UNCLASSIFIED

MASTER

CONFIDENTIAL

Report No. BMI-1286
C-25 Metallurgy and Ceramics
(M-3679, 21st Ed.)

Contract No. W-7405-eng-92

PROGRESS RELATING TO CIVILIAN APPLICATIONS DURING AUGUST, 1958

by

Russell W. Dayton
Clyde R. Tipton, Jr.

Classification cancelled (or changed to UNCLASSIFIED)
by authority of Str. Fl. D. J. Carroll, Chief Declassification Br. 10-15-58
by J. C. Rideout TIE, date 10-17-58

September 1, 1958

RESTRICTED DATA

This document contains restricted data as defined in the Atomic Energy Act of 1954. Its transmittal or disclosure of its contents in any manner to an unauthorized person is prohibited.

BATTELLE MEMORIAL INSTITUTE
505 King Avenue
Columbus 1, Ohio

CONFIDENTIAL

UNCLASSIFIED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

TABLE OF CONTENTS

TABLE OF CONTENTS
(Continued)

REPORTS RELATING TO CIVILIAN APPLICATIONS ISSUED DURING AUGUST, 1958

BMI-1275 "Examination of Factors Affecting the Quality of Vacuum Induction-Melted Uranium", by Roy W. Endebrock, Ellis L. Foster, Jr., and Ronald F. Dickerson.

BMI-1276 "Theoretical Studies of the Solidification of Uranium Castings", by Billie L. Fletcher, Ellis L. Foster, Jr., Charles K. Franklin, Andrew Lechler, Benjamin L. Schwartz, and Ronald F. Dickerson.

BMI-1280 "Progress Relating to Civilian Applications During July, 1958", by Russell W. Dayton and Clyde R. Tipton, Jr.

A-1

A. DEVELOPMENTS FOR ZIRCONIUM-CLAD FUEL ELEMENTS

F. R. Shober

Equipment has been constructed and methods are being explored to determine the effect of irradiation on the thermal and electrical conductivities of encapsulated uranium and on the thermal conductivity of UO_2. The thickness of the NaK between the specimen sample and the Zircaloy-2 cladding does not appear to influence the accuracy of the thermal-conductivity measurements. Long-time creep tests of 15 per cent cold-worked Zircaloy-2 have been in progress for approximately 7000 hr at 290, 345, and 400 C.

Seven high-strength zirconium alloys have been selected for further study on the basis of their corrosion resistance in 300 C water and hot-hardness values at 300 C.

Work on the modification and improvement of the experimental method of sink-float density measurements has been started. This method is to be used to identify factors affecting irradiation-induced volume changes in graphite. The time and temperature parameters necessary to grow single crystals of molybdenum in a modified Andrade-type furnace are being established.

Thermal Conductivity of Uranium and UO_2

C. F. Lucks and H. W. Deem

Equipment has been constructed and methods explored to determine the effect of irradiation of the thermal and electrical conductivities of uranium and on the thermal conductivity of uranium oxide.

Uranium

Measurements of thermal conductivity and electrical resistivity were continued on unirradiated specimens clad in Zircaloy-2 with NaK as the heat-transfer medium. Efforts were directed toward improving the accuracy of the measurements. Improvements were made in the techniques of making electrical-resistivity measurements, and the results to 750 C on one Zircaloy-2 specimen are not in error by more than ±5 per cent. Thermal-conductivity measurements on the same specimen showed errors of less than ±5 per cent to 300 C, but were outside this limit at higher temperatures. An effort is being made to improve accuracies at higher temperatures. The last clad Zircaloy-2 specimen measured was surrounded by a layer of NaK 0.040 in. thick, and there seemed to be no significant difference in the accuracy of measurements with this thickness of NaK over a thickness of 0.010 in. on a specimen previously measured.

During the next month measurements on other clad specimens will be continued with emphasis on the uranium-1.5 w/o zirconium and uranium specimens. After the unirradiated specimens are measured they will be irradiated and the measurements repeated in the hot cells.

Uranium Oxide

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

Three materials have been used to calibrate the apparatus: (1) clear fused quartz, which has a thermal conductivity lower than that expected for UO_2; (2) a titanium-6 w/o aluminum-4 w/o vanadium alloy, which has a conductivity at lower temperatures about the same as UO_2 and at elevated temperatures somewhat higher than UO_2; and (3) Inconel, which has a conductivity some five times that of UO_2 at higher temperatures.

The apparatus is very sensitive to guarding, and, because the guard cylinder is a ceramic material, it was possible to guard the quartz and titanium-alloy specimens better than the high-conductivity Inconel specimen.

Over a temperature range from 150 to 450 C, the average of the observed thermal-conductivity values for quartz was 4 per cent higher than the literature values. The observed values for the titanium alloy averaged 7 per cent lower than the literature values, and for Inconel the observed values were about 10 per cent lower than the literature values. With the experience in guarding gained during the calibration runs, it should be possible to reduce errors of future measurements.

It is noted that the experimental values for quartz were higher than literature values while the experimental values for the titanium alloy were lower than literature values. The expected thermal conductivity of UO_2 is between quartz and the alloy and for that reason the observed thermal conductivities of UO_2 should fall in the accurate region of the apparatus.

A change in thermal-insulation material will be made which will increase the thermal resistance between the specimen and guard cyclinder. Increasing this thermal resistance will decrease the effect of temperature unbalances between the specimen and guard cylinder.

During the next month, thermal-conductivity measurements will be started on unirradiated UO_2 specimens. Measurements made on UO_2 specimens of different densities will be a test of the apparatus to measure small differences in thermal conductivity.

Mechanical Properties of Zirconium Alloys

F. R. Shober and J. A. VanEcho

The creep strength of 15 per cent cold-worked Zircaloy-2 is being determined at elevated temperatures and evaluated as a part of a program to provide design data for

reactor components in the 290 to 400 C temperature range. Creep data are obtained from two types of tests; short-time stress rupture and long-time creep tests (10,000 hr or more). The time for initiation of third-stage creep and the total deformation associated with it are of specific interest. The ability of cold-worked zirconium to retain the additional strength achieved by cold working under sustained load at elevated temperatures is also being studied.

Twelve creep units are in use at the present time, with ten being used for long-time creep tests. It is expected that these tests will continue for 10,000 hr or more.

Three additional tests are to be started, two at 400 C at 30,000 and 35,000 psi, and one at 290 C and 37,500 psi. As tests are completed in the units presently occupied, it is planned to start a series of creep tests on annealed and cold-worked material in which the test temperature is cycled. The two test-temperature conditions are one series to cycle from room temperature to 290 C and a second series to cycle from room temperature to 345 C. The sequence of the cycle is to be approximately 6 days (144 hr) at the elevated temperature and 1 day (24 hr) at room temperature. The cycle will be repeated for a minimum test period of 1000 hr. Test stresses selected include stresses at which constant-temperature tests at 290 and 345 C have been conducted. A comparison of data from the constant-temperature tests and cyclic-temperature tests will be made to evaluate the effect of cyclic-temperature conditions on the creep properties of annealed and 15 per cent cold-worked Zircaloy-2.

Development of High-Strength Corrosion-Resistant Zirconium Alloys

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

The successful operation of reactors at elevated temperatures is dependent, in part, upon the development of fuel-cladding and structural materials with properties to withstand these temperatures. Neutron economy is of the upmost importance in power reactors; therefore, a higher strength zirconium alloy with satisfactory corrosion resistance is desirable. The development and use of stronger alloys can reduce the amount of zirconium used in the reactor system or allow higher reactor operating temperatures. It was believed that zirconium-base alloys having a 0.2 per cent offset yield strength of 35,000 psi at 300 C and good corrosion life in 300 C water could be developed. The objective of this program was the selection of the more promising alloys by screening approximately 100 compositions. Screening was based on room-temperature and 300 C hardnesses and corrosion properties in 300 C water. Hardness was used as an indication of the relative strengths of alloys prepared from the same base material. The expansion coefficients, nuclear properties, and thermal conductivities of the selected alloys should be similar to those of Zircaloy-2.

Nine series of alloys were prepared. They consisted of ternary zirconium-base alloys containing 2.0, 3.0, and 4.0 w/o tin plus 0 to 2.0 w/o molybdenum, ternaries containing 2.0, 3.0, and 4.0 w/o tin plus 0 to 3.0 w/o niobium, and quaternary alloys containing 2.0, 3.0, and 4.0 w/o tin plus 0.5 to 2.0 w/o molybdenum and 1.0 to 3.0 w/o niobium.

The alloys were fabricated and corrosion tested, and hardness measurements were made at room and elevated temperatures. The following conclusions were drawn from an evaluation of the data:

(1) The alloys having the desired combination of good corrosion resistance and high hardness are:

   (a) Zirconium-2.0 w/o tin-0.5 w/o molybdenum

   (b) Zirconium-2.0 w/o tin-2.0 w/o niobium-0.1 w/o iron-0.5 w/o nickel

   (c) Zirconium-2.0 w/o tin-3.0 w/o niobium-0.1 w/o iron-0.5 w/o nickel

   (d) Zirconium-3.0 w/o tin-0.1 w/o iron-0.5 w/o nickel

   (e) Zirconium-3.0 w/o tin-0.5 w/o molybdenum-0.1 w/o iron-0.5 w/o nickel

   (f) Zirconium-3.0 w/o tin-0.5 w/o molybdenum-1.0 w/o niobium-0.1 w/o iron-0.05 w/o nickel

   (g) Zirconium-4.0 w/o tin-0.5 w/o molybdenum

(2) The addition of tin improves both the strength and corrosion resistance of zirconium. At the 4.0 w/o tin level a slight deleterious effect on the rolling properties was noted during fabrication at 850 C.

(3) The addition of molybdenum up to 2.0 w/o improves the strength of zirconium considerably, as indicated by increased hardnesses, but amounts greater than 0.5 w/o tend to decrease the corrosion resistance.

(4) The addition of niobium does not improve strength greatly and does not decrease the corrosion resistance markedly.

(5) A detailed study of the room- and elevated-temperature mechanical properties of the more promising alloys is recommended before a final selection of an alloy or alloys is made.

Physical Distortion of Graphite

W. C. Riley, A. J. Roese, 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.

Modification and improvement of the experimental method are under way. The application of centrifugal force to the separation of density fractions is being studied.

Advantages of such a method may include the use of an extremely wide range of graphite particle sizes and an appreciable reduction in the time required for each determination. Equipment for testing the method has been procured, and a study of the reproducibility of separating density fractions of graphite samples is under way.

Preparation of Molybdenum Single Crystals

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

Single crystals of high-purity molybdenum are being prepared for irradiation-damage studies. A modified Andrade furnace was constructed to grow single crystals of molybdenum. The growth of single crystals from 1/4-in.-diameter stock molybdenum rod has been discontinued because sufficient power was not available to maintain the temperature high enough to grow single crystals from such large rods. An attempt will now be made to grow crystals from 1/8-in.-diameter rod.

In order to obtain increased power capacity for this purpose, relocation of the Andrade furnace was undertaken. This change has been completed, and a study of the length of time and exact temperatures required to grow molybdenum single crystals is being made. After these parameters are established, eight or ten single crystals of 1/8-in.-diameter rod will be prepared. When growth of the molybdenum single crystals has been completed satisfactorily, attempts to grow alpha-zirconium single crystals will be made.

B-1

B. DEVELOPMENTS FOR ALUMINUM-CLAD FUEL ELEMENTS

R. J. Carlson

Due to the lack of information concerning the behavior of UC under irradiation it has been deemed necessary to utilize the available physical-property data to estimate the performance of UC in a nuclear reactor. From the structure of UC it is estimated that about 74 per cent of the volume is occupied by uranium and carbon; the maximum symmetrical hole radius is approximately 0.44 A. Irradiation studies now in progress will supply experimental data concerning the ability of the UC lattice to accommodate fission fragments.

The program of study concerned with the casting of aluminum-uranium alloys in the form of hollow cylinders is continuing. A series of six ingots is being prepared for fabrication studies at another site. The thermal analyses of the aluminum-35 w/o uranium alloys containing ternary additions revealed that a number of these materials did not exhibit the thermal arrest associated with the peritectic reaction found in the binary alloys. A study of the compounds formed in aluminum-uranium alloys containing up to 83 w/o uranium has been initiated.

Preparation of Aluminum-Uranium Alloys

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

Aluminum-uranium alloys clad in aluminum have established themselves as one of the most satisfactory fuels for use in low-temperature water-cooled water-moderated reactors. These alloys – presently containing up to 20 w/o uranium – are used in the major portion of the research reactors and in many of the production-type reactors now in operation. The techniques for the production of the usual flat-plate-type fuel elements containing these alloys are well established. However, an increase in the uranium content of the alloys would result in increased neutron economy and longer fuel cycles. One reactor concept would employ aluminum-uranium alloys containing 35 w/o uranium in the form of hollow cylinders clad inside and outside with aluminum. The most advantageous method of producing such an element is by coextrusion. For this purpose a homogeneous, sound, nonporous hollow right cylinder is desirable. All work to date has indicated that the major problems encountered in casting extrusion billets of this shape and composition are segregation and porosity.

A number of castings of the aluminum-35 w/o uranium alloy have been made utilizing centrifugal-casting techniques employing a pouring spout designed to deposit the molten metal along the axis of rotation. One of the problems encountered in this method of introducing the metal into the mold is the control of the pouring rate. Freezing of the metal in the pouring spout has caused considerable delay in obtaining the desired results. However, it is believed that, with the proper design and control of the temperature of the spout, this problem will be alleviated. Using the present technique it is possible to obtain a yield of approximately 75 per cent.

Standard evaluation techniques – including chemical analyses, sectioning, and radiography – are being used to evaluate all ingots produced. A series of 6 ingots is being prepared for shipment to another site for fabrication studies.

Since the ultimate utilization of the high-uranium aluminum alloys is dependent upon their fabrication characteristics, and since alloys of this system contain compounds which are detrimental to fabricating, an attempt is being made to improve the casting and fabricating characteristics of the alloys by ternary additions. It is believed that improved fabricating characteristics can be obtained in the alloys either by refinement of the particle size of the compounds or by reduction of the amount of compound present in the alloys. For this investigation, alloy additions of 3 a/o have been made to the binary aluminum-35 w/o uranium alloy. Microscopic examination of ternary alloy specimens, press forged at 1000 F to approximately 45 per cent reduction in length, revealed that only the palladium alloy did not exhibit any evidence of microcracking. All others showed microcracks. Thermal analyses of the alloys have been completed, and the data are being interpreted. Some of the alloys exhibit no evidence of a thermal arrest which would correspond to the peritectic reaction which occurs in the binary alloys. Alloys containing lesser amounts of the ternary additions will be made to determine the lower limit of additions which will tend to inhibit the formation of UAl_4. Heat-treated specimens are being prepared and will be examined by metallographic techniques to determine the extent of compound formation that might occur during extended periods at elevated temperatures. Both quenched and furnace-cooled specimens are being used.

In an effort to more fully understand the UAl_3-UAl_4 reaction, five specimens containing 69 to 83 w/o uranium have been prepared and are being examined by metallographic techniques. These compositions were chosen to include all of the compounds known to exist in the binary system. Specimens in this series of alloys will be subjected to heat treatments similar to those given specimens containing the lesser amounts of uranium.

Future work will be concerned with improving current casting techniques and with obtaining castings suitable for fabrication studies. It is possible that the technique employed to produce the castings for fabrication will be a radical departure from the methods investigated to date. The program concerned with the ternary additions and the study of the UAl_3-UAl_4 reaction will be concerned primarily with the 35 w/o uranium alloy. However, it is thought that a comprehensive study of the alloys containing 69 to 83 w/o uranium may yield pertinent information regarding the formation of UAl_4. This information may indicate the mechanisms through which the various additions inhibit the formation or growth of the UAl_4 compound.

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

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

The collection of information from the literature pertinent to the preparation and properties of uranium monocarbide has been virtually completed. However, it is expected that new data will become available before completion of the program.

B-3 and B-4

The literature has been found especially lacking from the viewpoint of reactor-performance characteristics of UC. Consequently, in order to obtain an estimate of the behavior of UC in a nuclear reactor it was considered worthwhile to make whatever conclusions that could be drawn about this behavior on the basis of the available physical-property data. The carbide was compared with UO_2 with respect to accommodation and release of fission products. In the NaCl-type UC structure uranium and carbon account for about 74 per cent of the total volume, while in the CaF_2-type UO_2 structure the unit cell is only 64 per cent occupied by atoms. A large part of the UC lattice vacant space is composed of eight tetrahedral sites with a maximum symmetrical hole radius of 0.44 A. The largest interstitial positions in the oxide structure have a radius of about 1.0 A. The carbon, oxygen, and uranium may be viewed as being in either the atomic or ionic form without producing significant differences in the interstitial-vacancy dimensions. On the basis of these highly simplified observations it appears that the oxide could theoretically accommodate more and larger fission fragments interstitially than could the carbide. On the other hand, faster interstitial diffusion of fission products might be expected through the larger vacancies present in the oxide lattice. The more "open" lattice of UO_2, as compared with UC, might be expected to result in less lattice strain from fission fragments; but the diffusion of fission gases out of UO_2 may also be expected to be faster. The ionic nature of both compounds suggests that, up to the point of lattice rupture by fragmentation, both will accommodate fission fragments interstitially with very little change in density at temperatures below about 1200 C.

No reliable mechanical-property data exist to test the ability of the carbide to retain its integrity under conditions of strain resulting from the introduction of fission fragments into the lattice; however, it is expected that information concerning mechanical properties will be forthcoming from research presently under way. Irradiation studies now in progress will supply concrete experimental data to support or modify the theoretical forecasts discussed above.

C-1

C. RADIOISOTOPE AND RADIATION APPLICATIONS

P. Schall

A review of pertinent literature on quality-control procedures indicates that a number of analytical procedures used in cement-products manufacture could be greatly simplified by the use of radiochemical techniques. Experimental work has been started on the development of a radiometric titration technique for analysis of MgO in cement as the first specific application to be investigated.

In the investigation of the radiation chemistry of inclusion compounds it has been found that thiourea compounds prepared thus far do not have sufficient thermal stability to be suitable for irradiation studies. Of the urea compounds which have been prepared only cetane and stearic acid have suitable thermal stability. These materials are currently being irradiated.

Development of Radioactive-Tracer Quality-Control System

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

An investigation has been undertaken to establish the feasibility of the use of radioactive tracers for industrial quality control. The work, thus far, has consisted of a literature survey directed toward two purposes: First, the selection of a manufacturing process to which radioactive-tracer techniques are applicable with a resultant saving of time and money. Standard ASTM analytical procedures used in various operating processes were studied for the purpose of making this selection. Second, recent developments in the use of radioactive tracers as analytical tools were reviewed with special attention given to isotope-dilution techniques.

As a result of this study the cement industry was selected for the application of radioactive-tracer quality control. In the production of cement, several sets of chemical analyses are necessary. First, the raw materials (limestone, cement rock, slag, etc.) must be analyzed to assure that the proper mixture is fed into the kiln. Second, the finished product must pass standard ASTM chemical analyses. Finally, the cement may be analyzed by the retailer and/or buyer before it is finally used.

The required chemical analyses are given in the ASTM Handbook on Cement*. Some of these analyses require from 3 to 5 hr while others (MgO and sulfur trioxide) require at least 12 hr. If these analysis periods were shortened, considerable time and money could be saved. Thus, the usefulness of radioactive tracers in quality control would be clearly demonstrated.

Experimental work has begun to develop a radiometric titration procedure which will shorten and simplify the determination of MgO in cement. This procedure involves

*ASTM Standards on Cement, American Society For Testing Materials, Philadelphia, Pennsylvania (1955).

the titration of magnesium with standard phosphorus-32 labeled disodium phosphate solution. The end point is determined by counting aliquots of the supernatant liquid. When the radiophosphorus is detected in the supernatant liquid, the end point has been reached. Usually this type of procedure may be considerably simplified by determining only two points on the radiometric titration curve. From these two points, the quantity of disodium phosphate required to reach the end point can be calculated.

Consideration is also being given to applying radioisotope-dilution techniques to other cement analyses, and results will be reported as the work is completed.

Radiation Chemistry of Inclusion Compounds

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

The radiation chemistry of selected inclusion compounds of hydrocarbons and their derivatives is being investigated for potential application in the general area of radiation processing. The selection of four urea and two thiourea inclusion compounds for the preliminary irradiation experiments in the Battelle cobalt-60 irradiation facility was reported in BMI-1280.

Thiourea Compounds

Thiourea compounds of cyclohexane, isooctane, and cumene were prepared during August. After storage of the cyclohexane and isooctane compounds in dry air for 1 week at ambient temperature, the compounds were dissolved in water and the ratio of urea to hydrocarbon determined. This ratio increased 30 to 50 per cent over the prestorage values, showing that the thermal stability of these two compounds at ambient temperature is poor. For this reason, these two compounds will not be irradiated in the program. The study of the effect of storage time on the cumene compound has not been completed. If the thermal stability of the cumene compound and other candidate thiourea compounds is not satisfactory, thiourea compounds will not be irradiated in the program. Next month a few other thiourea compounds will be prepared for thermal-stability tests and the results will be used to re-evaluate the role of this class of inclusion compounds in the irradiation program.

Urea Compounds

Of the four urea compounds selected for study, heptane, cetane, stearic acid, and amyl acetate, only cetane and stearic acid were prepared in sufficient yield and exhibited satisfactory thermal stability for the irradiation experiments. These compounds are currently being irradiated in the Battelle cobalt-60 facility. In view of the poor thermal stability of thiourea compounds reported above, the search for additional urea compounds for the irradiation experiments will be increased. The results of the thermal-stability tests on both the urea and thiourea compounds suggest that only the inclusion compounds of the relatively high-molecular-weight hydrocarbons and their derivatives will be suitable for irradiation.

D-1

D. PROCESSING OF FEED MATERIALS

E. L. Foster, Jr.

This research is part of the National Lead Company of Ohio program to improve the quality of fabricated uranium fuel slugs. To aid the Feed Materials Production Center, the effect of processing variables on quality is being studied.

The fabrication of uranium fuel slugs for production-type reactors includes many processing steps. One of these steps is the vacuum melting and casting operation that produces large ingots from charges made up of various types of fabrication scrap and as-reduced uranium. To some extent the quality of the final wrought slugs is dependent upon the factors involved in the melting and casting operations. The resolution of factors important in the solidification of the uranium ingot and the evaluation of these factors in terms of future casting requirements is the objective of this program.

In studying the solidification of uranium ingots, a mathematical model has been devised that expresses the transfer of heat from the metal as it cools and solidifies. The initial solutions of this model appear reasonable and efforts are now principally concerned with an experimental program to provide data that will be compared with the theoretical calculated results.

Solidification of Uranium

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

Time-temperature relationships are being studied during the solidification of small cylindrical castings. Temperature histories, recorded at various points in the ingot and the mold and the immediate surroundings, are being used to establish the manner and rate of solidification. The solidification patterns, so obtained, are expected to give thermal data that can be compared with theoretically computed heat-flow data. With these data and comparisons as a basis, effects of casting variables on the casting of large-scale uranium ingots may be predicted.

In making preliminary tests with small-scale castings with thermocouples positioned in the mold and ingot, excessive stresses were placed on the innermost thermocouples by the liquid metal as it was poured. Shattering of the protective covering of some of the thermocouples occurred. The molds which are used in these experiments are being redesigned to hold reinforced thermocouples so that the high stresses along the center line of the castings may be overcome. Thermocouples placed along the surface of the mold and on the furnace jacket performed satisfactorily during the initial tests.

D-2

Investigation of the mechanical events taking place at the mold-metal interface during casting and solidification is beginning. In chill castings, it is known that a gap is formed between the mold and ingot surfaces during solidification. The appearance and nature of such a gap must be considered in analytically describing the heat flow in the metal or mold. In order that a full knowledge of the events occurring at this interface region may be obtained, a process must be devised that will record the instant of gap formation and which will follow its progression along the ingot surface as separation occurs. With this objective in mind, a study is being made using a network of electrical probes in the mold. The method used consists of placing probes at the separation interface, flush with the inner mold wall, and having a common wire at the bottom of the mold which should be in contact with the metal at all times. As the liquid rises in the mold each probe makes contact with the molten metal forming an electrical circuit. By means of a recording device which notes the time at which each circuit is closed or opened, a record of the time of formation of the gap and its rate of formation along the mold wall may be obtained. Some preliminary tests using aluminum metal for casting have been performed. The results of these initial tests are being evaluated and will be discussed in subsequent reports.

E-1 and E-2

E. DEVELOPMENTS FOR LMFRE

J. McCallum

Methods for preparing a thin protective coating of molybdenum on Croloy 2-1/4 are being investigated.

Electroless Plating

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

Products from molybdenum pentachloride and molybdenum hexacarbonyl reduced by Grignard reagents were observed to be insoluble in concentrated HCl. These products were analyzed by X-ray diffraction and found to be amorphous. Because of this result and the prior negative results with the Grignard reagents, no further work is planned with the Grignard reducing agents.

A few experiments were performed with the thermal decomposition of molybdenum hexacarbonyl dissolved in liquid hydrocarbons. The object was to check out some foreign patents that claimed this to be a good coating process. The coatings obtained were powdery and occurred on the Croloy above the solution phase. No further work is planned with the thermal decomposition of solutions of molybdenum hexacarbonyl.

Future work will attempt to find solvents for the lower valent molybdenum compounds together with a suitable reducing agent.

Vapor Plating

C. F. Powell and I. E. Campbell

Molybdenum was deposited by hydrogen reduction of molybdenum pentachloride at 800 C, and with conditions believed to be optimum for obtaining sound molybdenum coatings. The coatings obtained were not completely adherent. The interface beneath the coating was usually black or discolored, indicating contamination with lower chlorides or with oxidation products resulting from outgassing of the tube walls during the preheating period. This surface contamination was prevented, and adherent coatings obtained, by the application of about 0.2 mil of electroless nickel on the Croloy before coating.

Additional test specimens of sound molybdenum coatings on Croloy 2-1/4 will be prepared using optimum coating conditions.

F-1

F. RESEARCH FOR AEC REACTOR DEVELOPMENT DIVISION PROGRAM

S. J. Paprocki and R. F. Dickerson

REACTOR MATERIALS AND COMPONENTS

R. F. Dickerson

Previous research indicates that in a binary system of UO_2-La_2O_3 about 60 w/o La_2O_3 is required to form a stoichiometric structure after oxidation. Because this results in reduced uranium loadings, attempts are being made to reduce the amount of stabilizing additive by using ceramic ternary systems. The initial composition being investigated has equal amounts of UO_2 plus 60 w/o La_2O_3 and UO_2 plus 10 w/o CaO. Bodies of this material are being tested under oxidizing conditions at temperatures of 3000 F and higher.

The specimen-chamber configuration previously used to carry out high-temperature high-pressure studies is being modified and a larger graphite heater has been adopted. These changes will increase the pressure and temperature which will be generated. In addition, a die having a greatly simplified design is being constructed. This design should further increase the temperature and pressure limits.

Investigation of the hydrides of uranium-zirconium alloys has continued. Phase boundaries are being established from hydrogen-absorption isotherms in the zirconium-25 w/o uranium alloy at 710 C. Capsules for the irradiation of fueled zirconium hydride specimens have been prepared and will be delivered to the MTR.

Specimens of Type 347 stainless steel have been inserted in the ETR for exposure to fast-neutron flux. These particular specimens will be irradiated at process-water temperature. The core-filler piece needed to accommodate the required dosimetry will be inserted in the ETR. An instrumented capsule will be placed in the F-10-NW position for the purpose of correlating specimen temperature with gamma flux. When this has been done seven capsules designed to make use of gamma heat will be loaded into the proper test hole.

Valence Effects of Oxide Additions to Uranium Dioxide

W. B. Wilson and C. M. Schwartz

Experimental work has been initiated to reduce, if possible, the excessive amount of additive needed to prevent oxidation of UO_2 to U_3O_8. Previous research indicated that in a "binary" system such as the UO_2-La_2O_3 system nearly 60 w/o La_2O_3 is required to form a stoichiometric structure after oxidation. Since this results in waste of space where high specific core loadings are required in a reactor, it would appear desirable to reduce the amount of additive. One approach to this is to go to a ceramic ternary system (quaternary) utilizing principles previously discussed, where the

additional additive has a +2 valence. A composition having equal amounts of UO_2 plus 60 w/o La_2O_3 and UO_2 plus 10 w/o CaO has been selected as a starting point for this approach.

Other compositions will be evaluated utilizing the UO_2 plus 60 w/o Y_2O_3 composition as a base. This material appears more suitable for fuel use, in view of the smaller yttrium cross section.

In addition, it was previously pointed out that better UO_2-ThO_2 materials may possibly be produced by substituting UO_2-60 w/o La_2O_3 for the UO_2. While this may be unnecessary for a high ThO_2 content, it is apparently necessary to prevent rupture and/or vaporization of uranium at a higher UO_2 content.

Bodies of each of the above materials have been prepared and are currently being tested under oxidizing conditions at temperatures of 3000 F and higher.

High-Pressure High-Temperature Solid-State Studies

W. B. Wilson and C. M. Schwartz

The specimen-chamber configuration previously used to carry out high-temperature high-pressure studies is currently being modified. A significantly larger graphite heater has tentatively been adopted for a twofold purpose. The present equipment permits use of the "bomb" technique where it is planned to seal the pressure apparatus at 30,000 atm. Further pressure and temperature will be generated by internal electrical heating of the larger graphite heater to high temperature. Following temperature calibration, it is planned to further study the behavior of UO_2 with BeO and other oxides with this heater system.

A greatly simplified advanced die design has been proposed which is intuitively more attractive than the "geometrical-advantage" unit previously described. Construction of the unit is in progress.

Previous solid-state studies have been conducted at a temperature range (~ 1000 C) which is below that anticipated to be necessary for reaction of refractory materials. Attention will now be directed toward obtaining the temperature range required to produce reactions and at pressures which are higher than previously utilized.

Fueled Zirconium Hydride Moderator

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

Investigation of the hydrides of uranium-zirconium alloys for possible use in gas-cooled reactors has continued. Efforts during August were directed toward determination of additional isotherms in the zirconium-25 w/o uranium system, and the completion of capsules for radiation-damage studies of fueled-moderator samples.

F-3

Structure and Pressure-Composition-Temperature Studies

Rough data for the hydrogen-absorption isotherms in the zirconium-25 w/o uranium alloy at 710 C have been obtained. Processing of these data to establish the corresponding phase boundaries is now under way.

Hydrided specimens of the zirconium-1 w/o uranium alloy and the zirconium-50 w/o uranium alloy were sealed into quartz tubes in an argon atmosphere in preparation for high-temperature X-ray diffraction studies. The sealed samples were homogenized by heat treatment at approximately 1000 C before exposure to X-rays for structural determinations.

Hydrogen-absorption isotherms will be obtained at other temperatures, and X-ray diffraction work will be continued during the next month.

Radiation-Damage Studies

The final assembly and NaK loading of Capsule BMI-20-1, designed for irradiation at 1500 F in the MTR, is very nearly completed. The fueled-moderator specimens are being loaded into Capsule BMI-20-2, designed for irradiation at 1100 F. Both capsules should go to the MTR next month.

Irradiation Surveillance Program on Type 347 Stainless Steel

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

An irradiation-damage program on Type 347 stainless steel has been undertaken to study the changes in physical properties that are caused by continued exposure to a fast-neutron flux up to 3×10^{15} nvt and integrated flux levels of 14 to 16 x 10^{22} nvt. Changes in properties associated with integrated flux levels of 2.4, 4.8, 7.2, 9.6, 12, and 14.7 x 10^{22} nvt will be evaluated based on changes observed in the mechanical properties of the stainless at room temperature and 600 F. These properties include tensile, cyclic-strain-fatigue, and impact properties.

Eight "cold" capsules (those which are being exposed at ETR process-water temperature) are in the F10-NE and F10-SE positions in the lattice of the ETR. It is planned to discharge one capsule containing tensile specimens and cyclic-strain-fatigue specimens at intervals equivalent to a 6-month exposure of the KAPL-33 loop in the F-10 position. Since the experiment now occupies and will continue to occupy the F-10 position until the loop is installed, capsules will be discharged at 6-month intervals.

The C-4X core-filler piece to accommodate the desired dosimetry will soon be shipped to the ETR. The "mock-up" capsules containing the same weight per unit length of Type 347 stainless steel as the capsules in the ETR are being prepared for the ETRC. These capsules will be sent to the ETRC in September.

F-4

An instrumented capsule will be placed in the F-10-NW position with a 14-in. spacer to locate it in the center portion of the test position. The temperature of the capsule will be recorded throughout a complete ETR cycle to correlate the temperature of the specimens with the gamma flux. This temperature excursion is expected to occur during Cycle 6 of the ETR, scheduled to start in September. Upon completion of the evaluation of the temperature in the test position, the seven remaining capsules will be loaded into the F-10-NW and F-10-SW positions. These capsules have been designed and constructed to operate at approximately the same temperature as the lead capsules. The capsules contain tensile, cyclic-strain-fatigue, and impact specimens which are to be irradiated at elevated temperatures, preferably 600 F. All capsules will remain in the F-10 position until such time that the KAPL-33 loop is ready for installation in the F-10 position at which time all will be moved to the H-10 or a comparable position.

A dosimetry program for determination and evaluation of the neutron flux is being planned and will be initiated upon installation of the core-filler piece. It is planned to obtain some measure of fast-neutron flux, with energies greater than 1 Mev by use of nickel and sulfur dosimeters in enough locations to determine any variation of the fast-neutron flux along the vertical plane of the test hole. A cadmium-shielded cobalt wire can be used to determine the neutron flux with energies above 0.4 ev in the same location. Subtraction of this value from the flux as determined by nickel and sulfur will give some value of the neutron flux in the 0.4-ev to 2.5-Mev range or epithermal-energy range.

The cyclic-strain-fatigue properties of the unirradiated Type 347 stainless steel will be determined after suitable apparatus has been constructed.

STUDIES OF ALLOY FUELS

R. F. Dickerson

The study concerned with the properties of niobium-uranium alloys and the effect of impurities on these properties has been delayed due to slow delivery of the niobium needed. The necessary melting will begin as soon as the material becomes available.

As an initial step in the development of thorium-uranium alloys, a series of calculations has been made which indicate that 1.5 per cent volume increase per 1 a/o burnup can be expected for a thorium-10 w/o uranium alloy. Further calculations indicate that if the uranium particles are less than about 7 μ in diameter, the fission fragments will lodge in the matrix. Iodide thorium has been received and alloys are being prepared for initial study.

Development of Niobium-Uranium Alloys

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

The properties of alloys of the high-niobium portion of the niobium-uranium constitutional diagram are of interest because of the possible use of these alloys as nuclear

fuel materials. Past investigations have been limited to the uranium-rich portion of the niobium-uranium diagram (less than 20 w/o niobium). It is evident from the results of these investigations that the properties of uranium-niobium alloys are influenced greatly by the impurities present in the uranium and niobium. The present studies are concerned not only with the properties of the high-niobium alloys but also with the effects of the two major impurities, oxygen and zirconium, on the properties of niobium alloys containing 10 to 60 w/o uranium. The mechanical and physical properties will be studied at room and elevated temperatures. The cold- and hot-fabrication characteristics will be investigated and the effect of several corrosive atmospheres will be studied at temperatures of interest.

Alloys of uranium with 40, 50, 60, 70, 80, and 90 w/o niobium are to be prepared with three grades of niobium melting stock. These are, niobium with less than 300 ppm oxygen and less than 200 ppm zirconium, niobium containing approximately 800 ppm oxygen and less than 1.0 w/o zirconium, and niobium containing 800 ppm oxygen and less than 0.5 w/o zirconium.

Fabrication temperatures will be selected on the basis of previous fabrication studies, and on the basis of results of hot-hardness tests on special buttons cast for this purpose. Measurements of hot hardness will be obtained from 200 to 900 C, and it is believed that the results will provide a guide for selection of a fabrication temperature.

The alloys will be prepared as 3 to 5-lb ingots by consumable-electrode arc-melting techniques, and they will be fabricated by either rod rolling, conventional rolling, forging, and press forging or by a conbination of these techniques. Molybdenum cans may have to be used in rod rolling since prior investigations have shown a need for lateral restraint while fabricating certain of the uranium-rich alloys.

The mechanical and physical properties of alloys successfully fabricated will be investigated at room and elevated temperatures. A study of the effect of heat treatment upon properties will be made. The final phase of the program will be a study of the corrosion resistance of the alloys in sodium, water, carbon dioxide, and air at various temperatures, depending upon the corrosive environment. Tests will be conducted in sodium up to 890 C, the boiling point of sodium, in water at 300 C, in carbon dioxide at 600 C, and in air at 400 C.

The niobium to be used for melting stock is on order. Casting of the required ingots will proceed as soon as materials are received.

Development of Thorium-Uranium Alloys

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

A project aimed at developing thorium-uranium alloys with improved irradiation stability and corrosion resistance has begun. Approaches being considered are the control of distribution and particle size of the alpha-uranium phase, alloying to improve the properties and fission-gas-retention characteristics of the uranium phase, and alloying to increase the strength and corrosion resistance of the alpha-thorium phase. Improvement in corrosion behavior of the alloys in hot water is desired.

Calculations for a thorium-10 w/o uranium alloy show that a 1.5 per cent volume increase per 1 a/o burnup can be expected assuming that the fission fragments are introduced as an additive volume. Further calculations indicate that, considering the fission-fragment range in uranium, if the uranium particles are less than about 7 μ in diameter, the fission fragments will lodge in the thorium matrix. The importance of strengthening the thorium matrix by alloying is, therefore, indicated.

A comparison of the alpha- and beta-thorium structures was performed since some consideration was given to the possibility of alloying to stabilize the beta structure if improvement in either corrosion resistance or irradiation behavior could be expected. The comparison favors the alpha structure for irradiation resistance. Face-centered-cubic alpha thorium can be expected to show slightly lower volume changes, assuming substitutional positions for fission atoms, for a given burnup than body-centered-cubic beta thorium. The face-centered-cubic structure can accommodate a larger interstitial atom than can the body-centered-cubic structure. Fission-gas-diffusion rates and notch sensitivity can also be expected to be greater in the body-centered structure.

An initial supply of iodide thorium for the preparation of alloys has been obtained. Additional material is on order. Binary alloys containing 5 to 20 w/o uranium are to be cast initially, and will be investigated to determine the effects of casting variables, impurities, fabrication techniques, and heat treatments on microstructure.

GENERAL FUEL-ELEMENT DEVELOPMENT

S. J. Paprocki

Dispersions consisting of enriched UC and UN dispersed in a stainless steel matrix have been prepared, encapsulated, and forwarded to the MTR for irradiation. The irradiation behavior of these specimens will be compared with UO_2 dispersions containing an equivalent uranium-235 loading. Reports of this work will be resumed when irradiation-test data are available.

Investigations are being conducted to develop fabrication techniques for preparing dispersion fuel elements containing 60 to 90 volume per cent of UO_2, UN, and UC dispersed in stainless steel. These materials are of interest because they combine the advantages of both the conventional cermet and ceramic fuel. It is anticipated that the UO_2 dispersion will possess improved thermal conductivity over a pure UO_2 body because of the presence of the stainless steel phase and absence of severe cracking that is encountered in ceramic UO_2 material.

A technique utilizing gas pressure at elevated temperatures is being investigated as a bonding method for the cladding and joining of niobium and molybdenum fuel elements and assemblies. Initial attempts to bond niobium and molybdenum at temperatures of 1650 and 1750 F were unsuccessful. Future bonding tests will be conducted at higher temperatures.

In the pressure-bonding process, the initial step involves deformation of the materials to obtain intimate contact. At the present, the time, temperature, and pressure required to achieve this condition are determined by trial. It is believed that this

deformation is governed by a secondary-creep process. Torsion tests will be conducted to determine if creep parameters from this test can be applied to the basic creep law. The ultimate objective is to relate the creep properties of materials to their stress behavior during pressure bonding.

Fabrication of Cermet Fuel Elements

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

Fabrication techniques for producing dispersion fuel elements with cores of 60 to 90 volume per cent UO_2, UN, or UC dispersed in stainless steel, molybdenum, niobium, or chromium are being investigated. Core evaluations will be made on the basis of physical and mechanical properties.

Cores containing 75 volume per cent UO_2 dispersed in Type 302B prealloyed stainless steel have been hot press forged at 1900 F in evacuated stainless steel frames to determine the effect of total reduction in the microstructure and density. Reductions were varied from 44 to 55 per cent, resulting in theoretical densities of 82 to 91 per cent, respectively. All dispersions were uniform with continuous metallic skeletons. Forgings at 1900 F were done in Type 304 stainless steel packs. Cores of the same composition, prepared in the same manner, were also press forged in Type 318 stainless steel packs at 2000 and 2100 F. Reductions of 29 per cent at 2000 F resulted in a core density of 93 per cent of theoretical, while a theoretical density of only 88 per cent was realized with reductions of 29 per cent at 2100 F. This decrease in density at the higher forging temperature is probably due to the increased plasticity of the pack and stainless skeleton resulting in decreased lateral restraining forces and, consequently, a decreased hydrostatic-pressing effect.

Hot-press-forged cores of 80 volume per cent UO_2 dispersed in molybdenum and niobium are being prepared to determine the effect of UO_2 particle size upon the density and microstructure. Also, pack-design alterations are being made in an attempt to produce desirable core shapes for physical and mechanical testing.

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

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

The high-temperature strength and favorable nuclear properties of molybdenum and niobium make them promising structural materials for use in high-temperature inert-gas-cooled and some liquid-metal-cooled reactors. A conventional fabrication technique has not been developed for cladding of fuel elements with these materials. They are highly susceptible to oxidation; consequently, they must be protected with an oxidation-resistant outer container during roll cladding or coextrusion. The container materials that are usually employed alloy with the molybdenum and niobium during high-temperature fabrication. The molybdenum and niobium also offer much more resistance to deformation at the elevated bonding temperatures. Many of these problems associated with conventional fabrication techniques for cladding with molybdenum or niobium can be avoided by pressure bonding of these materials with an inert gas pressure at elevated temperatures.

F-8

In the initial phases of this investigation, bonding of either molybdenum or niobium to itself is being studied. The effect of prior fabrication history and surface preparation on the bonds obtained during pressure bonding is being investigated. A series of flat-plate specimens incorporating two flat plates of molybdenum and niobium was subjected to different surface-preparation treatments to study the effect on bonding of these materials. The specimens were bonded at 1650 and 1750 F for a period of 6 hr at a helium pressure of 10,000 psi. Preliminary evaluation of these specimens revealed only partial bonding. The niobium had not recrystallized and only partial recrystallization of the molybdenum was observed. No recrystallization of either material was observed along the mating surfaces after bonding.

Additional specimens have been assembled for bonding at temperatures in excess of 1750 F. The base materials will be cold worked severely in an attempt to produce recrystallization and grain growth across the bond interface at a lower temperature, as increased cold work significantly lowers the recrystallization temperature of both materials. These specimens will be studied metallographically to evaluate bond quality as a function of surface preparation and prior fabrication history.

Basic Studies of Pressure Bonding

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

An investigation of the gas-pressure-bonding process is being conducted. This process is a technique for the bonding of surfaces by the application of elevated temperatures and external pressure. In this study emphasis is placed on determining the conditions with which the mating surfaces will deform sufficiently to insure intimate contact.

Analytic Studies of Pressure Bonding

The deformation of the surface irregularities is assumed to be governed by the basic creep law, $\dot{\epsilon} = A\sigma^n$, which is a widely accepted relationship representing second-stage creep. The present effort in the analytic portion of the investigation is directed toward the determination of a suitable geometric model to represent the contours of the mating surfaces which will at the same time permit solution with a reasonable effort. Several models have been considered thus far. None of these models yielded a manageable mathematical solution.

Experimental Studies of Pressure Bonding

Preparations have been completed for running a series of torsion tests on 2S aluminum at 500 and 700 F. Data from these tests are expected to provide the creep parameters needed for the basic creep law. These values may then be used to predict the deformations of thick-walled circular cylinders under hydrostatic pressure. The predicted deformations may be compared with previously obtained experimental measurements as a check of the validity of the theoretical analysis.

G-1 and G-2

G. FATIGUE STUDIES OF INCONEL AND INOR-8

F. H. Lyon

Fatigue Studies of Inconel

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

During August a new series of calibration runs was made. Previously the fatigue apparatus was adjusted to produce a given load on a specimen while the specimen was at room temperature. When the specimen reached the temperature of the test, additional adjustment of the load was required. It will now be possible to adjust the load mechanism at room temperature so that a desired load at a given elevated temperature will automatically result. Thus, the elimination of the intermediate load adjustment makes it possible to record the first strain cycle at elevated temperature. Further modifications and refinements are planned next month. In addition, the instrumentation system as a unit will be calibrated and proof checked so that the resulting fatigue and strain data can be translated into quantitative units.

Fatigue Studies of INOR-8

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

Little has been accomplished on the program since the preliminary studies. When material for specimens arrives, machining of fatigue specimens and accumulation of data may begin. In the interim a second fatigue-testing machine will be modified to accommodate high-temperature equipment.

J-1

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

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

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

No severe attack of titanium specimens has been observed in any of the studies involving Darex solutions.

Stress-relief treatments have not been effective in improving the corrosion resistance of Carpenter 20 Cb to Sulfex-Thorex solutions. A stabilized grade of Nionel and an extra-low-carbon and -iron form of Illium are being evaluated in these solutions.

Studies of the fluoride-volatility process have shown fairly high corrosion rates, particularly at interface areas with coupons of INOR's and Hastelloys, and much less attack on tubular pieces.

The Darex Process

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

Dissolver Studies With Titanium

A metallographic study of several of the titanium specimens from the 2000-hr run of the flowing dissolver was completed. Special attention was paid to the bottom of crevices and to the highly stressed areas both around welds and at the apex of specimens held in the contour of U-bends. No evidence of cracking as a result of exposure to the conditions of the flowing dissolver could be found.

Titanium specimens from the new flowing dissolver experiment were given a caustic cleaning in place following a 1000-hr exposure and were removed for examination. Eight weekly 3-hr cleaning periods with 10 w/o NaOH, including the final cleaning, were completed during this exposure. No severe attack was detected, either by observations or weight-loss measurements.

FAT Studies With Titanium

Representative titanium specimens of the various plain, welded, and stressed types, which were exposed to boiling Initial FAT solutions, were sectioned and

examined. These had received a 4500-hr exposure. No signs of cracking or severe attack associated with crevices, weldments, or highly stressed areas were observed.

All of the titanium steam tubes which had been exposed to Initial or Final FAT solutions were cleaned. No apparent surface attack of any consequence was observed. Sections have been taken through critical locations in these tubes, and will be examined metallographically.

The Sulfex-Thorex Process

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

Experiments With Carpenter 20 Cb

Specimens of Carpenter 20 Cb were exposed to boiling 6 M H_2SO_4, while stainless steel rods were dissolved in a batchwise operation. A specimen in contact with the steel and another not in contact were submerged in the boiling liquid. The acid was renewed as soon as a buildup of about 5 g per liter of dissolved stainless steel had been reached. Some 50 such renewals resulted in a total exposure of 124 hr. By that time, stress cracking was apparent on the surface of the unconnected specimen but could not be detected on the connected one. The unconnected specimen was corroding at a rate of 2.9 mils per month at the end of this period, while the rate of the connected specimen was 0.8 mil per month. These results indicate that cathodic protection of the Carpenter 20 Cb might be expected as a result of the dissolving fuel elements acting as sacrificial anodes.

Specimens of 11- and 16-gage Carpenter 20 Cb containing weldments were given one of three heat treatments. These were: (1) a solution anneal at 1950 F for 15 min followed by a water quench; (2) a stress-relief treatment consisting of holding the specimens at 1650 F for 10 min and then cooling in the furnace; and (3) a combination of these two treatments. The specimens were then exposed to boiling 6 M H_2SO_4 for several hours. No conclusive evidence was obtained concerning the effect of these treatments on the tendency for stress cracking to occur. All treatments seemed to alleviate the tendency to some extent. However, the stress-relief treatment greatly increased the corrosion rate of this material, both when it was used alone and in combination with the solution anneal. The rate of attack is too great for such a treatment to be useful, even if it did solve the cracking problem. At present, stress-relief treatments at other temperatures are being investigated.

Experiments With Nionel

A stock of stabilized Nionel (or Ni-o-nel) has been secured. Specimens have been prepared and will be studied under cyclical exposures similar to those of previous studies.

Scouting Experiments With Other Metals

Scouting experiments were conducted with welded specimens of Illium R exposed to boiling Sulfex and Thorex solutions. Intergranular attack occurred over the faces of the specimens exposed to boiling Initial Thorex solutions even with 0.2 M Al^{+3} present. This attack was more pronounced in the heat-affected areas next to the weldments. This was the only place that it occurred on the specimens exposed to boiling Final Thorex solutions. No apparent intergranular attack was observed on specimens exposed to the boiling H_2SO_4 solutions. The corrosion rates closely approximate those calculated for unwelded specimens exposed to similar solutions. A new Illium alloy (Illium 98), which has an extra-low carbon and iron content has been supplied in cast form for use in further scouting experiments.

The Fluoride-Volatility Process

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

Specimens of various materials have been exposed for 500 hr in the 62 mole per cent NaF-38 mole per cent ZrF_4 salt at 650 C to flowing HF (10 g per hr). An INOR-8 tube specimen held on a Hastelloy W gas-impingement tube showed a penetration rate of 0.43 mil per month based on the weight loss. An INOR-1 tube specimen, positioned above the piece just mentioned, showed a rate of 0.03 mil per month.

In contrast to these results, specimens in the form of coupons cut from sheet material, placed at the liquid-vapor interface, showed appreciable attack at the areas exposed near the liquid level. Table J-1 summarizes the data as calculated for the specimens based on the weight loss. The table also gives maximum penetration values (based on micrometer readings) measured at the interface where the most severe attack occurred.

Also included are values for the specimens exposed to the vapor. It can be seen that the penetration of these specimens was much less than for the interface position.

The specimens exposed to the liquid phase were held on strips of Hastelloy W about 1/8 in. wide. These strips were completely severed at the liquid level, allowing the specimens to fall to the bottom of the container. They have not yet been examined. The gas-inlet tube and thermocouple tube of Hastelloy W used in the above experiment showed no noticeable attack at the interface area or below it. Further work should furnish more information regarding these differences in corrosion rates. An experiment

is now under way using the same salt and an HF flow rate 2.5 times greater than used in the study just discussed.

Nearly 400 hr of exposure has been completed in the equimolar $NaF-ZrF_4$ salt mixture during a new study. In this experiment, specimens are being exposed to a simulated dissolver effluent atmosphere (2.4 w/o hydrogen-97.6 w/o HF).

TABLE J-1. CORROSION RESULTS IN 62 MOLE PER CENT NaF-38 MOLE PER CENT ZrF_4 AT 650 C AFTER 500 HR

Specimen	Penetration Rate, mils per month	
	Over-All, Based on Weight Change	Maximum, Based on Micrometer Measurements
INOR-8 tubing, gas impingement, immersed in liquid	0.43	--
INOR-8 coupon, vapor	0.42	None
INOR-8 coupon, interface	6.26	11.0
INOR-1 tubing, gas impingement, immersed in liquid	0.03	--
INOR-1 coupon, vapor	0.41	None
INOR-1 coupon, interface	4.47	6.0
Hastelloy B coupon, interface	3.63	6.0

K-1

K. DEVELOPMENTS FOR SRE, OMRE, AND OMR

F. A. Rough and J. E. Gates

This is part of the Atomics International research program. The objectives of this research are to develop uranium monocarbide as a fuel for 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 first phase of this study of uranium monocarbide, the development of casting techniques, determination of physical properties, and preparation of irradiation specimens, is essentially complete. The irradiation of the monocarbide is well under way, and two capsules of specimens have been opened, although the results are not yet available for reporting.

Preliminary experiments to develop techniques for the study of fission-gas release during postirradiation heating are reported.

Casting Techniques for the Preparation of Uranium Monocarbide

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

The initial phase of this program, which included the development of an experimental-scale melting and casting procedure, the determination of some of the physical properties, and the preparation of enriched as-cast specimens for irradiation in the MTR, is being concluded with the preparation of a topical report. The report will cover the techniques used to prepare the irradiation specimens and information on the physical properties and other characteristics of cast UC.

Irradiation-Capsule Design for Uranium Carbide

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

One BRR capsule irradiation and one MTR capsule (BMI-23-1) irradiation have been completed. Two more MTR capsules (BMI-23-2 and BMI-23-3) have been loaded. Capsule BMI-23-2, which contains uranium-5.0 w/o carbon specimens, is scheduled for insertion in the MTR A-28-NE position during the Cycle 110 shutdown in September. Capsule BMI-23-3 (uranium-5.0 w/o carbon specimens) is tentatively planned for insertion during the Cycle 111 shutdown in September. Capsules BMI-23-4, BMI-23-5,

and BMI-23-6 are tentatively scheduled to be loaded with uranium-5.0 and -4.8 w/o carbon and uranium-4.6 w/o carbon specimens, respectively, as specimens become available.

Capsule A.I. UC BRR No. 1 was irradiated for 1036.9 hr or about 43 days at 1 megawatt in a flux of approximately $1 \pm 0.2 \times 10^{12}$ thermal nv. Calculated core and surface temperatures, respectively, were about 660 and 600 F. Capsule BMI-23-1 was irradiated 970 MWD at 39.8 megawatts in a flux of approximately $8 \pm 0.2 \times 10^{13}$ nv thermal. Extreme measured core surface temperatures, respectively, were about 1600 and 1000 F. An analysis of temperature data will be made soon.

The long-term NaK-UC-stainless steel compatibility test at 1100 F has been terminated. Posttest examinations are in progress.

Fission-Product Release From Irradiated Uranium Monocarbide

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

The purpose 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 postirradiation heating.

Preliminary experimental work has been carried out on a sample of uranium carbide of near theoretical density. The specimen received a radiation dosage of 2.95×10^{12} nvt as determined by cobalt dosimetry.

The uranium carbide was then placed in a closed, evacuated system and heated at several temperatures up to 1700 F. Samples of gas were collected by means of charcoal traps which had been evacuated and cooled with liquid nitrogen. After exposure to the fission gas the individual traps were isolated. Xenon-133 concentrations were measured by means of gamma-ray spectrometry.

It was found that small quantities of xenon-133 were collected at temperatures as low as 1000 F. Results are shown in Table K-1.

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

Sample	Temperature, F	Time, hr	Fraction of Total Present Xenon-133 Found
A	1000	1.5	0.00040
B	1250	1.5	0.00075
C	1500	1.5	0.00012
D	1700	1.5	0.0024
E	1700	17	0.0049[a]
F	1700	47.5	0.0066[a]
G	1700	66.5	0.0070[a]

(a) Cumulative values for total times at 1700 F.

Some difficulties were encountered in maintaining a vacuum in the furnace tube used in these experiments. A new tube is under construction and a second specimen, irradiated with the first, will be checked under the same conditions.

After this method is proved, specimens with a burnup of about 500 MWD/T burnup will be studied.

POSTIRRADIATION STUDIES OF SRE, OMRE, AND OMR FUEL MATERIALS

J. E. Gates

Radiochemical analyses and metallographic examinations of irradiated thorium-11 w/o uranium specimens were delayed.

Two capsules containing UO_2 slugs were opened and the tubes containing the specimens were examined. The specimen tube from Capsule NAA-28-1 was found to be distorted. The tube from Capsule NAA-29-1 was in good condition.

Examination of the specimens of structural materials irradiated in a dummy OMRE fuel element will be initiated in September.

The irradiated OMRE fuel element, OMRE-3, has been disassembled. The interior of the fuel box was free of obstructions and distortions. A thin adherent coating of tars was observed on all parts of the element. All fuel plates were in excellent condition.

SRE Fuel Material

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

Evaluation of the data obtained from the examination of the thorium-11 w/o uranium specimens from Capsules NAA-15-6 and NAA-15-7 is in progress. The amounts of cesium-137 contained in the NaK recovered from Specimens NAA-15-6-1 and NAA-15-7-F have been determined. The analyses of gases recovered from Specimens NAA-15-6-2, NAA-15-6-3, and NAA-15-6-4 have also been completed. These data will be evaluated as soon as the burnup of the specimens is determined by radiochemical analyses.

Radiochemical burnup analyses and metallographic inspections of several specimens will be initiated next month. This work, along with measurements of the linear thermal coefficient of expansion of two specimens, will complete this program.

K-4

Uranium Dioxide Specimens

The postirradiation evaluation of uranium dioxide specimens recovered from Capsules NAA-28-1 and NAA-29-1 has been initiated. The specimens were contained in a thin sealed stainless steel tube which was immersed in NaK. The evaluation will consist of visual examinations, the making of stereomacrographs, and determination of the dimensions of each specimen tube. The length of each specimen stack will also be measured and the amount of fission gas released from the specimens from Capsule NAA-29-1 will be determined. Metallography and burnup samples will be obtained from selected portions of each specimen stack.

Both Capsules NAA-28-1 and NAA-29-1 were opened and the detailed visual examination of the specimen tubes was completed. Capsule NAA-28-1 was irradiated for approximately 2 hr at a central core temperature in the specimens of approximately 1400 C. The specimen tube from this capsule was visibly warped and distorted. Capsule NAA-29-1 was irradiated to an estimated burnup of 0.75 total a/o at a control-specimen core temperature of approximately 2000 C. There was no visible distortion of the specimen tube from this capsule. Visual examination of the specimen tube from Capsule NAA-29-1 revealed several scratches on the tube surface. These marks appear to have been caused by contact of the fuel pin with sharp edges in the capsule during irradiation or handling.

Photomacrographs, measurements of the dimensions of each specimen tube, and analyses of the gas recovered from Capsule NAA-29-1 are scheduled for next month. Metallographic evaluations and radiochemical burnup analyses will be performed on selected specimens.

OMRE Fuel Elements

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

The postirradiation examination of Fuel Element OMRE-3 is approaching completion. Evaluation of the data obtained to date is in progress.

Examination of Fuel-Element Box

The outer surfaces of the fuel-element box were examined at magnifications at 32X. All surfaces were covered by a film of tar which was determined to be approximately 0.5 mil in thickness. In some areas where the tar could be removed, the metal surfaces of the box were shiny and revealed no indication of corrosion or other damages. The welds at the box corners were examined and were found to be in good condition.

Dimensional profiles were made of all four sides of the fuel-element box to determine the extent of distortion. Width and thickness measurements were made at both ends of the element box adjacent to the end pieces. These data are being evaluated. After this work was completed, the end pieces were cut from the fuel-element box, exposing the ends of the fuel plates.

K-5 and K-6

The variations in radioactivity along the length of the fuel-element box were measured with the aid of a gamma-ray spectrometer. The "gamma scans" indicated that the peak burnup occurred slightly below the middle of the element. Plate-spacing measurements were completed. Only minor changes in coolant-channel widths were noted. The fuel-element box was then disassembled to permit close examination of the individual plates.

Detailed Plate Examination

All 16 plates were covered with a thin, strongly adhering layer of tar. The tar surface was covered with what appeared to be small bubbles. These were located over the fueled area of the plates. The plates appeared to be in good condition generally. Three plates, one from either side and one from the middle of the assembly, were selected for close examination with the stereomacroscope. The plates were found to be in good condition but several pitted or eroded areas were noted. It is believed that these defects may have occurred during fabrication of the fuel plates.

The three plates selected for stereomacroscopic examination were gamma scanned transversely and longitudinally, and the results are now being evaluated. During September it is planned to measure the tar deposit and thickness of the three fuel plates selected for close examination. Specimens for radiochemical burnup analysis will be punched from selected areas of the three plates and the results compared with the gamma-activity scans.

OMR Fuel and Structural Materials

R. J. Burian and J. E. Whitney

It is planned to initiate the examination of the structural materials irradiated in a dummy OMRE fuel element during September.

L-1

L. STUDIES OF SODIUM-TANTALUM COMPATIBILITY AT ELEVATED TEMPERATURES

J. H. Stang

As indicated in BMI-1280, emphasis in studies conducted for Los Alamos Scientific Laboratory is being diverted to a program having the objective of developing core-construction materials for LAMPRE service. The only work during July in the previous LASL programs (tantalum-sodium compatibility, high-temperature mechanical properties of tantalum, and weldability of tantalum) was the completion of 1200 F inert-atmosphere creep tests with thermally degassed fine-grained tantalum.

In setting the stage for the materials-development program, attention was given during July to formulating procedures that minimize the possibility for contamination during arc melting of tantalum and tantalum-tungsten specimens. Results of the exploratory work completed are encouraging in this respect.

The new materials-development program begins next month. This new program also provides for a study of tantalum specimens which will be irradiated and an investigation of the effects of tungsten buildup (by transmutation of tantalum) on the mechanical properties.

High-Temperature Mechanical Properties of Tantalum

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

Final effort in this program has been concerned with creep tests at 1200 F in helium on 30-mil strip specimens of thermally degassed fine-grained sintered tantalum. The final data are given in Table L-1 and show that the creep strength of this material is considerably higher than that of annealed sintered tantalum. For example, at a stress of 12,000 psi the minimum creep rate for thermally degassed fine-grained sintered tantalum was only about 0.00001 per cent per hr; the corresponding creep rate for annealed sintered tantalum was about 0.00024 per cent per hr. It might also be noted that little, if any difference was found between the creep rates of fine-grained thermally degassed sintered tantalum and annealed arc-cast tantalum.

In the preliminary phase of the program directed toward the development of container materials for LAMPRE applications, several 100-g buttons and 150-g rods of tantalum and tantalum-3 and -6 w/o tungsten alloys were produced by arc melting in a helium atmosphere. Sheet stock of Fansteel arc-cast tantalum and tungsten were used in melting the unalloyed tantalum specimens, and the tantalum-tungsten specimens. Electron-beam-melted tantalum supplied by Temescal was also used in the preparation of unalloyed specimens. The specimens have been used to evaluate arc-melting techniques on the basis of purity, hardness, and microstructure. In addition, cold rolling and swaging behavior have been studied.

L-2

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

Specimen	Stress, psi	Time in Progress, hr	Initial Deformation, per cent	Total Deformation, per cent	Minimum Creep Rate, per cent per hr
21-B1	10,000	960.7	0.051	0.090	Nil
25-4	12,000	1200.0	0.012	0.098	0.00001
25-3	14,000	1032.3	0.058	0.158	0.000025
21-B2	16,000	1001.8	0.12	0.201	0.00004

Results to date indicate that arc melting does not significantly affect the purity of either the Fansteel material or the high-purity Temescal material. For example, it was found that the hardness of the Temescal material remained about the same (92 VHN) after arc melting a button specimen. Thus, it appears that high-purity alloys of tantalum can be produced by arc melting, provided that the melting stock is of high purity.

Microscopic examination revealed that the buttons and rods are internally sound. The microstructure of the tantalum-tungsten alloys consisted of very large grains of a homogeneous solid solution with uniformly distributed spherical particles of unidentified contaminants. No difficulty was encountered in cold rolling the tantalum-tungsten alloys without a lubricant; however, it was necessary to use kerosene as a lubricant to swage the rod specimens.

Studies will be continued on arc-melting techniques; an investigation will be made to establish a procedure for arc melting high-purity Temescal tantalum buttons weighing up to about 300 g. Additional studies will be made to develop a cold-rolling procedure which will avoid contamination of strip specimens.

M-1

M. DEVELOPMENTAL STUDIES FOR THE PWR

R. W. Dayton

Mixing studies in a model of the PWR core have been resumed. Some improvements were found from the use of deflector vanes over each inlet.

Work with the panoramic camera was completed with the taking of a color photograph which distinguished between dents and corrosion.

Reactor Flow Studies

L. J. Flanigan and H. R. Hazard

Flow studies using air in a quarter-scale model of the PWR are being conducted to determine the effects of lower-plenum geometry on mixing and on flow distribution in Core 2. Previously reported work includes completion of flow studies made with the 7.5-ft core design with and without lower-plenum mixing devices, and preparation of a report covering this work.

Early in August additional studies of lower-plenum mixing, and studies of upper-plenum mixing were begun. Included were exploratory studies of upper-plenum mixing to be made with the Core 1 configuration.

Upper-plenum mixing was studied with the 7.5-ft core design with swirl vanes for three different loop configurations. Measurements were made to determine the distribution of fluid from the traced inlet in the operating outlets. For operation with four loops with Inlet 2-1 traced, the distribution was: Outlet 2-1, 34 per cent; Outlet 1-4, 27 per cent; Outlet 4-3, 21 per cent; Outlet 3-2, 18 per cent. For perfect mixing a value of 25 per cent would be obtained for each outlet. The amount of fluid from the traced inlet found in any outlet is affected by the mixing occurring in the lower plenum.

To improve lower-plenum mixing over that obtained with the 30-deg swirl vanes attached to the flow baffle, deflector vanes were placed over each inlet. These rotated the flow in the outer thermal-shield passage, thus introducing shield flow from the traced inlet into the periphery of the core in an area of low concentration. Four deflector configurations were studied to determine the effect of vane angle and location on mixing and pressure loss. With one configuration some improvement in mixing was obtained with no increase in pressure loss.

In preparation for enlarging the holes in the flow baffle to study performance with reduced lower-plenum pressure loss, an aluminum core extension was added to simulate the 7.5-ft core. This reduced the space between the flow baffle and the core bottom from 5.9 to 3.3 in. This change had no effect on flow distribution in the core, but it was found that mixing between the inlets and the bottom of the core was reduced somewhat.

In September, the effect of flow-baffle hole size will be studied and upper-plenum-mixing studies will be made for the Core 1 design.

M-2

Improved Techniques for Corrosion Detection in Subassemblies and Clusters

G. G. Cocks and C. M. Schwartz

This work is concerned with the interpretation of photographs of fuel-plate assemblies taken with the panoramic camera. During this report period photographs were taken of a specimen furnished by Bettis. These photographs were taken using color film and the flat 45-deg probe mirror. The sample was one which had been dented and subsequently corroded. The dent, which is quite large, was easily visible and easily distinguishable from corrosion marks.

This completes the experimental work on the present contract.

N-1

N. DEVELOPMENTS FOR THE MGCR

W. H. Goldthwaite

This research is a part of the Maritime Gas Cooled Reactor program. There is interest in a carbon dioxide-cooled graphite-moderated concept with gas temperatures and pressures up to 1500 F and 2000 psi, respectively. The graphite will probably have to be clad to prevent CO_2-graphite reactions at these temperatures. A program is under way at Battelle to investigate the effects of radiation on the reactions of the cladding with the CO_2 and the graphite.

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

J. C. Smith, R. H. Barnes, W. S. Diethorn, J. D. Bray, and W. E. Murr

Two specimens each of graphite clad with Type 310 stainless steel, Type 446 stainless steel, and Inconel were irradiated in the Battelle Research Reactor. The specimens are 1 by 1-cm cylinders of AGOT graphite sealed in tightly fitting containers of the cladding materials of interest. Duplicate out-of-pile experiments conducted elsewhere will provide control data for a determination of the effects of radiation.

Irradiation Experiment

The irradiation was performed in a stainless steel capsule containing electric heaters. The CO_2 and specimens were exposed to an estimated gamma and fast neutron dose rate of 5×10^7 and 3×10^7 rads per hr, respectively. The gas temperature at the specimens averaged 1300 F for 4 weeks followed by 1520 F for 1 week. Average gas pressures at these temperatures were 940 and 1020 psi, respectively.

Several samples of the gas were taken and analyzed during the irradiation. After each sample was taken, the capsule was refilled with fresh gas taken directly from a tank of welding-grade CO_2. The important details of the irradiation are presented in Table N-1.

At an average temperature of 1300 F, after an initial rise in CO, consecutive gas samples showed a decreasing CO content. When the temperature was raised to 1500 F, the CO content rose and then dropped again. The reported oxygen contents are all roughly the same as that for the CO_2 tank supply.

Thermodynamic calculations of the CO_2-CO-oxygen system indicate that temperature alone would not be responsible for the CO concentrations that have been observed. The experimental data suggest either that radiation decomposes CO_2 into CO and oxygen and the oxygen oxidizes the cladding, leaving a residual CO content, or that the thermal reaction between CO_2 and the cladding yields the oxidized metal and CO. However, the results do not indicate which mechanism is dominant. The out-of-pile control experiments may help to clarify this point.

TABLE N-1. IRRADIATION HISTORY AND RESULTS OF GAS ANALYSES FOR EACH CONSECUTIVE GAS LOADING

Gas-Loading Designation	Irradiation History: Total Time[a], hr	Temperature, F	Time at Temperature[b], hr	Pressure, psi	Analysis of Gas Loading[c], volume per cent: CO	Oxygen[d]
B (tank analysis)	--	--	--	--	Nil	0.024
Initial purge	(15 sec)	--	--	--	--	0.06
1	29-1/2	250	27-1/2	Est. 900		
		1500	2	Est. 900	--	0.04
2	31-1/2	1500	7-1/2	900		
		1350	24	900	3.6 ± 0.2	0.05
3	72-1/2	1400	72-1/2	870	3.1 ± 0.2	0.04
4	149	1350	55	930		
		275	13	700		
		1260	25	900		
		275	4	700		
		1250	64	900	1.1 ± 0.2	0.04
5	220	1275	215	850	0.4 ± 0.1	0.07
		275	5	850		
6	69-1/2	1260	67	900		
	1/2 (0.1 megawatt)	<200	51	700		
		1410	1	930		
		1500	2	980	0.45 ± 0.15	0.04
7	99-1/2	1560	21	1000		
		1520	93	900	1.9 ± 0.2	0.07
8	52	1500	52	1000	0.6 ± 0.2	0.06

(a) Irradiation at 1 megawatt unless otherwise noted.
(b) In order of occurrence, includes reactor downtime.
(c) Only a portion of total volume analyzed was in irradiation field; hence, results are meaningful only in comparison with other results.
(d) Absolute accuracy of measurement approximately ± 50 per cent.

Examination of Specimens

The capsule was removed from the Battelle Research Reactor to the Battelle Hot-Cell Facility and was opened. The specimens appear to be in good condition. Although there were no gross distortions of the capsule or specimens, the surfaces of the specimens are no longer bright and there has definitely been some surface reaction.

A detailed examination of the specimens, including the making of photomacrographs and the measurement of weights and densities, will be made next month. The specimens will be sectioned and the microstructure will be studied to determine the extent and nature of the CO_2-cladding reactions and cladding-graphite reactions.

O-1

O. DEVELOPMENTS FOR NMSR

A. W. Hare and R. F. Dickerson

This research is part of The Babcock & Wilcox Company Nuclear Merchant Ship Reactor development. Current work is directed toward the development of fuel-pin fabrication techniques and the design, fabrication, loading, and subsequent shipment of irradiation capsules to the MTR. The irradiation capsules will be used both to test prototype fuel pins to 125 per cent design burnup and to test pins in which central melting has occurred.

Fabrication of Urania Fuel Pellets for Loop-Test Studies

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

Work on the preparation of slightly enriched UO_2 specimens for this program has been temporarily recessed, pending receipt of additional UO_2 powder.

Fabrication of Loop-Test Fuel Pins

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

The program concerned with testing prototype UO_2 fuel pins under anticipated NMSR reactor conditions is being continued.

As discussed in BMI-1280, all the short pins for the in-pile and out-of-pile loadings in the high-temperature high-pressure irradiation-test loop were assembled. Final examination of these pins was completed in August, and the pins were then shipped to the MTR.

An investigation was initiated to determine a method of satisfactorily welding Type 304 stainless steel tubing containing 300 ppm boron to Type 304 stainless steel end caps. In addition, an attempt was made to identify the deleterious phase or phases in the weld and base materials. Metallographic examination of defective welds indicated the existence of a second phase (possibly a boride) in the cladding heat-affected zone. Near the high-temperature edge of the heat-affected zone, a eutecticlike phase was observed at the grain boundaries. X-ray diffraction studies of these phases are at this time inconclusive.

Preliminary work was begun on an evaluation of the soundness of welds in seven specimens that were successfully closed by welding at both ends. After being tested and examined, the specimens were cycled between 1000 C and room temperature for 25 cycles. Upon completion of the thermal cycles, the specimens were examined and tested for defects and cracks in the weld material. The welds were leakproof and free of macrocracks. The welds were examined metallographically for microcracks and

other defects and were found to be satisfactory. Thus the thermal cycling does not seem to have had any undesirable effect on the welded sections.

During August, six long-term burnup specimens and two manufacturing control specimens were fabricated to be used in the capsule-irradiation program. These specimens were manufactured by the same techniques which were applied to the loop specimens. Three of the six specimens were prepared by arc welding Type 304 stainless steel end caps to Type 304 stainless steel tubing. The other three specimens were prepared by arc welding Type 304 stainless steel end caps to Type 304 stainless steel tubing containing 300 ppm boron. The tubing containing boron was welded to the stainless steel end caps by a welding procedure which eliminated the pressure inside the tubing during welding and thus prevented the formation of blow holes in the weld metal.

These specimens were examined for defects and failures in the weld and found to be satisfactory. Upon completion of the examinations, the six specimens were forwarded to the MTR for irradiation.

Fuel-Capsule-Irradiation Program

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

Calculations for capsule design, as well as the fabrication of some capsules, were achieved in August. Two of the six irradiation capsules intended for a long-term burnup study of the stainless steel-clad UO_2 fuel were sent to the MTR for irradiation. Three more capsules in the program will be sent to the MTR in September in addition to a thermocouple-implemented capsule.

The capsules are made of stainless steel and use NaK as a heat-transfer agent. Each capsule is provided with dosimeter wires both externally and internally for the entire length of the specimen. The implemented capsule has four internal and two external thermocouples. It is anticipated that the capsules will be subjected to the required thermal-neutron flux of 1×10^{14} nv in order to achieve the desired experimental conditions of temperature, specific fission-heat generation, and burnup rate.

The completion of the sixth capsule during the month of September will complete the construction of the long-term burnup capsules for the NMSR in-pile-capsule program.

Also, during September, work on six capsules containing a specimen each for specimen central-melting studies will be continued.

RWD:CRT/all