined surfaces possessing an 80 rms finish were pressure bonded at 2200 F for 3 hr at 10,000 psi of helium. These specimens exhibited excellent bond strength; however, bond areas with incomplete grain growth were observed. Although the bonded specimens possessed physical and mechanical properties comparable with unbonded niobium, it is planned to bond additional specimens with different surface treatments in an attempt to refine the bond conditions sufficiently to consistently produce bonds possessing complete grain growth.

Niobium-clad fuel specimens containing uranium dioxide cores were pressure bonded at 2200 F for 3 hr at 10,000 psi. The niobium components were pickled in a solution of 95 parts H_2SO_4, 4.5 parts HNO_3, 0.5 part HF and 18.8 g per liter of Cr_2O_3. This treatment resulted in excellent bonds in previous tests. Both specimens exhibited excellent mechanical bond strength; however, complete grain growth across the interface was not achieved. The characterization of the bond areas with incomplete grain growth has not been defined. These areas apparently are metallurgically bonded, as they are strong and corrosion resistant; however, they are considered to be undesirable, as they constitute potential planes of weakness.

Molybdenum fuel-plate specimens are being pressure bonded at 2300 to 2400 F for 3 hr at 10,000 psi. It appears that for the thicker material these higher temperatures will be required. Molybdenum plates machined to an 80 rms 0.035-in. thickness and surface conditioned prior to bonding by pickling in a solution of 95 parts H_2SO_4, 4.5 parts HNO_3 0.5 part HF, and 18.8 g per liter of Cr_2O_3 exhibited spotty bonds with complete grain growth where intimate contact was made. There were, however, a number of voids where the machined surfaces did not come into intimate contact. It appears that a greater degree of deformation is required for the molybdenum components in order to achieve sound metallurgical bonds.

Additional molybdenum- and niobium-clad fuel-plate-type specimens containing cores of pure uranium dioxide and cermets are being assembled for bonding.

F-14

The Irradiation of UC- and UN-Stainless Steel Dispersion-Type Fuel Elements

A. W. Hare, R. B. Price, D. L. Keller, and R. F. Dickerson

The objective of this study is to evaluate the irradiation resistance of dispersion fuel elements consisting of uranium carbide or uranium nitride fuel dispersed in a stainless steel matrix clad with stainless steel. The behavior of these specimens will be compared with stainless steel-UO_2 dispersion elements containing an equivalent amount of uranium-235 which have previously been irradiated to similar levels of burnup in the same approximate temperature range. It is anticipated that these specimens will be more resistant to irradiation than specimens containing UO_2 because of the lower volume loading of UN or UC for an equivalent uranium-235 content.

Twelve test specimens, six specimens of 24 w/o uranium mononitride in a matrix of stainless steel clad with stainless steel, and six specimens of 24 w/o uranium monocarbide in a matrix of stainless steel clad with stainless steel, have been fabricated and encapsulated in three irradiation capsules, and are currently being irradiated at the MTR. Each capsule contains two uranium nitride specimens and two uranium carbide specimens. Each specimen is about 1-1/2 in. long by 11/16 in. wide by 47 mils thick. The cladding thickness is approximately 8 mils.

The irradiation capsules were designed for specimen-core temperatures of about 1600 F. The irradiations are being accomplished in the A-38NE position of the MTR. Since the capsules are equipped with thermocouples, only one capsule will be irradiated in this position at a time.

Following is the irradiation schedule for the three capsules:

		Insertion		Discharge	
Capsule	MTR Cycles Required	MTR Cycle	Date	MTR Cycle	Date
BMI-18-1	2	113	11-10-58	115	12-22-58
BMI-18-2	1	115	12-22-58	116	1-12-59
BMI-18-3	4	116	1-12-59	120	4-6-59

It is estimated that the thermal flux in the A-38NE position in the MTR is about 1.7×10^{14} (thermal) nv, and with the capsule design which has been used, the specimens should undergo burnups of about 5, 10, and 15 a/o of the uranium-235 present.

Capsule BMI-18-1 was inserted in the MTR on schedule and was discharged on December 22. This capsule should be returned to Battelle for postirradiation examination during January. Although the complete thermocouple data and MTR reactor power-level data for MTR cycles 113 and 114 are not yet available, it is estimated that during Cycle 113 the four specimens in this capsule operated at a core temperature of from about 1525 F to an estimated maximum of about 1750 F.

F-15 and F-16

Capsule BMI-18-2 was inserted into the MTR on schedule and is currently being irradiated in the MTR in the A-38NE position. No temperature data on the specimens in this capsule are available at this time.

Factors Affecting Pressure Bonding

G. W. Cunningham and J. W. Spretnak

Solid-phase bonding of metals by the application of heat and pressure is being studied in an attempt to establish a bonding mechanism. Copper-to-copper and zirconium-to-zirconium bonds are being used for the initial work.

Some 1/2-in. copper squares have been hot pressed to establish the pressure and temperature range for bonding studies. A specimen pressed at 10,000 psi for 2 hr at 450 F showed partial bonding, while a specimen pressed at 2000 psi for 2 hr at 400 F was not bonded even though some plastic deformation occurred. The bond line of a specimen pressed at 1460 F for 2 hr at a pressure of 10,000 psi could not be detected by examination under the light microscope, although grain size was smaller at the interface area. Specimens pressed at 740 and 1100 F for 2 hr at 10,000 psi are being used to establish procedures for measuring density and electrical resistivity and have not been examined under the microscope. A second series of specimens is being prepared to study the effect of temperature in the range 400 to 1500 F.

Poorly bonded Zircaloy specimens have been cold rolled, annealed at 1400 F for 2 hr, and examined under the light microscope. Elongated void areas could be detected at the bond interface on specimens cold reduced 40 and 50 per cent in thickness, but the bond line could not be detected on specimens cold reduced 77 and 85 per cent in thickness.

G-1 and G-2

G. FATIGUE STUDIES OF INCONEL AND INOR-8

R. G. Carlson

Fatigue Studies of Inconel

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

During December, stress-cycling tests were continued on specimens at 1500 F cycled at 1 cps. The stress-strain traces and the hysteresis curves obtained are being analyzed to determine the relationship among significant variables.

During January, tests will be continued at both 1500 F and 1300 F with cycling at 1 cps.

Fatigue Studies of INOR-8

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

During December, tests were continued at 1500 F and 100 rpm. Because of specimen bending at high frequency and high loads, the maximum speed for high-frequency tests has been decreased from 10,000 to 3000 rpm. Tests at 1500 F are now being performed at 3000 rpm.

During January, tests will be continued at 1500 F at both 100 and 3000 rpm. Upon completion of the tests at 1500 F, specimens will be tested at 1100 F.

H-1

H. PHYSICAL RESEARCH

F. A. Rough

Projects of research concerning the reaction of niobium with nitrogen and the constitution of uranium-niobium alloys, reported bimonthly, are again included in this section. The balance of the projects supported by the AEC Division of Research are reported on alternate months.

In the study of niobium-nitrogen reactions, kinetics data have been obtained in the range of 400 to 1400 C. A cubic law appears to apply at 400 to 600 C and parabolic behavior is observed at 700 to 1400 C. The reaction products and, therefore, the mechanism appear to vary within the 700 to 1400 C range.

In the study of the constitution of uranium-niobium alloys, diffusion couples have been prepared and are being examined by the electron-probe microanalyzer. It is expected that both constitution and diffusion data will be obtained. Efforts are concentrated in the gamma-phase immiscibility or gamma-1 plus gamma-2 region.

Constitution of Uranium-Niobium Alloys

S. G. Epstein, A. A. Bauer, and R. F. Dickerson

A study of the constitution of uranium-niobium alloys is being conducted. Work has been concentrated on establishing the composition limits of the gamma-phase immiscibility gap in the system. Diffusion couples have been prepared and heat treated for this purpose. In addition to determining the phase limits by analysis of the resulting composition-versus-distance curves, it is planned to calculate diffusion coefficients for the system.

The diffusion couples are being analyzed by an electron-probe microanalyzer. Analysis of a sample annealed at 925 C for 70 hr has been completed and a corrected composition curve has been obtained. No break in the curve suggesting an immiscibility gap is evident, indicating either that the miscibility gap does not extend to this temperature or that it is too small at this temperature to be detected. Uranium-10 and -50 w/o niobium specimens were employed as standards to yield the final corrected curve.

Diffusion couples annealed at 1000 C for 50 hr, at 900 C for 68 hr, and at 700 C for 2500 hr are also being analyzed by the electron probe. It is planned to obtain and consider these data before calculations of diffusion coefficients are performed.

H-2

Niobium-Gas Reactions

W. M. Albrecht and W. D. Goode

Fundamental studies are being made of the kinetics and mechanism of the reaction of nitrogen with niobium. Various phases of the study include measuring the diffusion coefficient and solubility of nitrogen in the metal.

Several kinetic experiments have been made in the range 400 to 1400 C at a 1-atm nitrogen pressure on both cylindrical and sheet specimens. The data were obtained with a modified Sieverts apparatus (volumetric technique) and a microbalance (gravimetric technique). A summary of the kinetic experiments is given in Table H-1. At 400 to 600 C the data followed most nearly a cubic law, $w^3 = k_c t$, where w is the amount of nitrogen reacting per unit surface area in μg per cm^2, k_c is the cubic rate constant, and t is time in sec. For an exact cubic rate, a plot of the logarithm of w against the logarithm of t is a straight line with a slope of 0.33. The experimental slopes ranged from 0.30 to 0.38. From 700 to 1400 C the reaction followed a parabolic rate ($w^2 = k_p t$) where the log w - log t slope is 0.50. On the basis of data obtained thus far, there appears to be a change in the reaction mechanism in the parabolic range. X-ray diffraction measurements showed the reaction product at 800 C to be Nb_2N and that at 1400 C to be NbN. As previously reported, both Nb_2N and NbN are produced at 1000 C. Additional kinetic and X-ray diffraction data will be obtained to establish the temperature at which the change in the parabolic reaction mechanism occurs.

Diffusion experiments have been made at 1200, 1300, and 1400 C using a concentration-gradient technique in which analysis is made of a concentration gradient prepared in a cylindrical specimen. The terminal or maximum solubility of nitrogen in the metal was calculated from the concentration gradient. A summary of diffusion and solubility data obtained in this program and from the literature is given in Table H-2. It can be seen that the diffusion coefficients of this study are in the range of those obtained from internal-friction measurements.[1,2] In contrast, the solubility (from internal-friction measurements) reported by Ang and Wert[3] is much lower than value from this research for the same temperature. Solubilities will be checked by metallographic examination of structures resulting from direct saturation of niobium.

(1) Ang, C. Y., Acta Met., 1, 123 (1953).
(2) Powers, R. W., and Doyle, M. V., J. Metals, 9, 1285 (1957).
(3) Ang, C. Y., and Wert, C., Trans. AIME, 197, 1032 (1953).

H-3 and H-4

TABLE H-1. KINETIC DATA FOR THE REACTION OF NITROGEN WITH NIOBIUM

Temperature, C	Slope of Log-Log Plot	Rate Constant	
		Parabolic, $(\mu g/cm^2)^2/sec$	Cubic, $(\mu g/cm^2)^3/sec$
400	0.38	--	6.4×10^{-3}
500	0.37	--	1.3×10^{-1}
600	0.30	--	5.1×10^{-1}
700	0.46	9.4×10^{-2}	--
	0.54	6.3×10^{-2}	--
800	0.46	2.8×10^{-1}	--
900	0.56	6.9×10^{-1}	--
1000	0.49	2.1	--
1200	0.53	2.9×10^{2}	--
1300	0.47	1.1×10^{3}	--
	0.54	1.1×10^{3}	--
1400	0.54	2.0×10^{3}	--

TABLE H-2. DIFFUSION AND SOLUBILITY OF NITROGEN IN NIOBIUM

Temperature, C	Diffusion Coefficient, 10^{-7} cm^2 per sec			Terminal Solubility, a/o nitrogen	
	This Work	Reference (1)	Reference (2)	This Work	Reference (3)
1200	1.32	1.9	0.48	1.5	0.35
1300	3.05	4.3	--	2.4	--
1400	5.48	8.9	--	2.7	--

I-1

I. SOLID HOMOGENEOUS FUELED REACTORS

W. H. Goldthwaite

An evaluation of 1-1/2-in.-diameter spherical fuel elements for a proposed pebble-bed reactor is in progress. The evaluations include capsule irradiations in the BRR, preceded and followed by mechanical tests of strength and abrasion resistance and other tests to characterize the specimens and determine the effects of radiation. A capsule containing six spherical graphite specimens was discharged from the BRR in December. The capsule was opened, fission-gas samplings were made, and examination of the specimens was begun.

Postirradiation Evaluation of Spherical Fueled-Graphite Specimens

R. J. Burian and J. E. Gates

This program involves the examination and evaluation of six 1.5-in.-diameter fueled (UO_2) graphite spheres after irradiation. The spheres were canned in pairs, each pair having the UO_2 incorporated in them by different methods. The three cans were encapsulated and irradiated in the Battelle Research Reactor at specimen surface temperatures near 1100 F. The irradiation of the spheres was completed on December 1, 1958. The capsule has been opened and the examination of the spheres has been initiated.

The two spheres contained in the uppermost can (Can A) in the capsule were fabricated by a vendor. These spheres were fabricated by mixing UO_2 particles with graphite flour and binder pitch and forming to the required shape. The two spheres in the center can (Can B) were prepared at Battelle by dispersing UO_2 particles in a spherical graphite core 1 in. in diameter. An unfueled shell 0.25 in. thick was formed around the fueled core. Another vendor fabricated the remaining two spheres in Can C by machining them from extruded graphite. The fuel was added to these spheres by soaking them in uranyl-nitrate solution and heat treating to convert the nitrate to UO_2. The fuel loading of the six spheres was between 4.31 and 4.71 g of 93 per cent enriched uranium.

The irradiated capsule was opened by use of a power hack saw and remote milling machine. The ends were first removed with the saw, and then the capsule shell was split lengthwise on two sides with the mill. During the milling operation, the capsule slipped in the vise, allowing the milling tool to penetrate the middle can containing the two spheres fabricated at Battelle. The two halves of the capsule shell were pulled apart, freeing the inner cans.

Each of the three inner cans was recovered and examined. Can B (the middle can) was punctured during the milling operation, releasing any contained gases. The other two cans were in good condition.

Cans A and C were punctured and the contained gases sampled for fission gas. A leak, later confirmed by mass-spectrometer measurements, occurred in the vacuum system during the sampling of Can A, invalidating the fission-gas measurements. The gas contained in Can C was successfully sampled and has been determined to contain a significant amount of xenon-133. The radiation emitted from the highly radioactive xenon-133 prevents the measurement of the krypton-85 activity, which is considered to provide more reliable data for fission-gas determinations. The gas samples from Can C will be stored until the short-lived xenon-133 radioisotope decays to a level at which the krypton-85 activity can be measured.

Each canister was opened by slitting the sides and ends with the milling machine. The spheres were recovered along with the graphite flour used to fill the annulus around them. The neutron dosimeter wires included in each can were also recovered and are being analyzed.

The spheres were visually inspected. Only one specimen, BMI-12, fabricated at Battelle, was found to be cracked. All of the specimens were photographed, measured, and weighed. Five of the specimens, excluding BMI-12, were abrasion tested. One specimen from each pair was impact tested while the other one was compression tested. All of these tests were designed especially for this examination and were used in the preirradiation testing. A cylindrical core was drilled from Specimen BMI-12 for analysis of uranium distribution.

Evaluation of the data obtained from the various tests is in progress. Radiochemical analyses of the neutron dosimeters, the graphite flour, and the core sample have not as yet been initiated.

Encapsulation and Irradiation

G. E. Raines and J. H. Stang

During December, estimates of the performance of spherical fueled graphite specimens during irradiation were revised, indicating somewhat higher heat generations and temperatures than the original estimates which appeared in BMI-1301. These new values appear in Table I-1 and have arisen because of a hot-cell observation of strong adherence of the stainless steel inner shells to the graphite specimen-housing blocks and carburization of the stainless steel parts. This indicated contact of these parts during irradiation and thus precluded the possibility of the occurrence of the helium gap that was assumed would appear at temperature as a result of differential thermal expansion of the graphite and stainless steel parts. Apparently the 70-mil-thick outer shell (cold) gave the system sufficient rigidity to prevent significant thermal expansion.

Also during December machine-shop fabrication of parts for a second capsule with only minor changes from the first one was initiated. According to present plans, this irradiation is scheduled to start during January and continue for 6 weeks.

TABLE I-1. PERFORMANCE ESTIMATED FOR THE SIX SPECIMENS IN CAPSULE BRR-SP-1

Nominal uranium (fully enriched) content is 4-3/4 g per specimen.

Specimen Designation[a]	General Description	Estimated Heat Generation[b], kw		Estimated Effective Flux, 10^{13} nv		Estimated Surface Temperature at Equator of Specimen[c], F		Burnup, per cent of uranium-235 atoms by fission
		Initial	"Equilibrium"	Initial	"Equilibrium"	Initial	"Equilibrium"	
NC-86	Uniform mixture of UO_2 and graphite	1.9	1.7	1.0	0.9	1350	1220	1.5
NC-87	Uniform mixture of UO_2 and graphite	2.1	1.8	1.1	1.0	1440	1290	1.6
12[d]	Solid 1-in.-diameter spherical core (uniform mixture of UO_2 and graphite) surrounded by 1/4-in.	--	--	--	--	--	--	--
11	unfueled graphite cladding	2.0	1.7	1.1	0.9	1440	1200	1.5
E-16	Impregnated specimen with UO_2 uniformly distributed	2.3	1.9	1.4	1.1	1580	1330	1.9
E-13	Impregnated specimen with UO_2 uniformly distributed	2.2	1.9	1.3	1.1	1540	1320	1.8

(a) The specimens were located in the capsule in the order listed. The flux peaks (approximately 3.6×10^{13} nv, measured at the center of empty position) about midway between Specimens 11 and 12 and drops off toward the ends. The Battelle-fabricated specimens were located in the peak-flux zone because their relatively concentrated fuel loading was expected to result in a relatively high perturbation.

(b) Heat generations were estimated from temperature data and heat-transfer considerations. The reference thermocouples are located in graphite blocks in the equatorial planes. Initial temperatures were relatively high with "equilibrium" values occurring after the first 40 to 50 hr of each cycle. These blocks serve as the specimen housings, each specimen in them cushioned in a graphite-powder annulus 30 mils thick.

(c) These temperatures were estimated by using reference data in (b) above and a powder thermal conductivity based on packing density. Surface temperatures measured for Specimen NC-86 about 60 deg above the equatorial plane were about 1400 F initially and 1260 F at "equilibrium".

(d) No estimates are given for Specimen 12 because the core was nonconcentric with the shell.

J-1

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

C. L. Peterson, P. D. Miller, 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 Darex, Sulfex-Thorex, Zirflex, and Fluoride-Volatility processes of nuclear-fuel recovery has been continued.

Localized attack on titanium steam tubes exposed to Darex dissolver solutions appears to be connected with defective areas in the tubes. This study is being continued.

Heat treatment of Ni-o-nel for 0.5 hr at 1850 F with an air quench following welding has produced the most resistance to Thorex solutions of any treatment studied.

The conditions prevailing during dissolution of Type 304 stainless steel in boiling 6 M H_2SO_4 give corrosion rates in the range of 2 to 3 mils per month for Ni-o-nel specimens exposed to the vapor. Cathodic protection of liquid-phase specimens is afforded by the dissolving stainless steel.

Scouting experiments have shown the Zirflex decladding solution (6 M NH_4F, 1 M NH_4NO_3) to be excessively corrosive to Type 347 and Carpenter 20 Cb stainless steels and Ni-o-nel.

Raising the temperature from 650 to 700 C practically doubles the corrosiveness of the equimolar $NaF-ZrF_4$ salt. A silver specimen showed some promise in this salt but not in a final composition of this system. Temperature effects on the $NaF-LiF-ZrF_4$ system are also being studied.

The Darex Process

The Darex process is proposed for the recovery of uranium from fuel elements containing stainless steel as a diluent or cladding. Dissolution is accomplished in dilute aqua regia. Following this, the chlorides are stripped from the solution which is then so adjusted that the uranium may be recovered by conventional methods of solvent extraction.

Dissolver Studies With Titanium

Several specimens selected from the 2008-hr exposure in the flowing dissolver were sectioned and examined metallographically. No apparent attack was observed at crevices, weldments, or around scribed marks. The caustic cleaning procedure used during this exposure appears to be a safe operation from the standpoint of equipment corrosion.

Metallographic examinations were also made of sections cut from the titanium steam tubes which had been exposed to Initial and Beginning Darex dissolver solutions.

Sections were cut through the areas containing the fissures and crevices mentioned in BMI-1304. The bottoms of these defects appear flat or rounded and show no cracks proceeding farther into the metal. The general impression is that these defects may have been present from the outset of the exposure and were only opened and revealed by corrosion. However, to provide a better basis for judgment, these tubes have been re-welded and are being returned for continued exposure in new solutions to see if additional signs of localized attack develop.

Exposure of a tube to Middle Darex dissolver solution was halted after 2000 hr. Inspection of this tube has not been completed, but there is no gross evidence of severe attack.

Fission-Product-Recovery Solutions

Horseshoe-shaped specimens have been fabricated from Types 304 ELC and 347 stainless steel. The specimens are fashioned with longitudinal weldments which have been machined to a flat surface, while the keepers contain a transverse weld bead. These specimens, along with welded coupon-type specimens, are being exposed to boiling 3 M HNO_3 containing 60 g per liter dissolved stainless steel and 800 ppm chloride. If stress-corrosion cracking develops after continued exposure, lower chloride values will be studied until the threshold concentration for chloride is established.

The Sulfex-Thorex Process

Stainless-clad fuel elements of thorium or thoria could be dejacketed by dissolution in sulfuric acid. The thorium or thoria would then be dissolved by a solution of 13.0 M HNO_3, 0.05 M F^- containing an addition of aluminum to mitigate corrosion. The Final Thorex solution would be about 8.5 M HNO_3, 0.05 M F^-, 1.0 M $Th(NO_3)_4$, and, in addition, any aluminum that may have been introduced at the start.

Experiments With Carpenter 20 Cb

No experiments were conducted this month with Carpenter 20 Cb.

Experiments With Ni-o-nel

Stabilized Ni-o-nel which had been heat treated at 1650 F, prior to welding, had corrosion rates after 500 hr of 1.5 to 2.5 mils per month. These rates were increasing with continued exposure to boiling Initial Thorex solution with a 0.2 M aluminum addition. The surfaces and weldments showed considerable attack. General attack over the surface also developed after continued exposure of similar specimens, welded by the supplier, and heat treated at 1650 F after welding. The weldments of these latter specimens were severely attacked. A spectrographic comparison of one of the weldments with the parent metal indicated that a flux-coated niobium-stabilized weld rod had been used. Welding by the inert-gas process using a Ni-o-nel No. 65 wire, which has

approximately the same composition as the titanium-stabilized parent metal, is now recommended by the supplier for service in the Thorex solutions.

Specimens of stabilized Ni-o-nel were heat treated at 1850 F for 0.5 hr and air quenched prior to welding. Weldments by inert-gas techniques using strips cut from the parent metal are being compared with similar weldments made with the No. 65 wire. After 288 hr of exposure to boiling Initial Thorex solution containing 0.20 M Al^{+3}, the rates range from 0.8 to 1.0 mil per month, having decreased steadily from about 1.8 mils per month. Selective attack can be seen on weldments and intergranular attack on a zone of the heat-affected areas adjacent to the weldments. The weldments made from strips of parent metal appear slightly superior to those made from the No. 65 wire. Exposure of these specimens is being continued. Present data indicate heat treatment at 1850 F to be far superior to that at 1650 F and heat treatment after welding to be preferable. Further evaluation of 1850 F treatments after welding will be conducted.

Exposure of Ni-o-nel specimens to the conditions present during the batch dissolution of Type 304 stainless steel in boiling 6 M H_2SO_4 was terminated following 192 hr. The corrosion rates for this type of exposure are compared with rates obtained in straight reflux studies in Table J-1. The four unconnected specimens from the batch-dissolution run were left with bright, etched surfaces which approach being chemically polished. The liquid-phase specimen which had been in contact with the stainless steel had a much lower corrosion rate, was dull in appearance, and showed no etching or selective attack. Dissolution conditions, therefore, can be concluded to give higher corrosion rates in the vapor phase than might be predicted from reflux-type studies, but do afford cathodic protection to submerged metal.

TABLE J-1. CORROSION RATES OF WELDED, HEAT-TREATED, STABILIZED NI-O-NEL[a] IN BOILING 6 M H_2SO_4

Type of Exposure	Specimen Position	Corrosion Rate, mils per month					
		A[b]	B[b]	A	B	A	B
		48 hr		122 hr		192 hr	
Reflux, no dissolution	Vapor	1.03	0.98	0.84	0.72	0.87	0.73
	Liquid	2.73	2.69	2.24	2.25	2.07	2.08
		50 hr		122 hr		192 hr	
Reflux plus batch dissolution[c]	Vapor	2.76	1.93	2.31	1.88	2.13	1.78
	Liquid	2.16	2.33	2.35	2.46	2.18	2.31
	Liquid-contact	0.57	--	0.60	--	0.59	--

(a) Specimens of stabilized Ni-o-nel (titanium-to-carbon ratio of 27 to 1) containing weldments were heat treated at 1850 F for 0.5 hr, air quenched, and machined to give clean, smooth surfaces.

(b) A and B represent duplicate specimens.

(c) Batch dissolution of Type 304 stainless steel rod was carried out during this exposure. Seventy-seven batches were used for an average time of 2.5 hr per batch.

J-4

Scouting Experiments With Other Metals

No other metals were studied in Sulfex-Thorex solutions this month.

The Zirflex Process

Chemical decladding of Zircaloy-2-clad uranium fuel elements by 6 M NH_4F, 1 M NH_4NO_3 solutions, followed by core dissolution in 10 M HNO_3 containing 0.05 M HF and small amounts of zirconium and aluminum as contaminants, is a procedure proposed for the Zirflex process.

Scouting experiments are under way in Teflon and Kel-F units. Various austenitic stainless steels and other candidate materials of construction are being exposed for 24-hr periods to boiling solutions representing expected beginning and final conditions of both the decladding and core-dissolution processes. The liquid phase of the 6 M NH_4F, 1 M NH_4NO_3 solution gave corrosion rates in the range of 10 mils per month for specimens of Type 347 stainless steel, Ni-o-nel, and Carpenter 20 Cb. From these preliminary studies, the decladding solution seems more corrosive than was originally anticipated.

The Fluoride-Volatility Process

Evaluation of various metals and alloys for construction of the hydrofluorinator in the Fluoride-Volatility process has continued. An HF sparge through a bath of molten fluoride salts would be used to hydrofluorinate fuel elements containing zirconium as a diluent or cladding.

A run at 700 C has been completed with the equimolar $NaF-ZrF_4$ salt. Corrosion rates were much higher for this 200-hr exposure being, in some instances, about double the rates for similar specimens exposed at 650 C. A silver specimen included at the interface position as a scouting experiment had a rate of 8.5 mils per month, which was as good as any other material exposed. Silver was not observed to plate out on other specimens.

A second run was completed in the salt representative of a possible final composition in the $NaF-ZrF_4$ system: 43.5 NaF-56.4 ZrF_4-0.050 UF_4 (mole per cent). An HF flow of 10 g per hr for 200 hr was used with no addition of hydrogen. The attack, in the nature of a uniform etch, was pronounced in this run, with several of the corrosion rates in the range of 5 mils per month, whereas with 2.4 w/o hydrogen present in a similar run, all rates were less than 1 mil per month. The interpretation of these results is somewhat clouded, since a silver scouting specimen was included and a silver plate was observed on most of the INOR specimens at the end of the run. Whether this inhibited or promoted attack is not known.

A series of runs is under way in one of the low-melting compositions of the sodium-lithium-zirconium fluoride system. This composition is 19.3 NaF-25.6 LiF-54.9 ZrF_4-0.21 UF_4 (mole per cent). Three 200-hr exposures with an HF flow of 10 g per hr are

planned at 600, 650, and 700 C in order that temperature effects on corrosion may be determined. The first of these at 600 C has been completed. No severe attack was observed. Highest corrosion rates occurred on coupon-type specimens exposed to the liquid. These were 1.7 mils per month for INOR-1 and 2.5 for INOR-8.

K-1

K. DEVELOPMENTS FOR SRE, OMRE, AND OMR

F. A. Rough and J. E. Gates

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

EVALUATION OF URANIUM MONOCARBIDE AS A REACTOR FUEL

F. A. Rough

The evaluation of uranium monocarbide as a fuel for the SRE is proceeding about on schedule. Two capsules of specimens have been irradiated and examined, two capsules are presently in the MTR, and three additional ones are to be irradiated. Routine progress is reported on these irradiations.

Laboratory heating of a specimen of uranium-5 w/o carbon irradiated to an effective exposure of 3.1×10^{13} nvt has been completed. The heating was carried out for extended periods at 1500, 1700, and 1800 F with only very minor release of the xenon-133 present. Future studies will involve heating to higher temperatures in the hot cell and measurement of density and gas release after heating.

Irradiation of Uranium Monocarbide

R. B. Price and W. H. Goldthwaite

The encapsulation and irradiation of uranium monocarbide is continuing. A second position has been obtained at the MTR, and two capsules, BMI-23-2 and BMI-23-4, each containing two specimens of cast uranium-5 w/o carbon, are now in the reactor. Capsule BMI-23-2 will be discharged from the MTR in January, while BMI-23-4 will continue irradiation to about 5000 MWD/T. Another capsule, containing similar specimens, is at the MTR awaiting loading in January.

Postirradiation Examination of Uranium Monocarbide

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

Prior work on this program has resulted in the compilation of data obtained from specimens irradiated at the BRR and also from specimens irradiated at the MTR. An evaluation of all the preirradiation and postirradiation data was reported previously, plus an analysis of dosimetry and fission-gas-release information.

K-2

Metallography and burnup analyses have not been completed on specimens from the BRR capsule and Capsule BMI-23-1. However, the work is continuing on these phases, and results should be forthcoming in the near future.

At this time, plans have been made to receive Capsule BMI-23-2 at the Battelle Hot-Cell Facility after discharge from the MTR in early January. Receipt of this capsule is expected late in the month and examination and testing of the specimens will be initiated as soon as possible.

Fission-Product Release From Irradiated Uranium Monocarbide

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

The objective of this work is to determine the amount of fission product, xenon-133, released from uranium carbide as a function of time and temperature of post-irradiation heating.

A uranium carbide specimen of near theoretical density was used for the gas-release study. The sample was sealed under vacuum in a quartz tube which contained activated charcoal at one end. This was protected from possible contamination from the sample by means of a quartz wool plug. The specimen was irradiated and received an effective exposure of 3.12×10^{13} nvt as determined by cobalt dosimetry, corrected for flux attenuation by the specimen.

After a decay period of 5 days, the portion of the quartz tube containing the charcoal was removed and measured for xenon-133 activity by means of gamma-ray spectrometry. The measured xenon-133 concentration is given in Table K-1 as Sample X-3.

TABLE K-1. FISSION-GAS RELEASE FROM URANIUM CARBIDE AS A FUNCTION OF TIME AND TEMPERATURE OF POSTIRRADIATION HEAT TREATMENT

Gas Sample	Temperature, F	Total Time at Temperature, hr	Fraction of Total Xenon-133 Present Found[a]
A-3	1500	24	3.57×10^{-4}
B-3	1500	95	8.35×10^{-4}
C-3	1700	24	1.40×10^{-4}
D-3	1700	48	3.40×10^{-4}
E-3	1800	24	2.97×10^{-4}
F-3	1800	48	4.93×10^{-4}
G-3	1800	120	9.48×10^{-4}
X-3	500[b]	15 sec	5.11×10^{-4}

(a) Cumulative values for time at each temperature.
(b) Estimated from fission rate.

The irradiated uranium carbide specimen was then placed in a closed, evacuated system and heated at several temperatures up to 1800 F. As was done in previous

experiments, samples of gas were collected by absorption in charcoal traps which had been evacuated and cooled with liquid nitrogen. The individual traps were isolated and xenon-133 concentrations were measured. Results of the gas analyses are shown in Table K-1.

POSTIRRADIATION STUDIES OF SRE, OMRE, AND OMR FUEL MATERIALS

J. E. Gates

The results of measurements of fission-gas release from thorium-11 w/o uranium specimens irradiated in Capsule Trains NAA-15-6 and NAA-15-7 are reported. Also included in this section are results of measurements of cesium-137 release to the NaK coolant in the capsule. Metallography and measurements of the coefficients of linear thermal expansion of the irradiated specimens are in progress.

The examinations of thermocouples from Capsule NAA-28-1 have been completed. The sheath of the thermocouple penetrating to the middle of the UO_2 specimen stack appeared to have melted in one area. This completes the examination of the stainless steel-clad UO_2 fuel pins irradiated in Capsules NAA-28-1 and NAA-29-1.

The results of the gamma scan and metallographic examination of fuel plates from the OMRE-3 subassembly are reported. This completes the examination of this subassembly.

The results of the examination and testing of tensile, impact, and corrosion specimens of structural materials of interest to the OMR program are reported. The specimens were irradiated in Dummy Element OMR-1 in the OMRE. This completes the examination of these specimens.

SRE Fuel Material

J. H. Saling, G. E. Lamale, and W. Chubb

Thorium-Uranium Specimens

The postirradiation evaluation of twelve samples of thorium-11 w/o uranium alloy irradiated in NaK to burnups ranging from 0.57 to 1.5 total a/o at temperatures near 1200 F is in progress. All specimens were initially 3/8 in. long, and were prepared from 93 per cent enriched uranium. Specimens were irradiated in the swaged condition in two capsule trains, NAA-15-6 and NAA-15-7, each containing six individually encapsulated specimens in the MTR.

K-4

Three of the inner capsules of the NAA-15-6 assembly, Capsules 2, 3, and 5, were sampled for fission gas. The gases were analyzed in a mass spectrometer. Tables K-2 and K-3 summarize the results.

TABLE K-2. MASS SPECTROMETRIC ANALYSIS OF GAS COLLECTED FROM NAA-15-6 CAPSULES

Capsule	Gas Sample Volume, cm^3	Volume Per Cent of Components					
		H_2	He	A_{39}	A_{40}	Kr (total)	Xe (total)
2	1.99	1.7	91	1.5	2.9	0.42	2.6
3	2.12	0.23	96	1.3	0.13	0.37	2.1
5	2.60	2.7	73	1.1	0.55	3.2	20

TABLE K-3. ESTIMATED FISSION-GAS RELEASE FROM THORIUM SPECIMENS IN NAA-15-6 CAPSULES

Capsule	*Estimated Gas Release, per cent*
2	0.24
3	0.18
5	1.9

Specimens from Capsules NAA-15-6-2 and NAA-15-6-3 were in relatively good condition while the specimen from Capsule NAA-15-6-5 was severely bulged and cracked near the top. This correlates with the high gas release associated with this specimen.

The amount of cesium-137 escaping from selected specimens during irradiation was measured. The NaK contained in the capsules in which the selected specimens were irradiated was reacted with butyl alcohol. The alcohol-NaK solution was analyzed for cesium-137 with a pulse-height analyzer after completing other routine procedures. The results are summarized in Table K-4.

TABLE K-4. RESULTS OF ANALYSES OF NaK FROM SELECTED CAPSULES FOR CESIUM-137

Capsule	Total Volume of Analyzed Solution, cm^3	Amount of Cesium-137 Activity, disintegrations/(min)(ml)	Estimated Amount of Cesium-137 Released, per cent
NAA-15-7-A	578	4.1×10^8	19
NAA-15-6-1	530	0.63×10^8	1.8

The Capsule NAA-15-7-A specimen was badly swollen and cracked while the specimen in Capsule NAA-15-6-1 was in relatively good condition. The high loss of cesium-137 from the one specimen is considered to be due to the tremendously increased surface area exposed to the NaK coolant. The high loss of cesium-137 is not considered to have an effect on the radiochemically determined burnup, since a section from the relatively unaffected center area of this specimen was analyzed. The relatively low loss of cesium-137 from the undamaged specimen in Capsule NAA-16-6-1 tends to verify this. The analysis of the NaK contained in an additional capsule is being rechecked.

The only experimental work remaining is the metallographic examinations and the coefficient of linear thermal-expansion measurements.

Uranium Dioxide Specimens

The examination of the tubes containing uranium dioxide pellets irradiated in Capsules NAA-28-1 and NAA-29-1 has been completed. The results of the examinations of these two pins have been reported previously. The hot junctions of the two thermocouples from Capsule NAA-28-1 were removed and examined. There was evidence of melting in the sheath of the thermocouple which extended to the center of the specimen stack. The thermocouple which extended 1 in. into the UO_2 did not show any visible evidence of melting of the sheath. This completes the examination of the fuel pins in Capsules NAA-28-1 and NAA-29-1.

OMRE Fuel Elements

R. J. Burian

All phases of the examination and evaluation of the OMRE-3 subassembly have been completed, including a metallographic examination of sections from two stainless-clad stainless-UO_2-fueled plates.

The results of the burnup analyses have been correlated with the gamma-scan data. Burnup profiles of the three scanned plates have been prepared. These data indicate that the burnup of the fuel plates ranged up to about 4.0 a/o of the uranium in the outer plates. The burnup of the center plate ranged up to about 2.0 a/o of the uranium.

A measurement of the organic residue deposit on one fuel plate was made metallographically. The residue thickness was found to vary between 0.00012 and 0.00037 in., with an estimated average thickness of 0.00025 in.

The metallographic examination of the fuel plates indicated nothing unusual. There were no visible cracks or other defects in the matrix or UO_2 particles, and the cladding-core bond appeared to be in good condition.

K-6

This completes the examination of the OMRE-3 subassembly.

OMR Structural Materials

R. J. Burian and E. G. Bodine

The effects of radiation and corrosion by the organic moderator on certain structural materials planned for use in the Organic Moderated Reactor (OMR) are being studied. A total of 54 irradiated specimens of structural materials were irradiated in a dummy fuel element in the OMRE. Included in the group of specimens were 12 tensile, 12 V-notch charpy impact, 18 U-bend corrosion, six flat-welded corrosion, and six flat unwelded corrosion specimens. The specimens were grouped in bundles of six around a holder rod along the axis of the dummy element, except for the flat welded and unwelded corrosion specimens. The 12 flat corrosion specimens formed one bundle.

The total of eight bundles were distributed along the center 28 in. of the element. Beginning at the top of the element, the bundles were arranged in the following order: 12 flat corrosion specimens; six U-bend corrosion specimens; six impact specimens; six tensile specimens; six impact specimens; six tensile specimens; two bundles of six U-bend corrosion specimens. Two dosimeter wires made of aluminum-0.5 w/o cobalt alloy and nickel were strung along the length of the center rod.

The specimens in the dummy fuel element were irradiated in the OMRE in a position on the perimeter of the core. Upon completion of the irradiation, the specimens were recovered and examined.

The end pieces were remotely cut from the dummy fuel element to permit removal of the center rod holding the specimens. The specimens were removed from the center rod and identified. The two dosimeter wires were removed and prepared for scanning for gamma-ray activity.

The relative intensity of the gamma radiation emitted from the aluminum-0.5 w/o cobalt dosimeter wire was measured by scanning with a gamma-ray spectrometer. Three sections were then cut from the wire at equal intervals along its length. Each section was analyzed radiochemically to determine the thermal neutron flux. The results are included in Table K-5. Then, by relating the results of the analyses to the relative gamma intensity data, the thermal neutron flux could be estimated at any point along the length of the dummy element. An attempt to scan the nickel dosimeter wire was not made because of its brittleness.

The nickel dosimeter wire was cut into 12 sections, six of which were radiochemically analyzed to determine the fast neutron flux. The fast neutron flux derived from this analysis includes all neutrons of ferrous spectrum energies as calculated from the activation of the nickel-58 by n-p reaction to form cobalt-58. The cross section and threshold energy values were taken as 91 millibarns and 32 Mev, respectively. The results are included in Table K-5. These data were then used to estimate the fast

TABLE K-5. RESULTS OF ANALYSES OF DOSIMETER WIRES IRRADIATED IN DUMMY FUEL ELEMENT OMR-1

Dosimeter Wire Composition	Section Number[a]	Distance From Top of Uppermost Bundle, in.	Average Integrated Neutron Flux[b], x 10^{19} nvt
Al-0.5 w/o Co	1	3	6.99
	2	11	7.99
	3	19	11.2
Nickel	1	3	20.5
	2	7	25.2
	3	11	29.6
	4	15	32.8
	5	19	33.1
	6	23	30.0

(a) Each section analyzed was approximately 2 inches long.

(b) The values given for the aluminum-0.5 w/o cobalt wire are thermal neutron fluxes and those for the nickel wire are fast neutron fluxes as defined in the text of the report.

neutron flux at the location of the specimen bundles. The integrated fast neutron flux estimated for each bundle position is given in Table K-6.

The data obtained from the examination of the test specimens are discussed separately below.

Flat Welded Corrosion Specimens

The six specimens of this series were made by butt-welding two dissimilar steels together to form one specimen. The combination included 4130 steel and Type 304 sensitized stainless steel, 4130 steel and Type 410 stainless steel, carbon steel and Type 410 stainless steel, carbon steel and Type 304 sensitized stainless steel, and two specimens of carbon steel and Type 304 anodized stainless steel. Visual examination of the welded areas and the virgin metal adjacent to the welds at magnifications to 32X revealed no evidence of corrosive attack by the organic moderator. Several large isolated pits were noted in two or three welds but the size, rarity, and surface condition of the pits indicated that they were probably the result of the welding operation.

Flat Unwelded Corrosion Specimens

The six flat unwelded corrosion specimens were made from aluminum, magnesium, Type 304 anodized stainless steel, Type 304 sensitized stainless steel, Type 410 stainless steel, and 4130 alloy steel. The specimens were cleaned, dry weight measurements were taken, and photographs were made of all specimens. The four steel specimens indicated weight changes of less than 0.2 per cent. The aluminum and

TABLE K-6. APPROXIMATE FAST NEUTRON EXPOSURE OF EACH SPECIMEN BUNDLE IN DUMMY FUEL ELEMENT OMR-1

Specimen Bundle, Number	Distance From Top of Bundle Groups, inches	Type of Specimens	Specimen Numbers	Average Integrated Fast Neutron Flux, x 10^{19} nvt
1	0.8	Flat corrosion	B7K, C7K, D7K, E7K, J7K, H H7K, A7K-C8K, A7K-B8K, A8K-B8K, C8K-J8K, H8K-J8K, A8K-H8K	18.3
2	3.5	U-bend corrosion	J30K, J31K, J32K, B23K, C24K, C25K	21.1
3	6.2	Impact	B20K, B21K, B22K, B23K, C24K, C25K	24.2
4	10.9	Tensile	B10K, B11K, B12K, C10K, C11K, C12K	29.4
5	15.5	Impact	H20K, H21K, H22K, H23K, C22K, C23K	33.0
6	20.1	Tensile	H10K, H11K, H12K, A13K, B13K, J13K	32.4
7	24.7	U-bend corrosion	B30K, B31K, B32K, C32K, C33K, C34K	27.8
8	27.0	U-bend corrosion	A30K, A31K, A32K, H32K, H33K, H34K	24.2

magnesium specimens experience much larger weight changes. However, a control test to determine the effect of the cleaning solution indicated that the majority of the weight lost by the aluminum specimen was probably the result of postirradiation cleaning of the specimen. The magnesium specimen showed a lesser amount of attack by the cleaning solution. The results of the examination of the aluminum and magnesium specimens are therefore considered to be inconclusive.

U-Bend Corrosion Specimens

The 18 specimens of this group were bent and maintained in a U-shape during irradiation to study the effects of stresses on the corrosion of various materials. The materials used in this phase of the program included Type 304 anodized and Type 304 sensitized stainless steel, Type 410 stainless steel, 4130 alloy steel, carbon steel, and aluminum. Visual examination of the bent regions revealed fine shallow pits in the aluminum specimen. However, data describing the preirradiation appearance of these specimens are not available. It was, therefore, not possible to determine if these fine pits were formed during irradiation or by the stretching of the metal when the specimen was formed. The other specimens appeared unaffected. Although the integrated neutron flux varied by as much as 31 per cent between the U-bend specimens, there were no observable differences.

Impact Specimens

Four specimens of each of three different steels were irradiated and impact tested. The three materials included Type 304 sensitized and Type 304 anodized stainless steel, and Type 410 stainless steel. Fourteen unirradiated control specimens were also tested at the same time. The controls were made of 4130 alloy steel and carbon steel, in addition to the three types of stainless steels used for the irradiated specimens. All tests were conducted on a Riehle Model PI-2 combination impact machine. By interchanging hammers, three energy ranges were possible; zero to 60, zero to 120, and zero to 240 ft-lb. Tables K-7 and K-8 show the results of the tests on the unirradiated and irradiated specimens. It is not considered good practice to compare energy values above 80 ft-lb when testing V-notch Charpy specimens, since for fracture energies above this value the specimen does not break cleanly and tends to drag as it passes through the split anvil.(2) Therefore, it is believed that conclusions cannot be drawn from these tests. The unirradiated 4130 alloy-steel and the carbon-steel specimens which broke at acceptable energies had no irradiated counterparts.

Tensile Specimens

Fifteen unirradiated control and 12 irradiated tensile specimens were tested. These specimens were made of five types of steel including Type 304 sensitized and Type 304 anodized stainless steel, Type 410 stainless steel, 4130 alloy steel, and carbon steel. The specimens had a nominal 0.250-in.-diameter by 1.0-in.-long gage section. The irradiated specimens were tested with a remotely operated Riehle Model PH-30 universal testing machine. The unirradiated control specimens were

(2) Metals Handbook, 1948 Edition, American Society for Metals, Cleveland, pp 112-115.

K-10

TABLE K-7. RESULTS OF IMPACT TESTS ON UNIRRADIATED SPECIMENS[a]

Material	Specimen	Energy Absorbed at Impact[b], ft-lb
Carbon steel	A-20-N	67.0
	A-21-N	52.0
	A-22-N	55.5
Type 304 stainless, anodized	B-20-N	Absorbed 120 ft-lb without breaking[c]
	B-21-N	Absorbed 240 ft-lb without breaking
	B-22-N	Absorbed 240 ft-lb without breaking
Type 304 stainless, sensitized	C-20-N	Absorbed 120 ft-lb without breaking[c]
	C-21-N	Absorbed 240 ft-lb without breaking
Type 410 stainless	H-21-N	Absorbed 120 ft-lb without breaking[c]
	H-22-N	145
	H-23-N	161
4130 alloy steel	J-20-N	40.5
	J-21-N	42.5
	J-22-N	47.0

(a) Type of test: Simple beam V-notch Charpy
Linear velocity of hammer at impact: 16 fps
Maximum energy available at impact: 240 ft-lb except as noted
Temperature: Ambient (approximately 78 F)

(b) Data in excess of 80 ft-lb are not considered reliable.

(c) Two specimens were tested using an impact hammer capable of delivering a maximum of 120 ft-lb of energy.

TABLE K-8. RESULTS OF IMPACT TESTS ON SPECIMENS IRRADIATED IN DUMMY ELEMENT OMR-1[a]

Material	Specimen	Integrated Fast Neutron Flux, x 10^{19} nvt	Energy Absorbed at Impact[b], ft-lb
Type 304 stainless, anodized	B-20-K	24.2	Absorbed 240 ft-lb without breaking
	B-21-K	24.2	Absorbed 240 ft-lb without breaking
	B-22-K	24.2	Absorbed 240 ft-lb without breaking
	B-23-K	24.2	Absorbed 240 ft-lb without breaking
Type 304 stainless, sensitized	C-22-K	33.0	227.7
	C-23-K	33.0	Absorbed 240 ft-lb without breaking
	C-24-K	24.2	236.9
	C-25-K	24.2	Absorbed 240 ft-lb without breaking
Type 410 stainless	H-20-N	33.0	77.0
	H-21-K	33.0	93.4
	H-22-K	33.0	95.2
	H-23-K	33.0	89.1

(a) Type of test: Simple beam V-notch Charpy
Linear velocity of hammer at impact: 16 fps
Maximum energy available at impact: 240 ft-lb except as noted
Temperature: Ambient (approximately 78 F)

(b) Data in excess of 80 ft-lb are not considered reliable.

TABLE K-9. TENSILE-TEST RESULTS OF IRRADIATED AND CONTROL SPECIMENS FROM OMR-1

(Strain Rate 0.001 in./in./min)

Material	Specimen[a]	Integrated Fast Neutron Flux, x 10^{19} nvt	Tensile Strength, 10^3 psi	Breaking Strength, 10^3 psi	2 Per Cent Offset Yield Stress, 10^3 psi	Elongation in 1-in. Gage Length, per cent	Reduction of Area, per cent	Modulus of Elasticity, 10^6 psi
Carbon steel	A-13-K	32.4	78.4	67.2	58.5	23.4	36.5	28.4
	A-14-N	Control	65.3	48.9	43.3	--	65.3	29.8
	A-15-N	Control	66.2	50.6	42.1	31	61.3	28.4
	A-16-N	Control	65.2	50.8	40.5	25	59.5	27.5[b]
Type 304 stainless, anodized	B-10-K	29.4	83.7	59.0	43.0	61.0	56.5	31.8
	B-11-K	29.4	81.0	59.4	49.5	--	52.0	31.2
	B-12-K	29.4	84.8	60.8	49.0	62.5	52.2	29.9
	B-13-K	32.4	86.0	59.2	45.0	66.0	63.0	31.5
	B-14-N	Control	85.1	54.9	40.4	72	78.9	30.7
	B-15-N	Control	87.0	55.8	40.8	69	79.5	28.8
	B-16-N	Control	85.0	53.2	41.8	78	84.7	33.1
Type 304 stainless, sensitized	C-10-K	29.4	84.4	58.3	49.2	--	47.2	29.4
	C-11-K	29.4	87.0	59.2	46.0	64.0	53.3	29.9
	C-12-K	29.4	87.7	60.7	54.5	55.0	60.0	29.4
	C-14-N	Control	85.3	61.1	40.2	70	75.4	27.3
	C-15-N	Control	85.3	56.2	39.1	69	75.4	26.7
	C-16-N	Control	86.3	59.1	39.8	70	78.5	28.7
Type 410 stainless	H-10-K	32.4	103.2	62.3	87.0	12.3	43.0	30.5
	H-11-K	32.4	102.5	61.2	85.9	26.3	34.0	31.0
	H-12-K	32.4	102.8	62.2	86.0	25.0	59.0	30.3
	H-14-N	Control	89.4	53.8	73.4	28	73.8	29.8
	H-15-N	Control	89.1	52.5	75.3	26	73.3	28.1[b]
	H-16-N	Control	88.2	52.9	80.0	26	71.4	27.2[b]
4130 steel	J-13-K	32.4	109.0	82.3	80.0	31.0	48.4	29.1
	J-14-N	Control	96.4	76.6	70.0	21	57.0	30.5[b]
	J-15-N	Control	95.5	72.7	66.7	25	61.0	26.1
	J-16-N	Control	98.4	80.2	76.0	--	57.7	30.0[b]

(a) Explanation of specimen numbering system: K = Irradiated specimens; N = Unirradiated specimens; J = 4130 steel.
A = Carbon steel; B = Type 304 anodized stainless steel
C = Type 304 sensitized stainless steel; H = Type 410 stainless steel

(b) Modulus adjusted for fracture occurring at minimum diameter on specimen.

tested with a Baldwin T-E universal testing machine. All tests were performed at a strain rate of approximately 0.001 in. per in. per minute. The strain rate was recorded as a function of the load using a conventional extensometer and a high-speed autographic recorder.

To provide an accurate basis for computation of the modulus of elasticity, loads within the elastic range were applied and released several times before the specimen was loaded to failure. The data obtained before and after irradiation are compared in Table K-9. Values of average mechanical properties as given in the literature for the materials tested are presented in Table K-10. The data presented in Table K-9 indicate a tendency in the material toward embrittlement due to irradiation. This is indicated by the decrease in reduction of area and elongation. Increases in the fracture and maximum tensile strengths and the 2 per cent offset yield stress are in the order of the effects caused by cold working. This tendency is most pronounced for the carbon steel, the 4130 alloy steel, and the Type 410 stainless steel. The Type 304 stainless steel is less affected. The variation of 10 per cent in the integrated fast neutron flux experienced by the tensile specimens apparently did not produce observable effects.

A metallographic examination was performed on irradiated samples of a carbon steel and a Type 410 stainless steel. The sections examined were in the unstressed region of the grip ends of tensile specimens. The areas of both specimens examined had an appearance not unlike that of similar unirradiated specimens. The pearlite structure was very evident in the carbon-steel specimen. Knoop microhardness measurements made on each specimen gave average values of 179 and 228 Knoop for the carbon steel and the Type 410 stainless steel, respectively. The literature[3] gives a hardness of 160 to 190 on the Brinell hardness scale for Type 410 stainless steel in the annealed condition. This converts to approximately 160 to 190 on the Knoop hardness scale. No comparable value can be obtained for the carbon steel because information is not available on the exact carbon content.

TABLE K-10. AVERAGE MECHANICAL PROPERTIES OF UNIRRADIATED STEELS OBTAINED FROM THE LITERATURE

	Type 410 Stainless[a]	Type 304 Stainless[b]	4130[c] Alloy
Brinell Hardness	160-190	150	180-220
Rockwell Hardness	78-88 B Scale	80 C Scale	--
Tensile Strength, psi	65-85,000	85,000	90-118,000
Yield Point, psi	40-60,000	40,000	60-70,000
Elongation, per cent in 2 in.	35-25	60	25-30
Reduction of Area, per cent	75-60	70	45-60

(a) Metals Handbook, 1948 Edition, pp 555.
(b) "Nickel Alloy Steels", 2nd Edition (1947), International Nickel Company, Section 7, p 25.
(c) "Molybdenum in Steel", Climax Molybdenum Company, Section 2, p 10.

(3) Metals Handbook, 1948 Edition, American Society for Metals, Cleveland, p 555.

L-1

L. TANTALUM AND TANTALUM-ALLOY STUDIES

J. H. Stang

Current research for Los Alamos Scientific Laboratory (LAMPRE program) includes a study of tantalum-tungsten and other tantalum-bearing alloys and an investigation of irradiation damage of tantalum. During December, high-purity tantalum and tantalum-tungsten specimens fabricated from arc-welded cold-rolled sheet were forwarded to LASL for corrosion studies in plutonium-alloy fuel mixtures. Annealing studies were also conducted prior to preparation of annealed specimens for LASL.

Development of Container Materials for LAMPRE Applications

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

Nine specimens of tantalum and tantalum-tungsten alloys have been sent to LASL for plutonium-alloy compatibility testing. The specimens, measuring approximately 1 by 3 by 0.030 in., were cold rolled to a total reduction in thickness of about 90 per cent and were etched for the removal of surface contaminants. X-ray fluorescence tests on the rolled surfaces after etching indicated that the specimens were relatively free from surface iron contamination. Tentative data on chemical analysis, hardness, and grain size of the selected materials in the as-cast and as-rolled conditions are given in Table L-1.

Annealing studies were conducted on cold-rolled (90 per cent reduction) strip specimens to determine the temperature at which the tantalum and tantalum-1.5 to 6 w/o tungsten alloys should be heat treated to provide minimum hardness and relatively fine grain size. Specimens were heated for 30 min in vacuo at temperatures from 2000 to 3270 F. Based on the Vickers hardness data given in Table L-2 and on tentative grain-size measurements, a temperature of 2600 F has been selected for annealing of the tantalum metal and the tungsten-containing alloys.

Annealed specimens identical to those listed in Table L-1 are being prepared for plutonium-corrosion tests at LASL. Also, room-temperature tensile properties are to be obtained for the nine selected compositions in both the as-rolled and as-annealed conditions.

The next series of alloys will be composed of tantalum plus rhenium. Following this, attention will be turned to alloys in which carbon and yttrium are added. One reason for interest in these two elements is their thermodynamic potential to deoxidize tantalum.

L-2

TABLE L-1. PROPERTIES OF ARC-MELTED TANTALUM AND TANTALUM-TUNGSTEN ALLOYS

	Melting Stock		Unalloyed Tantalum Specimens			Tantalum-Tungsten-Alloy Specimens					
						Ta-1.5 w/o W		Ta-3 w/o W		Ta-6 w/o W	
	Ta	W	13	11	10	19	3	6	4	9	8
Chemical Analysis on Cast Buttons, ppm											
Tungsten	50	(a)	50	50	(b)	1.45(c)	1.71(c)	2.94(c)	2.92(c)	6.06(c)	5.80(c)
Oxygen	11	8	18	23	21	11	20	17	12	13	33
Hydrogen	2	1	3	5	6	4	6	3	5	4	3
Carbon	10	10	20	30	80	40	80	50	80	60	100
Nitrogen	10-20	10	20	30	60	10	40	30	20	40	110
Iron	7-30	5	30	10	(b)	3	8	3	30	3	15
Silicon	30-100	100	100	50	(b)	30	30	20	100	200	30
Copper	15-50	10	25	50	(b)	30	10	10	15	5	200
Niobium	100-300	(a)	150	100	(b)	50	300	50	100	50	150
Zirconium	10	10	20	10	(b)	10	20	10	10	10	20
Aluminum	10-30	20	30	20	(b)	10	20	10	30	10	20
Molybdenum	10	10	10	10	(b)	20	20	20	10	20	50
Nickel	3-30	10	5	20	(b)	3	8	3	3	3	25
Chromium	2	50	7	2	(b)	3	5	5	5	2	60
Calcium	(a)	(a)	(a)	(a)	(b)	(a)	4	(a)	(a)	(a)	6
Vickers Hardness (10-Kg Load)											
As Cast			92.5	106	123	108	126	134	157	186	214
As Cold Rolled			166	171	174	203	233	266	254	297	306
Average Grain Diameter (As Cast), mm											
			2.9	2.4	2.6	2.1	2.0	1.9	2.0	1.6	1.2

(a) Not analyzed.
(b) Analysis in progress.
(c) This analysis in weight per cent.

TABLE L-2. EFFECT OF ANNEALING TEMPERATURE ON HARDNESS OF COLD-ROLLED TANTALUM AND TANTALUM-TUNGSTEN ALLOYS

Specimen	Tungsten Content (Balance Tantalum), w/o	Hardness, VHN, After Annealing 30 Min in Vacuum at Indicated Temperature						
		As Rolled	2010 F	2205 F	2415 F	2610 F	2815 F	3270 F
16	--	171	121	86.5	75	74	75	71
18	1.5	227	169	114	92	93.5	93.5	85
5	3	243	220	175.5	119	116	114	109
7	6	299	276	268	180	176.5	176.5	164

L-3 and L-4

Irradiation Damage of Tantalum

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

A study of the effect of neutron irradiation on the mechanical properties of tantalum is in process. The basic objective is to obtain comparative data about the strengthening effects of tungsten alloying additions made by (1) conventional arc-melting techniques, and (2) by irradiation-induced transmutation of tantalum to tungsten.

This study has been delayed pending the arrival of tantalum stock which will be used in the experiments.

M-1

M. DEVELOPMENTAL STUDIES FOR THE PWR

R. W. Dayton

A quarter-scale model of the prototype baffling arrangement was constructed for study in the PWR flow model.

Difficulties with brittleness of pressure-bonded PWR oxide-containing fuel plates have been traced to hydrogen contamination of the helium. Samples of the helium cylinder gas are being analyzed for hydrogen. Putting the samples in a close-fitting container has eliminated significant contamination, and ductile plates are now being prepared. Bond-line voids seem to be eliminated by evacuating packs before pressure bonding. Reaction between UO_2 and Zircaloy-2 can be prevented, except for small amounts at corners, by heavier graphite coatings on the UO_2.

Reactor Flow Studies

L. J. Flanigan and H. R. Hazard

The effects of lower-plenum geometry on mixing and flow distribution in Core 2 of the PWR are being studied in a quarter-scale air-flow model. Past work included development of an optimum baffling arrangement for the lower plenum with the 7.5-ft core design and design of a similar baffling arrangement by Bettis suitable for incorporation into the prototype.

In December, a quarter-scale model of the prototype baffling arrangement was constructed.

In January, the baffle arrangement will be installed in the model, flow studies will be run to verify its performance, and a report covering the flow studies will be prepared.

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

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

A flat-plate Zircaloy-2-clad fuel element containing compartmented uranium dioxide fuel is being considered for Core 2 of the PWR. An investigation is being conducted to study the preparation of these elements by a gas-pressure-bonding technique. In the technique presently being developed, the Zircaloy-2 cladding components are edge welded, evacuated, and sealed to form a gastight assembly which is then pressure bonded at 1550 F and 10,000 psi for 4 hr using helium gas. Portions of elements are being given a heat treatment at 1850 F for 5 min subsequent to pressure bonding. The

receptacle plates to receive the cores are assembled from strip components; the bonding surfaces of all components are belt abraded. A graphite coating on the UO_2 cores is being used to prevent fuel-cladding reaction.

Defected-compartment corrosion tests in 680 F water have been continued on eight small specimens prepared by this technique. For exposure times of up to 48 days, no significant growth has been observed in specimens bonded at 1550 or 1750 F with or without an additional 1850 F heat treatment for 5 min. Since zones of oxygen-rich Zircaloy-2 surrounding the cores were observed metallographically in these specimens, corrosion tests are being continued to determine if this small amount of apparent core-cladding reaction has a deleterious effect on long-time corrosion behavior.

Additional large-scale specimens have been prepared incorporating various modifications in an attempt to eliminate problems encountered in earlier elements with cladding embrittlement, core-cladding reaction, and dimensional control of the cladding over the ribs. The most severe problem has been that of brittle behavior of the Zircaloy-2 cladding after bonding. Extensive tests with cladding plates have demonstrated that the embrittlement was caused by hydrogen absorption. Possible sources of contamination were moisture in the autoclave insulating material, oil vapor from the compressor, and high hydrogen contamination present in helium used to conduct the tests. Specimens prepared under these adverse conditions contained 400 to 775 ppm of hydrogen and behaved in a very brittle fashion during compartment burst tests and bend tests. The autoclave insulating material was heated to dry it, and an additional oil filter was inserted in the exit line from the compressor.

The analysis of the helium gas in one cylinder revealed a hydrogen level of 1 mole per cent. Additional analyses of other cylinders are being obtained to determine the source of this contamination as the average content of hydrogen in helium is approximately 10 ppm. To minimize contamination from the gas all recent specimens have been bonded in a copper container designed to contain a minimum amount of gas and restrict gas circulation.

Elements with very ductile cladding were prepared after these precautionary measures were exercised. The Zircaloy-2 possessed a hydrogen content in the range of 15 to 35 ppm. The modifications incorporated have also greatly reduced contamination of the Zircaloy-2 by oxygen, which was an additional contributing factor to embrittlement.

Previously reported results of metallographic examination of large-scale elements indicated that the Zircaloy-to-Zircaloy bonds were generally good, showing a large percentage of grain growth across the original bond interface, especially in specimens which had received the 1850 F heat treatment. It was noted, however, that there were small voids and narrow oxygen-rich zones along the interface which were believed due to entrapped gas, since these elements were not evacuated before final sealing in air. To eliminate this defect, two alternate techniques for evacuating and sealing have been developed and used successfully. Both sealing techniques make use of a narrow projection machined on one end of the element which contains a slot to permit evacuation of the element after it has been sealed around the edges by welding in a helium tank. In one method, the element is placed inside a chamber which is

evacuated and the final hole is sealed by melting the end of the projection with an induction coil contained in the chamber. The other technique consists of resistance-upset welding the projection in air while the inside of the specimen is being evacuated by means of a vacuum connection to the end of the projection. Both of these methods are presently being used to attempt to determine the most reliable one. Preliminary examination of specimens which were evacuated has revealed no voids at the bond interface and grain growth was observed across 95 per cent of the original interface. The bonding surfaces of these elements were prepared for bonding by belt abrading.

Areas of core-cladding reaction have also been observed in the large-scale specimens that contained cores coated with 1.5 to 2 mg per in.^{2} of graphite as evidenced by zones of oxygen-rich Zircaloy-2 adjacent to the cores. Corrosion tests on the large-scale specimens have revealed no significant growth during 21 days of exposure. However, it is desirable to eliminate or minimize the possible source of contamination as well as to determine its effects on long-time corrosion. Previous cores were given a graphite coating of a density of about 1 to 3 mg per in.^{2} in attempts to prevent reaction. Specimens have now been prepared containing cores with controlled densities of graphite coatings ranging from 1 to 9 mg per in.^{2} to determine the effect of the thickness of the graphite barrier layer in preventing core-cladding reaction. Specimens recently examined that contained cores coated with 5 to 6 mg per in.^{2} evidenced no appreciable amounts of core-to-cladding reaction on surfaces, but small amounts of reaction on corners.

Areas in which the Zircaloy-2 cladding protruded above the surface of the cores had been observed in the large-scale elements initially bonded. This was apparently caused by excessive flow of cladding material to fill void space in the assembly. The effect has been minimized by reducing slightly the original thickness of the ribs and permitting the longitudinal ribs freedom of movement to fill any excessive void space present prior to final edge welding. The resultant assembly bonds with a minimum amount of cladding distortion over the rib and core areas.

About 20 additional elements measuring 0.100 by 4.6 by 15.0 in. are being prepared using the modifications developed by the studies thus far. These specimens have belt-abraded bonding surfaces and graphite-core-coating densities of about 6 mg per in.^{2}, and will be evacuated prior to bonding. Twelve irradiation specimens are also being prepared by this technique. A full evaluation will be made of all of these specimens to determine process feasibility as a method of preparing PWR-type fuel elements on a production basis.

N-1

N. DEVELOPMENTS FOR THE MGCR

W. H. Goldthwaite

Research in several areas of assistance to the Maritime Gas-Cooled Reactor Program is reported in this section. The postirradiation examination of two stainless steel-clad UO_2 specimens is continuing. A program which includes the development of fabrication techniques and the irradiation of three potential fuel materials has just been initiated. An investigation of the effects of radiation on the transport of carbon by impurities in helium coolant gas has just started.

THE POSTIRRADIATION EXAMINATION OF GA-BNL SINTERED UO_2 FUEL SPECIMENS CLAD WITH STAINLESS STEEL

G. E. Lamale and J. E. Gates

The postirradiation examination of the UO_2 samples obtained from Brookhaven National Laboratory continues. One of the samples contained two pellets of UO_2 sintered to a density of 93 per cent of theoretical and was clad with 10 mils of Type 316 stainless steel. This sample was irradiated in a CO_2 atmosphere to an integrated exposure of 5.2×10^{18} nvt at a temperature of 1600 F. The cladding on this sample was swollen and split. The second sample contained two pellets of UO_2 sintered to a density of 93 per cent of theoretical and was clad with 5 mils of Type 316 stainless steel. This sample was irradiated in a helium atmosphere and to an exposure of 4.7×10^{19} nvt at a temperature of 1200 F. This sample did not fail.

Measurements of the dimensions of the UO_2 pellets in the sample with the 5-mil cladding have been completed. Both pellets were cracked and no attempt was made to remove the cladding from the pellet. The results of the measurements are given in Table N-1.

TABLE N-1. PHYSICAL DIMENSIONS OF IRRADIATED BROOKHAVEN SAMPLES 2 AND 8

Sample	Thickness of Type 316 Stainless Cladding, mils	Sample Condition	Dimensions, in. Diameter Sample	Diameter UO_2 Pellets[a]	Length Sample	Length UO_2 Pellets	Remarks
2	5	As received	0.4520	--	1.9732	--	Cladding not removable
8	10	As received	0.4975	--	--	--	--
		Cladding removed	--	0.4254	--	0.5300	Opposite clamped end
				0.4250	--	0.5294	At clamped end

(a) Average preirradiation pellet diameter was 0.422 in.

N-2

Both of the pellets from the sample with the 10-mil cladding were intact although the cladding ruptured. These pellets were removed from the cladding and their dimensions measured. The results of these measurements are shown in Table N-1. A burnup wafer was cut from one pellet and the remainder of the pellet was sectioned longitudinally for metallographic examination. The cladding of this specimen was sectioned transversely at the rupture for metallographic examination.

The postirradiation measurements indicate that the pellets in the sample with the 5-mil cladding cracked and increased in diameter by about 4 per cent, using the average reported preirradiation value of 0.422 in. for the diameter of the pellets. In the case of the sample with the 10-mil cladding, the pellets show an increase of approximately 0.7 per cent in diameter. Sufficient data are not available to permit evaluation of length changes.

The analysis of the gas obtained by puncturing the sample with the 5-mil cladding indicated less than detectable amounts of fission gas present. The lack of detectable amounts of fission gas is considered to be unusual, considering the type of fuel material, the apparent swelling, and the irradiation conditions. Definite conclusions regarding the reliability of the gas measurements can probably be made after the specimen is examined metallographically.

Future work will include the dissolution of the two burnup wafers and their analysis for cesium-137 and total uranium content. The metallographic examination of both samples will also be continued.

FABRICATION AND IRRADIATION OF FUEL MATERIALS

An investigation of potential fuel materials for the MGCR has just been initiated. Procedures for the fabrication of BeO-UO_2, graphite-UC, and graphite-UC_2 fuel specimens clad with Type 316 stainless are being developed. The elements will contain approximately 20 volume per cent of 30 per cent enriched fuel. Specimens 1/4 in. in diameter are to be irradiated to burnups of 6, 12, and 20 per cent of the contained uranium-235 at a heat flux of about 200,000 Btu/(hr)(ft^2) and a surface temperature of 1500 F.

Fabrication of BeO-UO_2 Fuel Elements

A. K. Smalley, W. C. Riley, and W. H. Duckworth

Experiments leading to the development of a BeO-UO_2 fuel element were begun. The fuel element will consist of 20 volume per cent of UO_2 dispersed uniformly in a matrix of fine-grained densely sintered BeO. The UO_2 grains will be in the range of 150 μ in diameter.

For the initial experiments, specimens were prepared in the following manner:

(1) Unsintered UO_2 grains, measuring from about 175 to 200 μ, were prepared from MCW ceramic-grade natural UO_2 by treating the powder with 3 w/o of beeswax, compacting the powder, granulating the compact, and recovering grains in the desired size range by screening.

(2) UO_2 grains were dry mixed with Brush Beryllium Company LOH-grade BeO by tumbling the mixture end-over-end in a jar for 10 min.

(3) The BeO-20 volume per cent UO_2 mixture was compacted isostatically in rubber molds at 50,000 psi.

(4) The compacts were sintered for 1 hr at 2800 F in flowing hydrogen.

The sintered specimens were evaluated by measuring their bulk densities and by examining microscopically a mounted and polished section.

The bulk densities of 18 BeO-UO_2 specimens varied from about 91 to 103 per cent of the calculated theoretical density. This indicated that the UO_2 content of the pellets was widely variable, as a result either of inadequate mixing or of segregation of the UO_2 grains during handling. Examination of the polished section indicated that the UO_2 grains were somewhat larger than the desired 150 μ, and that considerable dusting of the UO_2 during mixing and handling had occurred.

In future work, efficient techniques for mixing fine-grained BeO with large, dense UO_2 grains will be sought. Either presintered or fully sintered UO_2 grain will be used, in an attempt to minimize dusting of the UO_2. When suitable techniques for preparing BeO-UO_2 specimens are developed, specimens containing enriched UO_2 will be prepared for irradiation.

Fabrication of Graphite Fuel Elements

W. A. Hedden, A. B. Tripler, A. J. Roese, W. C. Riley,
and W. H. Duckworth

Work was initiated during December to produce experimental fuel-element bodies consisting of 80 volume per cent graphite and 20 volume per cent fuel compound. The fuel compounds to be used are UC and UC_2.

In preliminary work, bodies were prepared from mixtures of (1) pulverized petroleum coke and UO_2, bonded with coal-tar pitch, and (2) pulverized AGOT-grade graphite and UC or UC_2, bonded with either phenolformaldehyde resin or coal-tar pitch.

N-4

The bodies were formed into compacts about 1/2 in. in diameter and 1/2 in. long and baked at 2000 F. The specimens containing UO_2 will be heated to a temperature of about 4600 F to convert the UO_2 to UC_2.

Results obtained from the preparation of bodies containing natural uranium fuel will be used to select compositions and processing conditions for fabrication of test specimens 1/4 in. in diameter and 1/4 in. long containing enriched fuel.

O-1 and O-2

O. ENGINEERING ASSISTANCE TO KAISER ENGINEERS

Reactor Flow Studies

L. J. Flanigan and H. R. Hazard

Studies of flow in a quarter-scale air-flow model of the PEGCPR are to provide design information for the prototype. Work to date includes establishment of the general model design, and completion of a major portion of the detailed design.

In December, the detailed design of the plastic parts for the model was completed. Bids to manufacture the plastic parts were received and the successful bidder is being selected. Construction of the model core is in progress at Battelle.

In January, fabrication of the plastic parts will begin and construction of the core will continue.

P-1

P. DEVELOPMENTAL STUDIES FOR THE APPR

S. J Paprocki

Progress reported in this section is for the program concerned with the development of material specifications for the advanced APPR Core 1B. This work is part of the Alco Products program of the design and development of the APPR Core 1B.

The proposed design and operational specifications for Core 1B will result in a significant increase in performance over APPR Core 1. This increased performance will impose more severe requirements on the fuel material. The rate of burnup and total burnup will increase and the fuel core temperature will be raised. In order to achieve the higher constant power output over an equivalent core life the loading of boron burnable poison will also be increased.

The average burnup of the proposed Core 1B fuel is 38 per cent and the peak burnup is 77 per cent of the contained uranium-235. It will require approximately 12 months to achieve the peak burnup of 77 per cent utilizing a flux in excess of 3×10^{14} nv. All of the effort on this program over the next several months will be directed toward the design and fabrication of irradiation capsules and the development of reference and alternate fuel materials incorporating boron as a burnable poison.

Encapsulation Studies

W. E. Murr, A. K. Hopkins, D. B. Hamilton, and J. H. Stang

Preliminary capsule-design studies have been initiated in the APPR Core 1B program. It is planned to irradiate ten capsules containing six fueled specimens each. The fueled cores will consist of dispersions of stainless-UO_2 and will contain 28 w/o UO_2 (highly enriched uranium) with approximately 0.048 w/o boron-10.

Basic irradiation parameters include a specimen-surface temperature of 650 F and two levels of burnup, 38 a/o and 77 a/o of contained uranium-235. In addition, the over-all program schedule demands that the irradiation phase be completed approximately 1 year after the capsules are inserted in the reactor (April-May, 1959). Consequently, the unperturbed flux requirements for the 77 a/o burnups are in the neighborhood of 3×10^{14} nv while those for the 38 a/o burnups are about 2×10^{14} nv. To obtain such fluxes, it seems necessary to irradiate the high-burnup capsules in the beryllium-reflector positions of the ETR. The low-burnup capsules may be irradiated either at the ETR or the MTR.

As now conceived, the capsules will be fabricated from Type 347 stainless steel and the specimens contained will be suspended in a NaK alloy for heat-transfer purposes. The capsules will be highly instrumented with thermocouples and heaters. The thermocouples will be standard Chromel-Alumel MgO-insulated stainless steel-sheathed units placed in the immediate vicinity of the specimens. A principal function

of the supplementary electrical heaters will be to maintain specimen temperature as burnup progresses. There are, however, limitations in this situation because (1) the original heat generation will be very high, almost 3 kw per specimen, and (2) lack of space will probably preclude the incorporation of more than six heaters (each nominally rated at 1 kw) per capsule. As things now stand, the resulting necessity to compromise appears to entail the following:

(1) The heaters will surround only the top four specimens in each capsule. Thus, the temperature of the bottom two specimens will depend on fission-heat generation and will decline steadily as burnup increases.

(2) There will be sufficient auxiliary heat available to maintain the design temperature level (650 F) until the uranium-235 burnup level has reached approximately 40 per cent. Thus, the 38 a/o burnup capsules will have the full benefit of auxiliary heat during their life but the 77 a/o burnup capsules will not. It is estimated that, in the latter cases, the specimen surface temperatures will have decreased to the neighborhood of 400 F as the 77 a/o level is approached.

During the next 2 months, the design of the capsules will be completed. A part of this effort will be devoted to evaluating ways that can be employed to improve the compromise situation described above. One promising avenue here is the utilization of heaters with a nominal rating higher than 1 kw; however, such units having configurations suitable for encapsulation practice are, insofar as is now known, not standard commercial items.

Development of Fuel Materials

S. J. Paprocki, D. L. Keller, G. W. Cunningham, J. B. Fox,
D. E. Lozier, and W. M. Pardue

The objective of these studies is to develop fuel, absorber, and suppressor materials for the APPR Core 1B. Because of the long irradiation time that is required to achieve reference burnups, all of the immediate materials research effort is being directed toward the development of reference and alternate fuel materials incorporating boron burnable poison. Test specimens will be fabricated, irradiated, and evaluated to select the fuel possessing the optimum burnup potential.

Approximately 70 g of boron-10 is required in the APPR Core 1B as a burnable poison. It is believed that the most desirable method for introducing the burnable poison is as a uniform dispersion within the fuel-element cores. Furthermore, it would be most desirable to add the boron as a stable, nonreactive compound of high boron content, thereby preventing boron diffusion into the cladding. In order to minimize irradiation damage the boron should probably be in the natural rather than the enriched form.

P-3 and P-4

During the past month compatibility studies of natural boron compounds dispersed in prealloyed Type 347 powder and elemental chromium, iron, and nickel powders were begun. Initial studies were conducted in both a hydrogen atmosphere and in a vacuum.

Cold-pressed compacts of ZrB_2 dispersed in Type 347 stainless steel, chromium, iron, and nickel were sintered in a dry hydrogen atmosphere for 3 hr at 2300 F. The temperature and time selected represent the severest conditions which might be imposed during normal fuel-element processing. In each of the tests the ZrB_2 reacted extensively with the metallic matrix.

A second series of specimens consisting of both ZrB_2 and TiB_2 dispersed in Type 347 stainless steel, iron, and chromium, and CrB_2 dispersed in Type 347 stainless steel was heat treated in vacuum at 2300 F for 3 hr. During the heat treatments some volatization occurred, being most noticeable with the TiB_2 series. Initial metallographic examination of these specimens shows that (1) the reactions between ZrB_2 and the metallic matrices are greatly reduced in vacuum as compared with those which occurred in hydrogen, (2) there is a slight reaction between ZrB_2 and TiB_2 and Type 347 stainless steel, (3) there is a more noticeable reaction between both ZrB_2 and TiB_2 chromium, but the reaction extends to a depth of only a few microns of the surfaces of the boron-containing particles, (4) there is a still more obvious reaction beween the ZrB_2 and TiB_2 and the iron, and (5) there appears to be no reaction between the CrB_2 and the stainless steel.

As yet the tests are too preliminary to present an accurate explanation for the results observed; however, it is probable that the oxygen contained in the metal powders and perhaps in the atmospheres is playing a major role in the reactions. For example, in the iron powders, where the oxygen content is high and the affinity is low, the reaction is most noticeable. If the boron compound getters the oxygen, the resulting boron oxide will subsequently volatize, explaining both the volatization during sintering and the appearance of the reaction zone in the particles.

Tests are now under way to investigate the use of low-oxygen metal powders. Also, these same boron compounds will be heated in both hydrogen and vacuum to isolate any reactions which might occur with the atmosphere or as a result of thermal decomposition.

Other boron compounds whose compatibility with stainless steel will be investigated in both hydrogen and in vacuo include CrB, CrB_2, FeB, FeB_2, MoB, NbC_2, NiB, Ni_2B, SiB, and pure boron.

In addition to the compatibility studies with cold-pressed and sintered compacts, clad fuel elements have been fabricated with additions of 2 w/o ZrB_2 and TiB_2. These elements contained either 28 w/o UO_2 or 24 w/o UN dispersed in both prealloyed and elemental Type 347 stainless steel. The elements were hot rolled at 2000 F in evacuated packs without prior sintering of the core compacts. Preliminary metallographic results indicate a slight reaction between the matrix and boron compounds in those elements containing UO_2 and little or no reaction in the elements containing UN as the fuel material. Further studies on these elements will include additional heat treatments at temperatures above 2000 F and more detailed metallographic examination.

RWD:CRT/all