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PROGRESS RELATING TO CIVILIAN
APPLICATIONS DURING MAY 1958

By
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June 1, 1958

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DURING MAY, 1958

by

Russell W. Dayton
Clyde R. Tipton, Jr.

June 1, 1958

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REPORTS RELATING TO CIVILIAN APPLICATIONS
ISSUED DURING MAY, 1958

BMI-1262 "Progress Relating to Civilian Applications During April, 1958", by
Russell W. Dayton and Clyde R. Tipton, Jr.

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A-1

A. DEVELOPMENTS FOR ZIRCONIUM-CLAD FUEL ELEMENTS

F. R. Shober

Calibration of the equipment designed to measure the thermal conductivity of UO_2 has been completed using a fused-quartz specimen. Measurements to approximately 834 F show good agreement with reported values. Thermal-conductivity measurements of unirradiated UO_2 have been started.

The creep and rupture strengths of 15 per cent cold-worked Zircaloy 2 are being determined at 290, 345, and 400 C. At 345 C, 30,000 psi caused failure in 4205 hr with an extended third-stage-creep period of approximately 3000 hr; tests at 15,000 and 20,000 psi indicate a minimum creep rate of <0.00001 per cent per hr after 4000 hr.

Corrosion testing of high-strength zirconium alloys at 300 C is in progress. Zirconium-2 w/o tin alloys containing small additions of molybdenum and niobium show corrosion comparable to Zircaloy 2 after 4 weeks on test. The alloying addition of molybdenum and niobium to the zirconium-2 w/o tin base alloy does not appear to change the hot hardness significantly over the 200 to 700 C temperature range.

Thermal Conductivity of Uranium and UO_2

H. W. Deem and C. F. Lucks

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

Uranium

Measurements are now being made on one of the unirradiated clad Zircaloy 2 specimens and should be concluded soon. Other specimens are being prepared and measurements will be continued.

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 thermal gradients over measured lengths. Guarding to prevent stray heat flows has been provided.

A-2

Calibration of the apparatus with fused quartz has been completed, and measurements to 834 F show good agreement with values reported in the literature; the observed points lie within ± 10 per cent of the reported values. Above 900 F, radiant heat transfer becomes significant in quartz and makes it unreliable as a standard above this temperature.

Measurements have begun on UO_2 Specimen 70. Dimensional and weight measurements made at Battelle prior to the conductivity measurements showed values different from those reported by a cooperating laboratory. The specimen gained 0.9596 in weight and the diameter and length increased 0.003 and 0.012 in., respectively. This is a 3.9 per cent increase in weight and a 2.8 per cent increase in volume from measurements made at the site where the specimen was fabricated. The density of Specimen 70 is 10.46 g per cm^3 . The specimen was stored in the shipping container prior to the measurements.

Table A-1 gives the measured thermal conductivity at observed mean temperatures for Specimen 70. These values are somewhat lower than were expected for a specimen of this density. The measurements were discontinued so that the specimen could be examined. Examination after the run showed no significant changes in weight and dimensions.

TABLE A-1. OBSERVED THERMAL-CONDUCTIVITY VERSUS TEMPERATURE
FOR UO_2 SPECIMEN 70

Temperature, F	Thermal Conductivity, $\text{Btu}/(\text{hr})(\text{ft})(\text{F})$	Order in Which Points Determined
198	2.3 ₉	3
216	2.5 ₅	3
343	1.8 ₉	2
369	2.0 ₅	1
399	2.0 ₃	2
423	2.0 ₃	1
535	2.2 ₂	4
640	2.0 ₀	4

Thermal-conductivity measurements on UO_2 will continue. Specimen 70 will be examined further for changes.

Mechanical Properties of Zirconium Alloys

F. R. Shober and J. A. VanECHO

The creep and stress-rupture properties of 15 per cent cold-worked Zircaloy 2 are being investigated over the 290 to 400 C temperature range. The deformation associated with the beginning of third-stage creep is of importance for the design of reactor components operating in this temperature range. A comparison is being made between deformations at the beginning of third-stage creep in short-time tests and tests

A-3

involving failure after times up to 10,000 hr. Long-time tests will also provide data on the metallurgical stability of 15 per cent cold-worked Zircaloy 2 under load at elevated temperatures.

Three additional short-time stress-rupture tests were completed at 400 C. A specimen on test at 345 C and 30,000 psi failed in 4205 hr, after exhibiting an increasing creep rate for approximately 3000 hr. Tests at 15,000 and 20,000 psi at 345 C after approximately 4000 hr have a creep rate of <0.00001 per cent per hr. This rate is comparable to the rate obtained in tests at 20,000 and 25,000 psi at 290 C. An increase of 55 C lowers the creep strength from approximately 25,000 to 20,000 psi. It appears that an increase in temperature to 400 C will decrease the stress required to produce a creep rate of <0.00001 per cent per hr perhaps to less than 10,000 psi.

The tests at lower stresses will be continued to 10,000 hr if fracture does not occur. Additional short-time rupture tests will be made at 400 C.

Development of High-Strength Corrosion-Resistant Zirconium Alloys

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

Improved reactor technology is closely related to the metallurgy of metals applicable for reactor construction. In the case of a reactor fueled with natural uranium, it is necessary that the amount of parasitic material in a core-cladding system be kept to a minimum. Therefore, cladding materials containing less parasitic material and strong enough to allow reductions in the thickness of the cladding would be advantageous. The development of zirconium alloys which would have twice the strength of Zircaloy 2 and good corrosion life in high-purity water at 300 C would allow such a reduction. Alloys developed should have nuclear properties, thermal conductivity, and an expansion coefficient similar to Zircaloy 2. Previous work has shown that additions of tin, molybdenum, and niobium (less than 5.0 w/o) strengthen zirconium with only slightly adverse effects on the corrosion life in high-temperature water.

The alloys being examined consist of ternary zirconium-base alloys containing 2, 3, and 4 w/o tin plus 0 to 2 w/o molybdenum, ternaries containing 2, 3, and 4 w/o tin plus 0 to 3 w/o niobium, and quaternary alloys containing 2, 3, and 4 w/o tin plus 0.5 to 2 w/o molybdenum and 1 to 3 w/o niobium. These alloys are being screened by corrosion testing in high-purity water at 300 C for 1000 hr and by room-temperature hardness measurements. Parameters of a maximum weight gain of 34 mg per dm^2 under test conditions and a minimum room-temperature hardness of 250 DPH were selected for the initial screening process. Further screening will be made by examination of hot-hardness measurements to 800 C on alloys containing the maximum and minimum quantity of alloying elements in the systems being screened. A number of the more attractive alloys which fulfill the screening requirements will be selected for further physical- and mechanical-properties studies.

All of the alloys have been arc melted, cast into 1 by 1 by 1/4-in. bars, rolled at 850 C from a helium-atmosphere furnace to 0.070-in. sheet, annealed at 700 C for 4 hr, and furnace cooled. The alloys containing 2 and 3 w/o tin were reduced 15 per cent

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per pass with no evidence of cracking. The 4 w/o tin alloys showed slight signs of edge cracking. Smaller reductions (10 per cent) per pass eliminated further edge cracking. Three corrosion specimens, 1 by 3/4 by 0.070 in., were sheared from each alloy. Specimens were prepared for corrosion by shaping 2 to 3 mils from each surface and then pickling 1 to 2 mils from the surface (45 HNO₃-5 HF-50 H₂O₃ in volume per cent). All of the corrosion specimens are currently on test. Four-week corrosion data are available on the majority of the 2 w/o tin alloys. In general, the corrosion data are favorable: most of the alloys have not exceeded the 34 mg per dm² maximum corrosion limit. Corrosion data on eight alloys which have shown good corrosion rates plus adequate hardness are given in Table A-2. The corrosion rates for these alloys, with the exception of the zirconium-2 w/o tin-2 w/o molybdenum-2 w/o niobium alloy, are quite good.

TABLE A-2. CORROSION OF ZIRCONIUM ALLOYS

Alloy Composition (Balance Zirconium), w/o	Weight Gain After 672-Hr Exposure, mg per dm ²	Corrosion-Film Color	Remarks
-2Sn-0.5Mo	18	Shiny black	No evidence of flaking
	16	Shiny black	No evidence of flaking
	17	Shiny black	No evidence of flaking
-2Sn-0.5Mo-1.0Nb	19	Shiny black	No evidence of flaking
	21	Shiny black	No evidence of flaking
	23	Shiny black	No evidence of flaking
-2Sn-1.0Mo-2.0Nb	22	Slightly milky	No evidence of flaking
	22	Slightly milky	No evidence of flaking
	21	Slightly milky	No evidence of flaking
-2Sn-1.0Mo-2.0Nb-0.1Fe-0.05Ni	21	Black	No evidence of flaking
	20	Black	No evidence of flaking
	17	Black	No evidence of flaking
-2Sn-1.0Mo-3.0Nb	16	Slightly milky	No evidence of flaking
	21	Slightly milky	No evidence of flaking
	18	Slightly milky	No evidence of flaking
-2Sn-2.0Mo-2.0Nb	12	Milky	Slightly flaking
	38	Milky	No flaking
	37	Milky	No flaking
-1.5Sn	17	Shiny black	No evidence of flaking
	14	Shiny black	No evidence of flaking
	12	Shiny black	No evidence of flaking
-1.5Sn-0.1Fe-0.05Ni	15	Shiny black	No evidence of flaking
	14	Shiny black	No evidence of flaking
	20	Shiny black	No evidence of flaking

Room-temperature hardness measurements have been completed on all of the alloys. All of the alloys containing 3 w/o and 4 w/o tin have hardness readings above the minimum parameter (250 DPH), these alloys have hardnesses between 280 and 330. Hot-hardness measurements for the six alloys which showed the most favorable corrosion

A-5 and A-6

rates have been completed. Alloying additions of molybdenum and niobium to the zirconium-2 w/o tin base alloy do not appear to change the hot hardness significantly.

Further evaluation of these alloy systems will depend upon 6-week corrosion data. Corrosion data for the 3 and 4 w/o tin alloys will be available in June.

B-1

B. DEVELOPMENTS FOR ALUMINUM-CLAD FUEL ELEMENTS

R. J. Carlson

Recent studies of centrifugal-casting techniques for aluminum-uranium have indicated that mold designs which promote end-to-end solidification of the billet produce a product with less shrink porosity and a better interior surface than designs which promote radial solidification. Two separate designs are being investigated.

Optimum time-temperature-pressure relationships have been determined for hydrostatic pressure bonding of nickel-plated uranium slugs in aluminum sheaths. These conditions are 1 hr at 950 F and 10,000 psi. Evaluation has been by metallography and peel tests. Additional specimens will be clad and shipped to the cooperating laboratory for a more complete evaluation.

Corrosion testing has been completed on a series of specimens from a study to develop a natural-uranium fuel alloy containing no more than the cross-section equivalent of 4 w/o zirconium in alloy additions but having better corrosion resistance. Three of the 17 alloys had a corrosion rate about 20 per cent lower than the uranium-2 w/o zirconium control specimen.

Preparation of Aluminum-Uranium Alloys

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

One of the most satisfactory fuels developed for use in low-temperature water-cooled and -moderated reactors are those alloys in the aluminum-uranium system containing up to 20 w/o uranium. However, in order to increase the fuel loading and decrease parasitic neutron capture it is advantageous to increase the uranium concentration. For some applications, aluminum-uranium alloys containing up to 35 w/o uranium are required in the form of tubular fuel elements. Studies at Battelle have indicated that the major difficulties encountered in melting and casting these alloys are porosity and segregation. The porosity is attributed to the evolution of hydrogen and shrinkage during solidification. The segregation is due to the density differences between the phases present and to the large difference between the solidus and liquidus temperatures of the alloys.

Centrifugal-casting studies of air-melted aluminum-25 w/o uranium extrusion billets indicated that porosity caused by shrinkage and the internal surface condition of the billets were of more concern than segregation or gas porosity. It was found that the porosity manifested itself in the form of a band about 1 in. wide in that area of the casting that solidified last. The present program is intended to find methods for reducing or removing this porosity and improving the internal surfaces of the castings. For this purpose, various mold designs and pouring techniques are being investigated.

During May, a series of melts was made, utilizing a steel mold with a graphite liner. Melts were poured at 2200, 2300, and 2400 F with the mold rotating at 550 rpm.

B-2

The mold was preheated to 500 F at the forward end where the melt entered. This series of melts was designed to investigate the possibility of solidifying the cast shape from front to back, instead of radially. Visual examination of these melts revealed no areas of gross porosity. All of the melts exhibited good exterior surfaces; however, the melts poured at 2300 F and less contained folds and cold-shuts on the interior surface, indicating that the molten metal was not sufficiently fluid. The melt poured at 2400 F had a somewhat better interior surface. Sections from these melts will be examined by metallographic and radiographic techniques.

Another series of melts utilizing a steel mold with a variable wall thickness is planned. It is believed that this mold with its higher heat capacity at one end will impart directional solidification to the melt.

Since it has been found that the high-uranium alloys exhibit relatively poor fabrication characteristics, and since it is believed that this is due to the large proportion of UAl_4 particles present in the alloys, a study concerning the suppression of the UAl_3 - UAl_4 reaction is in progress concurrently with the casting program. It is believed that the retention of UAl_3 in an alloy containing silicon may be due to the formation of USi_3 . Both USi_3 and UAl_3 have the same crystal structure and a size factor of 6 per cent is conducive to the formation of a stable solid solution of UAl_3 - USi_3 . Therefore, the majority of the additions now being made to the basic aluminum-35 w/o uranium also form compounds of the USi_3 type. Among these elements are germanium, lead, palladium, platinum, and tin. Though these additions may not be suitable from a neutron-economy viewpoint, an evaluation of their effects upon the alloy may tend to support the hypothesis advanced for the increased fabricability of alloys containing silicon. On the basis of the results obtained, suitable alloy additions can be chosen which may fulfill both requirements: increased fabricability and compatibility with the chemical re-processing operations. The initial melts in this series are being made in the form of 1-in.-diameter by 8-in.-long ingots cast in graphite molds. These melts will be evaluated by metallographic examination, X-ray diffraction, and thermal analysis.

Future work will be concerned primarily with the 35 w/o uranium alloys. Mold designs and pouring techniques will be evaluated in addition to the effect of numerous ternary additions upon the basic aluminum-35 w/o uranium alloy.

Aluminum Cladding

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

The use of pressure bonding, incorporating a helium-gas pressure at elevated temperatures, is being investigated for cladding internally and externally cooled fuel elements, consisting of nickel-plated uranium cores clad with aluminum.

Various pressure-bonding conditions have been employed in an effort to determine the minimum conditions necessary to secure sound metallurgical bonds. During this study, temperatures have been varied between 750 and 950 F, times from 5 min to 1-1/2 hr, and pressures between 5,000 and 10,000 psi. Metallurgical bonds have been

B-3

obtained for all the conditions as determined by metallographic examination. The latest condition, which was 1-1/2 hr at 750 F and 5,000 psi, is being examined metallographically.

The integrity of these metallurgical bonds has been evaluated by the chisel and peel tests. The data obtained for these results revealed that in all cases the nickel plate bonded quite well to the aluminum, but that a uniform, strong uranium-to-nickel bond was only secured while bonding for 1 hr at 950 F and 10,000 psi. Decreasing the bonding time at 950 F and 10,000 psi from 1 hr to 5 min resulted in an average decrease in uranium-to-nickel diffusion of only 0.00003 in., yet the bond produced was not quite as strong and was spotty. A drop in pressure or temperature had a more marked effect on bond integrity, with the bond strength decreasing with decreasing temperature and pressure.

It is quite evident from these results that, although metallurgical bonding is realized with lower temperatures and pressures, 950 F for 1 hr at 10,000 psi is required to obtain a continuous metallurgical bond with good strength properties. The remaining I and E slugs will be pressure bonded using these optimum bonding conditions and shipped to another site for testing.

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

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

Development of a high-uranium fuel alloy that has improved corrosion resistance in 300 C water is being undertaken. Design requirements are that this alloy must possess corrosion resistance better than that of a uranium-2 w/o zirconium alloy, but have a thermal-neutron cross section that is no greater than that of a uranium-4 w/o zirconium alloy.

Seventeen alloy compositions are being investigated by means of metallography and corrosion tests. Results of the first corrosion test of all 17 alloys, heat treated 1 hr at 900 C and furnace cooled, are presented in Table B-1.

The conditions of the test were as follows. The specimens, 1/4 by 1 by 0.06 in., were wet ground on 240-grit paper and suspended in an autoclave. The autoclave was sealed, then flushed with argon and evacuated several times. Heating to 300 C took place in 2 hr, after which preheated water was admitted into the autoclave. The specimens were corrosion tested for 15 min at 300 ± 3 C, after which the pressure was released and steam was bled from the system. Cold argon was admitted to permit rapid cooling of the specimens. When the autoclave reached 204 C it was water quenched.

The test results show that the uranium-2 w/o zirconium-0.05 w/o platinum, uranium-2 w/o zirconium-0.16 w/o ruthenium, and uranium-2 w/o zirconium-0.46 w/o aluminum alloys performed the best. A decrease of about 20 per cent in corrosion rate over the uranium-2 w/o zirconium alloy was observed.

B-4

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

Sample	Alloy Composition (Balance Uranium), w/o	Weight Loss ^(a) , mg/(cm ²)(hr)	Penetration, mils per hr
1	2 Zr	1700	40
2	4 Zr	2080	54
3	5 Zr	2040	56
4	2 Zr-0.04 V	2100	52
5	2 Zr-0.33 Nb	1710	44
6	2 Zr-0.047 Ni	1690	44
7	2 Zr-0.03 Ti	1760	44
8	2 Zr-0.14 Mo	2250	52
9	2 Zr-0.05 Pt	1360	30
10	2 Zr-0.07 Cr	1760	38
11	2 Zr-0.015 V-0.15 Si-0.01 Mo	2230	52
12	0.015 V-0.15 Si-0.01 Mo	2640	62
13	2 Zr-0.46 Al	1400	30
14	1 Zr-0.5 Nb	1950	46
15	2 Zr-0.15 Nb-0.05 Mo	3110	54
16	2 Zr-0.16 Ru	1360	28
17	0.04 Cr-0.04 V-0.1 Nb-0.2 Zr	2240	50

(a) Based on the original area.

Similar tests are to be performed on these alloys after heat treatment designed to improve radiation stability and also after a heat treatment intended to duplicate the diffusion anneal which the eventual fuel elements are to receive.

C-1 and C-2

C. PLANT ASSISTANCE TO MCW

D. A. Vaughan

The scope of this program has been changed from refinery problems to metal-fabrication problems. A mathematical model is being developed to relate, quantitatively, the orientation of crystallites in uranium to the intensity of X-ray diffraction lines, as an aid in the fabrication of various core shapes to meet anticipated specifications.

Orientation in Uranium

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

A study of the various X-ray diffraction techniques for analyzing the orientation of crystallites in uranium indicates that the number of measurements required would be too time consuming and the data are not sufficiently quantitative for production control. If the diffraction-line intensity data can be related, quantitatively, to the amount of preferred orientation, it is possible that fewer measurements would provide the information required. An initial attempt to develop a mathematical model which would quantify the degree of orientation in terms of the measured X-ray diffraction-line intensities is being based upon the methods employed for quantitative analysis of phases.

D-1

D. PROCESSING OF FEED MATERIALS

E. L. Foster, Jr.

A continuing interest in the improvement of unalloyed uranium fuel-slug material has been responsible for two lines of inquiry presently being followed. One program is concerned with the quality of induction-melted uranium specifically as it pertains to impurity content and the factors that play a role in their occurrence. The second program is concerned with the theoretical considerations in the solidification of uranium castings. In addition to the contributions that these programs will make to present uranium-casting technology, the solidification study will permit evaluation of melting and casting variables for future casting needs.

Solidification of UraniumE. L. Foster, C. K. Franklin, B. L. Fletcher,
B. Schwartz, and R. F. Dickerson

This program deals with the phenomena associated with the solidification of uranium castings. Pouring temperatures, pouring rates, mold design, mold materials, and various other factors will be studied. In addition, castings will be made with thermocouples strategically placed in the metal and mold to obtain a record of the temperature as the metal freezes. The data will be used to evaluate the relative importance of the above-mentioned variables upon the internal soundness of the ingot.

Investigations were first directed toward finding a suitable method for describing the heat-flow patterns existing in the casting. A mathematical model consisting of cellular divisions was devised. The cells represent parts of a pie section of the ingot and mold materials. By studying the transfer of heat between the cells, a stepwise process of analysis was devised by which the flow of heat within the ingot and from the ingot to the mold could be followed. In an application of this model, the movement of the solidification front in the casting would be described by the movement of the solidus temperature through the system.

Current activity has involved the preparation of preliminary solutions of the expanded 72-cell mathematical model with the IBM 650 electronic computer. These computer runs have been made using the best available thermal- and physical-property data for production-size ingots. The results are being analyzed and their significance evaluated. This will conclude the preliminary computations for the production-size ingot.

Since accurate theoretical analysis requires accurate physical constants of materials, physical-property measurements of uranium and graphite are being made. The total normal emissivity of oxidized uranium has been determined at temperatures from 1600 F to near the melting point. The values increased slightly with increased temperatures from 0.53 to 0.54. Emissivity determinations have been made on graphite specimens coated with MgO mold wash between 1500 and 3000 F. Thick-walled tubular

D-2

specimens of graphite were used for this test and the observed values are being corrected for temperature gradients existing in the material. Measurements of the thermal conductivity and linear thermal expansion of the materials will be reported as they are obtained.

Examination of Factors Affecting the Quality of
Induction-Melted Uranium

R. W. Endebrook, E. L. Foster, and R. F. Dickerson

This program concerns the various chemical reactions that may occur during the vacuum-induction melting of uranium. The reactions of specific interest are those which can vary considerably from charge to charge in such a way as to be detrimental to the cast ingot. By obtaining a basic knowledge of these reactions, a means for their control may be suggested, and greater uniformity of the uranium ingots should result.

During May, the experimental portion of the program was completed and mass-spectrometer analyses of gas samples taken during the tests were reported. The final series of melts was made by admitting to the evacuated furnace significant amounts each of the gases hydrogen, nitrogen, CO, CO₂, O₂, and H₂O so as to obtain a record of furnace pressure versus time over uranium held at a temperature of 2500 F. The metal, contained in uncoated graphite crucibles, was initially blanketed under gas pressures of approximately 550 μ . Gas analyses were omitted in the initial profiles, but were obtained at the apparent reaction end point in two succeeding replicate tests. These replicate tests were run under as nearly identical conditions as possible.

Although the original plan of these experiments was to obtain data regarding equilibrium gas concentrations over uranium, theoretical thermodynamics of the reactions involved indicated that at equilibrium at 2500 F these gases would not be expected to be present over uranium in detectable amounts. Of considerably greater importance, however, was the indicated mode of reaction and relative reactivity of each gas with the uranium-graphite system.

In those tests in which oxygen was added, a crust immediately formed over the metal. The reaction was rapid as manifested by a low initial furnace pressure (about 200 μ) and a rapid drop in pressure. Analyses of furnace gas 3 min after the oxygen addition showed that oxygen reacted simultaneously with the metal and the graphite crucible, since a large percentage of CO was found. In like manner, nitrogen formed a crust over the melt. This gas also continued to react, and an analysis of furnace gas sampled 33 min after the nitrogen admission showed that the nitrogen content had fallen to roughly 3.5 volume per cent. Similarly, CO and CO₂ reacted with uranium to form a crust of oxide and carbide over the melt, but these reactions appeared to be less rapid than those of oxygen and nitrogen with the metal-graphite system. A large percentage of CO was found in the furnace gas 35 min after either CO or CO₂ had been admitted. Percentages varied from 53 to 77 volume per cent CO. Pouring the metal in this furnace atmosphere resulted in an abrupt drop in furnace pressure, indicating loss of the protective action of the oxide-carbide crust. Water vapor reacted quite rapidly and completely, and the reaction gas was found to be predominantly hydrogen with only small

D-3 and D-4

amounts of CO (about 3 volume per cent). In those tests in which hydrogen was added, except for a slight initial drop in furnace pressure (about 50 μ), hydrogen appeared to be nonreactive in the uranium-graphite system. It is suspected that the initial pressure drop might be the result of some hydrogen solution by the metal. In all of the above cases, reproducibility of data evidently depended on the ability to reproduce identical surface conditions on the melt. In general, both pressure data and gas analyses were in fair agreement in the replicated tests.

Perhaps the most important outcome of this final series of experiments is an indication of a surface-protection effect of dross stemming from the reaction of these various gases with the surface of the uranium. These effects are summarized below:

- (1) The cover formed over the melt by the reaction of either CO or CO₂ offered good protection against further reaction, as evidenced by a markedly decreased reaction rate after an initial period and the high residual CO gas over the melt.
- (2) The cover formed when oxygen was the reactant gas offered definite protection against residual CO, CO₂, and oxygen, although a large amount of oxygen had to be reacted before a protective cover was obtained.
- (3) The covers produced by the reaction of either water vapor or nitrogen offered little protection, as indicated by the completeness of these reactions and the ultimate high-hydrogen furnace atmosphere.

These effects imply that the carbides produced in surface reactions with uranium form protective covers (even in the oxygen experiments); while oxides and nitrides, by themselves, do not. This implication points to the possibility of using high-integrity protective covers or slags during the melting cycle as a means of controlling ingot quality.

Evaluation of the data obtained up to the present time is in progress.

E-1 and E-2

E. GENERAL FUEL-ELEMENT DEVELOPMENT

F. A. Rough and D. C. Carmichael

A fuel-element development program supported by the AEC Division of Reactor Development is reported in this section.

A study is being made of the feasibility of a fueled zirconium hydride moderator. Pressure-composition-temperature studies and high-temperature structural studies are continuing.

Fueled Zirconium Hydride Moderator

H. E. Bigony and H. H. Krause

The hydrides of uranium-zirconium alloys are being investigated as fueled moderators for potential use in gas-cooled reactors. Efforts are being directed toward preparation of a zirconium-25 w/o uranium alloy for phase studies and alloys containing 1 and 50 w/o uranium for structural studies.

Structure and Pressure-Composition-Temperature Studies

Samples of a zirconium-25 w/o uranium alloy of the required purity for pressure-composition-temperature studies have been prepared. Hydrogen-absorption isotherms will be run on these samples to get data for completion of the phase diagram of the system. New Vycor reaction tubes have been tested and installed in the vacuum line used for the hydrogen-absorption work. The preparation of alloy hydrides containing 1 and 50 w/o uranium for high temperature X-ray investigation is continuing.

F-1

F. STUDIES OF URANIUM AND URANIUM-ALLOY FUELS

F. A. Rough and A. W. Hare

The research and development of uranium-alloy fuels reported in this section are part of the AEC Reactor Development Division's program. Being concluded are the studies concerned with the radiation stability of uranium-zirconium alloys, the development of gamma-phase uranium alloys, and the mechanism of aqueous corrosion. The research concerned with the preparation of uranium-niobium alloys is continuing.

In the study of the mechanism of uranium corrosion in boiling water, uncorroded metal particles in the oxide scale have been observed that affect the apparent corrosion-product balance.

Radiation Stability of Uranium-Zirconium AlloysD. J. Hammon, A. W. Hare, A. A. Bauer,
and R. F. Dickerson

Irradiation Capsules BMI 8-5 and BMI 8-6 were discharged from the MTR during the Cycle 104 shutdown. Preliminary calculations indicate a total burnup of 2.25 a/o for the two specimens of zirconium-50 w/o uranium and 1.79 a/o for the three specimens of zirconium-22 w/o uranium. The capsules will be returned to the Battelle hot-cell facility; however, postirradiation examination of these specimens will be delayed.

Development of Gamma-Phase Uranium AlloysV. W. Storhok, A. A. Bauer,
and R. F. Dickerson

In a program aimed at developing a metastable gamma-phase uranium alloy, ternary and quaternary alloys of uranium with chromium, molybdenum, niobium, ruthenium, vanadium, and zirconium are being investigated.

Transformation-kinetics studies of uranium-10 and -12 w/o molybdenum-base alloys indicate that 1 w/o additions of niobium improve gamma stability. However, increasing niobium to 3 or 5 w/o results in progressively more rapid transformation. A uranium-12 w/o molybdenum-2 w/o ruthenium alloy also shows improved gamma stability. Quaternary alloys with 1 to 2 w/o ruthenium additions and 2 to 3 w/o niobium additions were prepared to study whether a combination of the two elements would have additional gamma-stabilizing effects. This, however, was not the case, as uranium-10 and -12 w/o molybdenum-1 w/o ruthenium-2 w/o niobium alloys, while being superior to the binary alloys, were poorer than the ternary alloys containing 1 w/o niobium or 2 w/o ruthenium. Increasing the ruthenium and niobium contents to 2 and 3 w/o, respectively, resulted in still poorer gamma stability.

Hot-hardness data indicate uranium-10 and -12 w/o molybdenum-1 w/o niobium alloys are slightly harder at all temperatures up to 900 C than are similar alloys with 3 or 5 w/o niobium. Also, a uranium-10 w/o molybdenum-1 w/o ruthenium-2 w/o niobium alloy exhibited a higher hardness and a higher softening temperature than a uranium-10 w/o molybdenum-2 w/o ruthenium-3 w/o niobium alloy. For comparison, diamond-pyramid hardness numbers of 120 and 84.8 were obtained for the latter two alloys, respectively, at 600 C. At 900 C, hardness numbers of 14.9 and 10.6 were noted.

Corrosion testing of gamma-quenched alloys in 680 F water is continuing. After 1515 hr of test, a uranium-20 w/o niobium alloy is corroding at a rate of 0.032 mg/(cm²)(hr), a uranium-20 w/o niobium-5 w/o zirconium alloy at 0.020 mg/(cm²)(hr), and a uranium-7.5 w/o molybdenum-2 w/o ruthenium alloy at 0.083 mg/(cm²)(hr). All other alloys either have failed or have been removed from test because of heavy attack.

Experimental work is nearly complete and a topical report is being prepared.

Preparation of Uranium-Niobium Alloys

J. B. Melehan, E. L. Foster,
and R. F. Dickerson

This research program is one phase of a study of the possible application of uranium-niobium alloys in fuel-elements. The program involves the melting and fabrication of alloys of this system and determination of various physical, mechanical, and chemical properties which are significant in reactor metallurgy.

Several experiments have been conducted in inert-electrode skull melting and casting of uranium-20 w/o, -40 w/o, and -80 w/o niobium alloys. The 20 w/o niobium alloy was melted and cast into a cylindrical graphite mold about 1 in. long and 3/8 in. in diameter. Surface irregularities on this ingot indicate that the pouring temperature was not quite high enough, causing an irregular flow of metal into the mold. A barely perceptible variation in radiograph density is suggestive of some sort of vertical inhomogeneity. This slight segregation could probably be eliminated by increasing the pouring temperature. Melting of the 40 and 80 w/o niobium alloys must await modification of equipment to either increase power capacity or decrease the loss of heat by conduction or radiation.

Additional experiments have been conducted in the fabrication of consumable-electrode arc-melted castings of the three alloys. Disk-shaped specimens 1/2-in. thick by 2 in. in diameter were machined from 2-1/4-in. -diameter ingots. The 20 w/o niobium disks were sealed in stainless steel and rolled at 650 C. After negligible reduction, the specimens fractured into several segments transverse to the rolling direction. The 40 and 80 w/o niobium alloys were cold press forged using a press load of 300 tons. Only minor reductions were possible on the 40 w/o niobium alloy but the 80 w/o niobium alloy was worked to a final diameter of more than 2-1/2 in. Further efforts to cold roll or warm hammer forge these specimens after removal of the cladding resulted

F-3 and F-4

in severe radial cracking. Two additional cylindrical specimens of the 20 w/o niobium alloy 3/4 in. in diameter by 1 in. long were sealed in stainless steel. One was initially upset forged and rolled at 700 C to a thickness of 0.150 in. before the capsule cracked. It was then removed from the capsule and rolled out of a helium atmosphere at 700 C to a final thickness of 22 mils. The second encapsulated cylinder was successfully reduced by standard forging at 700 C to a final thickness of 90 mils.

Homogenization heat treatments of segregated ingot material, reported in BMI-1262, have been continued with heat treatments of the 40 w/o niobium alloy at 1575 C for 4 hr and of the 80 w/o niobium alloy at 1950 C for 1/2 hr. Radiographs after heat treatment show that banding persists; these results in addition to those reported in BMI-1262 indicate that, in order to materially decrease the segregation, heat treatments must be carried out for periods longer than 8 hr or at higher temperatures. The solidus temperature limits further experiments of this type.

An oxidation-testing program of limited scope is under way. Interrupted-exposure tests in air were carried out with measurement of weight gains after 24-hr exposure periods. An initial exposure at 400 C was followed by similar exposures at 500 and 600 C. Results are tabulated below. The 20 and 40 w/o niobium alloys were completely reacted to oxides and only a small metallic fragment of the 80 w/o niobium specimen remained after the 600 C exposure. A second specimen of the 80 w/o niobium alloy still on test had, after 114 hr at 400 C in air, a total weight gain of only 8 mg.

Niobium Content (Balance Uranium), w/o	Specimen	Initial Weight, g	Initial Area, cm	Weight After 24-Hr Exposure at Indicated Temperature, g		
				400 C	500 C	600 C
20	U-20-01	16.3090	10.0967	16.3740	17.0523	19.4273
40	U-40-01	17.7163	10.7213	17.7733	18.8183	22.8619
80	U-80-01	8.4745	7.4275	8.4769	8.5682	10.8939

Mechanism of Aqueous Corrosion

E. Adelson, C. M. Schwartz, D. A. Vaughan,
O. M. Stewart, W. E. Berry, P. D. Miller,
and F. W. Fink

Studies of the water and steam corrosion of uranium, which were directed toward understanding the lack of material balance of the corrosion products, have been concluded. In experiments in boiling water, it appears that the inequality between the gaseous product, hydrogen, and the weight loss of the specimen is due to an incorrect assumption in regard to the amount of metal oxidized. It was found that uncorroded metal particles were present in the oxide scale. This results in a lower apparent hydrogen evolution and a higher apparent specimen weight loss than would be observed if the metal corroded uniformly. Experimental work during May was limited to phase studies on the residue obtained in the dissolution of the solid corrosion products in dilute nitric acid solution. Several faint X-ray diffraction lines, not part of the pattern of UO₂ or metallic uranium, were observed but not identified.

G-1 and G-2

G. FATIGUE STUDIES OF INCONEL AND INOR-8

W. S. Hyler and F. H. Lyon

Fatigue Studies of Inconel

This program has the objectives of obtaining basic fatigue information on Inconel and of establishing quantitative relationships among the variables of temperature, stress, strain, time, and cyclic frequency for Inconel.

The current phase of this program is concerned with the measuring and recording of strain associated with the cyclic portion of a combined load. During May, fabrication of the specimens and the extensometer system was completed. In addition, modification of the fatigue machine was completed.

During June, it is expected that calibration and checking of equipment will be completed. Preliminary tests will be made to evaluate the experimental setup.

Fatigue Studies of INOR-8

A program to evaluate the fatigue properties of the INOR-8 alloy is under way. The planned modification of fatigue machines and furnaces to accommodate temperatures up to about 1500 F has been completed. Fatigue specimens have been heat treated and machined, and are now being final polished prior to fatigue testing.

During June, it is expected that initial studies of the alloy at a temperature of 1250 F will be made.

H. REACTOR MATERIALS AND COMPONENTS

F. A. Rough and A. W. Hare

Progress on parts of the AEC Division of Reactor Development Program is reported in this section. Routine progress is reported for the programs concerned with oxidation-resistant niobium alloys, valence effects of oxide solutions in uranium dioxide, high-temperature high-pressure solid-state studies, rubbing surfaces in sodium environment, and basic studies of pressure bonding.

An investigation is being conducted to study the effect of surface preparation on the quality of the bonds obtained during gas-pressure bonding of Zircaloy 2 to Zircaloy 2. Excellent bonds have been obtained using surfaces which were machine belt abraded. Various other preparations are in the process of being evaluated.

Oxidation-Resistant Niobium Alloys

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

This is a study of the oxidation behavior of unalloyed niobium and of niobium alloys with the objective of correlating the oxidation behavior of the metal with the effects of alloying on a fundamental basis. Current work includes oxidation testing of niobium ternary alloys, X-ray diffraction examination of scales from previous alloys, calculations to correlate oxidation weight gains with decreases in core thicknesses, and preparation of additional material for study.

Oxidation tests have been completed on two ternary alloys, with results as given in Table H-1. The niobium-10.5 a/o chromium-7.5 a/o molybdenum alloy (as-cast) had good oxidation resistance, both at 1000 and 1200 C. At 1000 C, the scale was protective and a parabolic relation between scale growth and time was observed for time periods up to at least 6 hr. At 1200 C, linear behavior was observed after 10 to 15 min, indicating that the outer scale had become porous and nonprotective. The weight gains at the conclusion of both tests (6 hr) were 20 per cent lower than recorded for niobium-25 a/o titanium, one of the most oxidation-resistant binary alloys. Comparison of the behavior of this alloy with that of niobium-5.5 a/o molybdenum-8 a/o vanadium, reported in BMI-1262, shows that elements which improve oxidation behavior through a "size effect" in Nb_2O_5 (chromium, molybdenum, vanadium) are additive in improving oxidation resistance, and each appears to have little effect on solubility limits of the others in Nb_2O_5 in ternary alloys with niobium.

The addition of 0.5 a/o chromium to a niobium-7.5 a/o titanium base effected a significant improvement in the oxidation resistance. The oxidation rate was equivalent to that of a binary alloy containing 15 to 20 a/o titanium, but the new alloy still oxidized faster than the better ternary alloys.

In BMI-1262, it was pointed out that material-balance calculations on the niobium-zirconium alloys indicated an appreciable fraction of the oxidation weight gain was associated with subscale formation. These calculations have been extended to unalloyed niobium, and niobium-chromium, -iron, -molybdenum, -titanium, -tungsten, and -vanadium alloys. These additional calculations indicate that no more than qualitative

TABLE H-1. OXIDATION DATA FOR NIOBIUM AND NIOBIUM ALLOYS IN DRY AIR

Sample	Composition(a)	Parabolic	Transition to Linear Rate, hr	Linear Rate Constant, mg/(cm ²)(hr)	Weight Gain at Indicated Exposure, mg per cm ²					
	(Balance Niobium), a/o	Rate Constant, (mg/cm ²) ² per hr								
					1 Hr	2 Hr	3 Hr	4 Hr	5 Hr	6 Hr
<u>1000 C</u>										
O-30	100Nb ^(b)	100	0.1	30.0	30.0	61.3	89.8	114.2	136.9	158.6
O-37	25Ti ^(b)	54	2.2	2.56	7.4	10.5	13.1	15.7	18.2	20.8
O-135	10.5Cr-7.5Mo	28.5	>6	--	5.1	7.4	9.2	10.7	12.0	13.0
O-137	7.5Ti-0.5Cr	50	0.25	(5) ^(c)	10.7	16.3	21.0	25.7	30.6	35.7
<u>1200 C</u>										
O-77	100Nb ^(b)	--	<0.1	(93)	107.7	--	--	--	--	--
O-82	25Ti ^(b)	193	0.45	10.6	15.8	26.8	37.4	47.4	57.0	67.0
O-136	10.5Cr-7.5Mo	47.6	0.2	8.1	11.2	19.2	27.6	35.7	43.4	50.8
O-138	7.5Ti-0.5Cr	--	<0.1	(20)	33.3	53.6	69.4	--	--	--

(a) Ternary compositions were estimated to nearest 0.5 a/o on basis of weight-loss data.

(b) Data from previous tests are included for comparison.

(c) Rates in parentheses are estimated.

agreement can be expected between weight gains calculated from metal-loss data and from weight-gain data. Best agreement (± 20 per cent) was found for alloys which formed protective scales, e. g., niobium-molybdenum alloys at 1000 C, but in many cases the calculated weight gains were as much as 50 per cent lower. Thus, the conclusion relating to the niobium-zirconium alloys must be regarded as qualitative only.

The results of X-ray diffraction examinations on scales from three previously oxidized alloys are given in Table H-2. The inner portion of the niobium-10 a/o tungsten scale contained metallic tungsten, indicating preferential oxidation of niobium. The oxygen pressure at the metal-oxide interface is evidently on the order of 10^{-12} to 10^{-14} atm, since WO_2 is reduced to metallic tungsten at 10^{-12} atm, while Nb_2O_5 is reduced to NbO_2 at 10^{-14} atm. Several extra lines in the diffraction pattern of this scale may be due to a complex oxide. Some solubility of iron in Nb_2O_5 is indicated from examination of the niobium-5 a/o iron scale, since the M-type Nb_2O_5 was stabilized in the temperature range where the H-type is normally stable. No effects of rhenium were found in the scale from the niobium-5 a/o rhenium alloy. Both the niobium-iron and niobium-rhenium scale exhibited preferred orientation of the inner black scale, as found on pure niobium.

Ingots of niobium-15 a/o molybdenum and niobium-6 and -12.5 a/o chromium were cast and shaped to provide test samples. The niobium-15 a/o molybdenum cracked during shaping. Completion of tests on the two chromium alloys will conclude experimental work on this program.

TABLE H-2. IDENTIFICATION OF OXIDES ON NIOBIUM ALLOYS

Alloy Composition, a/o	Oxidation Conditions		Oxide Phases Present		Orientation of Inner Scale
	Time, hr	Temperature, C	Inner Scale	Outer Scale	
Nb-10W	6	1200	N- Nb_2O_5 +X ^(a) +W	H- Nb_2O_5 +X ^(a)	Random
Nb-5Fe	4	1000	M- Nb_2O_5	M- Nb_2O_5	Preferred
Nb-5Re	1	1000	H- Nb_2O_5	H- Nb_2O_5	Preferred

(a) Several extra lines appeared in the oxide pattern which may be due to a complex niobium-tungsten-oxygen phase.

Valence Effects of Oxide Solutions in Uranium Dioxide

W. B. Wilson and C. M. Schwartz

An investigation of the effects of cation valence of solute additions upon the stability of uranium dioxide is in progress. Studies of the physical processes involved in oxidation and vaporization of uranium oxide containing La_2O_3 and ThO_2 were made during May. Bodies of uranium oxide with 60 w/o La_2O_3 and with 50 w/o ThO_2 were tested 107 hr in air at 2500 F. The La_2O_3 additive body remained sound and indicated a slight increase in weight associated with oxidation. The ThO_2 body powdered and indicated a loss in weight. Structural studies and chemical analyses of the materials were continued.

High-Temperature High-Pressure Solid-State Studies

W. B. Wilson and C. M. Schwartz

The effects of pressure and temperature on uranium oxide containing various oxide additions are being investigated. Divalent, trivalent, and tetravalent additives are being or will be studied at a pressure in excess of 30,000 atm at several temperatures. In addition to the reaction studies, work is also continuing to find a suitable method of recording temperature by use of a thermocouple in the high-pressure region.

Rubbing Surfaces in Sodium Environments

J. W. Kissel and J. H. Stang

Dry-friction and sodium-lubricated tests have been completed using a molybdenum-molybdenum rubbing combination. The moving (about 0.1 mm per sec) element was a polished flat plate while the stationary element was a 1/4-in. -diameter polished ball resting on the plate with a calculated average contact pressure of 83,000 psi. The general findings from these runs are summarized in Table H-3. Noteworthy points are as follows:

- (1) The highest coefficients of friction obtained were those for dry sliding. This is different behavior than found in prior experiments for a cermet (WC-20 w/o cobalt) sliding against itself; as reported in BMI-1262, this latter combination exhibited somewhat higher friction with sodium present than without.

Although the molybdenum specimens were carefully cleaned prior to the exposures, it is suspected that residual films were present on the surfaces. One reason for believing this is that slip-stick behavior, which is characteristic of metal-metal sliding, was not apparent during the dry-friction runs at low temperature (although stick-slip was observed repeatedly after sodium was introduced). The beginning stages of breakdown of such films may have been responsible for the erratic friction behavior above 900 F. For example, above this temperature, the value for kinetic friction coefficient alternated between 1.6 and 0.8 during a single traverse.

- (2) Friction in the 250 to 525 F range, with sodium present but with no observed wetting tendency, was quite low. It is not understood why the mere presence of the sodium should result in this substantial reduction in friction. It is not likely, according to thermodynamic considerations, that low-temperature chemical reactions could occur and yield surface films having lubricating qualities.
- (3) Friction increased markedly after the wetting of specimen surfaces had occurred; however, from this point on to high temperatures, there was little sensitivity to ambient-temperature variation. The fact that friction increases as wetting ensues is also difficult to explain, although it seems likely that some kind of surface-chemistry mechanism is responsible.

TABLE H-3. SUMMARY OF INFORMATION FROM SLIDING-SURFACE EXPERIMENTS WITH THE MOLYBDENUM-MOLYBDENUM COMBINATION

The Runs Listed are in Chronological Order

Surface Condition	Temperature, F	Average Coefficient of Friction (Kinetic)	Comments
Dry	80 to 1300	0.3 to 1.6	Approximate linear increase in friction with increasing temperature; however, above 900 F, friction was erratic
Sodium present	250 to 525	0.09 to 0.05	Uniformly low friction
Sodium present (wetting initiated) ^(a)	525 (maintained overnight)	0.5 to 0.2	Overnight exposure initiated wetting and resulted in increased friction
Sodium present (surfaces wetted)	600 to 1300 ^(b)	0.2 to 0.4	Friction remained relatively constant as temperature increased
Dry (sodium evaporated) ^(c)	100 to 600	0.7 to 0.2	Friction decreased slightly with increasing temperature
Dry (sodium evaporated) ^(c)	600 to 1200	0.2 to 0.4	Friction was not markedly changed by increasing temperature

- (a) Visual observations of wetting behavior were confirmed by measurement of electrical-contact resistance. Prior to wetting, the resistances were relatively high; as wetting occurred, the resistances were markedly lowered. However, following the evaporation of sodium at the high temperatures, high resistances again prevailed.
- (b) The sodium began to evaporate at about 900 F, and at 1300 F was completely removed from the surfaces.
- (c) Several more descending- and ascending-temperature cycles were made with dry (sodium evaporated) surfaces; the friction results were not significantly different from those indicated.

- (4) Evaporation of the sodium, which must have left a residue of sodium oxide on the surfaces, had little effect on friction in the 600 to 1200 F range. Below this, however, there was a reversal of the usual form, i. e., friction increased with decreasing temperature.

Following the tests, the flat specimen was removed from its holder and carefully examined. Wear tracks displaying varying degrees of metal damage were present for each of the tests. The most pronounced wear tracks were produced by dry sliding; tracks produced at high temperatures were, in all cases, more prominent than those produced at lower temperatures. Tiny, white, variously shaped particles of debris were evident in those tracks produced while sodium was present. The disposition of these particles suggested that they were actually formed during the sliding process. Their composition is unknown, although they were found to be soluble in alcohol and water wash. Highly magnified views of the central portions of pronounced wear tracks (areas of greatest stress concentration) often showed transverse, parallel, curved, surface cracks, suggestive of the brittle rupture of a work-hardened surface or, possibly, the fracture of a surface layer.

Basic Studies of Pressure Bonding

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

An investigation is in progress with the purpose of analyzing the pressure-bonding process from the standpoint of the creep properties of the material involved. It is expected that the principles developed here will have wide application as a means of estimating the time, temperature, and pressure conditions under which two surfaces of a given material will undergo the necessary deformations to provide the sufficiently close contact needed for bonding.

Analytical Studies of Pressure Bonding

In this investigation deformations are being analyzed using a simple power law for creep. The creep parameters are obtained by analysis of the deformations under external hydrostatic pressure of thick-walled tubes. As a representative material 2S aluminum is being used. Because of wide discrepancies in the experimental results it is thought that a more readily controlled test procedure should be developed.

The power law for creep can be used to relate the angular velocity and torque for a torsion test. Consideration is being given to the idea of performing several torsion tests in hopes of obtaining the creep parameters in a more expeditious manner.

In order to apply the creep law to the case of surfaces being bonded, it is necessary to make assumptions concerning the nature of the voids between the surfaces. It is felt that during the process of bringing the surfaces to temperature and pressure the voids will assume a shape which is more or less spherical. The collapse of these spherical voids due to creep can be expressed in terms of the power law, using the creep parameters obtained from the most reliable source. When the diameters of the voids have been reduced to a certain size, which is yet undetermined, it can be said that the surfaces have achieved complete contact. The predicted deformations will be compared with the values measured in the tests on the flat-plate specimens described below.

Experimental Studies of Pressure Bonding

In the experimental phase of this program, specimens are being prepared to obtain data which can be compared with calculated values in order to determine the validity of using postulated mathematical models to predict the flow characteristics of metals during pressure bonding. Evacuated and sealed thick-walled tubular aluminum specimens are externally pressurized at different temperatures to determine if the amount of deformation produced with respect to time can be previously predicted by applying the theory of primary creep to the pressure-bonding processes. For each set of conditions, duplicate specimens are used in order to insure reliability of results.

A series of tubular specimens has been subjected to pressure-bonding parameters of 6000 psi at 700 F for periods of 1/2, 1, 2, 4, and 6 hr. Radiographic and metallographic examinations of the specimens indicated that complete closure of the holes was achieved for all of the time parameters at 6000 psi and 700 F. In order to compare the extent of deformation achieved at two pressure levels, a series of tubular specimens is now in the process of being subjected to 3000 psi at 700 F for periods of 2, 4, and 6 hr. The results for these specimens will be compared with previous data obtained at 4000 psi and 700 F.

In an effort to predict the extent of deformation between mating surfaces during a pressure-bonding process from the results obtained by the analytical studies on tubular specimens, two sets of flat-plate aluminum specimens have been designed and machined to finishes of 250 and 750 rms, respectively. Two evacuated and sealed composite flat-plate specimens consisting of each rms finish have been externally pressurized at 4000 psi at 700 F for zero time in order to determine the extent of an initial hole size. Metallographic studies of these specimens are in progress. Additional flat-plate specimens with 250 and 750 rms finishes are now in the process of being subjected to 4000 psi at 700 F for periods of 2, 4, and 6 hr as prescribed by the analytical studies.

Effect of Surface Preparation on Bonds Obtained During Pressure Bonding of Zircaloy 2

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

A minimum amount of deformation occurs between mating surfaces during a hydrostatic pressure-bonding process; consequently, it is believed that the bond quality of Zircaloy 2-to-Zircaloy 2 bonds is dependent upon the prior surface condition of the materials. This investigation is intended to study the effect of surface preparation of Zircaloy 2 on the quality of the bonds obtained during pressure bonding. Surface preparation and bonding variables, such as the cold-worked condition of the metal, surface contamination, surface roughness, and recrystallization and grain growth, which appear to have an influence on the integrity of the bond obtained during pressure bonding will be studied to determine their effect on the bonding process.

Hot-rolled and annealed Zircaloy 2 plate material has been selected as the base material to be used in this investigation. After annealing, the majority of the plates have been belt abraded on the bonding surfaces prior to subsequent surface treatment. For the study of the effect of surface roughness on bonding, a few of the plates have been sheared to specimen size and have been individually machined to different surface finishes.

In this investigation, the specimens will be contained in evacuated and sealed Ti-Namel cans, and the cans will be subjected to pressure-bonding parameters of 10,000 psi of helium gas at 1550 F for a period of 4 hr. To insure reliability of results, each can will contain a Zircaloy 2 reference specimen prepared with belt-abraded surfaces.

The Zircaloy 2 reference surface-conditioned specimens, which have belt-abraded surfaces, have been pressure bonded. Preliminary metallographic examination of these specimens as viewed under the light microscope indicated that excellent bonding was achieved along the original interface, except for a few minute areas which contained pits or contamination particles. Under polarized light, complete grain growth across the original interface was observed.

Duplicate multispecimen cans containing surface-machined specimens ranging from 50 to 1000-rms finishes have also been pressure bonded. Sections from each specimen have been submitted for metallographic preparation, and evaluations of the bonds obtained are nearly completed.

Specimens for the study of the effect of cold work on bonding have been prepared and are now in the process of being pressure bonded. The specimen material was prepared by cold rolling several annealed and belt-abraded Zircaloy 2 plates to reductions of between 2 and 67 per cent. In order to minimize or prevent the contamination of the surfaces for the subsequent bonding studies, the plates were degreased and washed before the cold-rolling operation. An attempt was made to avoid contamination from the rolls by cold rolling an experimental plate between protective plates of Zircaloy 2, but it was found that this method of rolling was unsatisfactory. Therefore, the plates were rolled without the use of protective plates. However, during the cold-rolling operation for each plate, the rolls were washed several times with acetone. After cold rolling, a section of each plate was belt abraded, and the plates in the two conditions -- as rolled, and rolled and abraded -- were sheared to specimen size.

Surface layers of carbon, chromium, iron, niobium, tin, titanium, and zirconium oxide have been applied to Zircaloy 2 specimens and are now in the process of being assembled and pressure bonded to study their effect on bonding and to observe the movements of these layers during the bonding process. In addition, specimens to be used to study the effect of pickling Zircaloy 2 surfaces prior to bonding are being prepared.

I-1

I. PHYSICAL RESEARCH

F. A. Rough

This research is part of the program of the Metallurgy and Ceramics Section of the AEC Division of Research.

Several projects were reported last month; progress on the balance is reported this month.

The study of bonding fundamentals is essentially complete, and present effort is on completion of the analysis of data.

Additional analyses of the niobium-hydrogen system and its kinetics have been made. Kinetic data are now available from hydrogen-to-niobium ratios of 0.05 to 0.70 and equilibrium data from hydrogen-to-niobium ratios of 0.01 to 0.85 at pressures of 0.1 to 1000 mm of mercury at 100 to 900 C.

Experimental and theoretical study of the mechanism of hydrogen migration in zirconium hydride has continued. Diffusion coefficients needed for the theoretical analysis are being obtained.

Study of Bonding Fundamentals

F. C. Holden, H. R. Ogden, and R. I. Jaffee

During this report period efforts have been directed toward the analysis of the experimental solid-state bonding data.

Calculations on the influence of grain-boundary diffusion on bond growth of single asperities have been made. The presence of the initial bond interface provides an effective grain boundary for the diffusion of vacancies out of the bond junction; thus, the effective diffusivity of the system is increased above the level predicted by lattice diffusion alone. It is believed that much of the observed scatter in bond-strength values can be attributed to the random orientations between the crystal lattices of the asperity and the flat. If this variance is taken into account, calculated and experimental data are in good agreement.

A first draft of the final report is being prepared in the form of a technical paper.

Niobium-Gas Reactions

W. M. Albrecht and W. D. Goode

A study of the kinetics and mechanisms of the reaction of hydrogen with niobium is being made. Kinetic data have been reported for the formation of solution of

hydrogen in niobium for hydrogen-to-niobium ratios in the range 0.05 to 0.70. Also, diffusion coefficients obtained from degassing rates have been reported over the range 450 to 650 C.

A low-pressure equilibria study in the range 0.1 to 100 mm of mercury pressure has been concluded. The results have been combined with those previously reported for the niobium-hydrogen system to give comprehensive coverage of the ranges 100 to 900 C, 0.1 to 1000 mm of mercury pressure, and 0.01 to 0.85 hydrogen-to-niobium ratios. Figure I-1 is a logarithmic plot of equilibrium pressure against composition for various temperatures.

An elevated temperature X-ray study of the system has been completed. Results obtained with specimens containing 0.097, 0.24, and 0.54 hydrogen-to-niobium ratios at room temperature to 400 C have outlined the boundary of a two-phase region. The data are shown in the phase diagram presented in Figure I-2. Several isobars for the system are also shown. From these data the upper limit of the two-phase region was determined to be at about 150 C and at an equilibrium pressure less than 0.1 mm of mercury.

The basis for a mathematical interpretation of the equilibria in the niobium-hydrogen system is the consideration of the formation of various products (solid solutions) having definite hydrogen-to-niobium ratios. Various isopleths were obtained by plotting the logarithm of equilibrium pressure against reciprocal temperature. As previously reported, a series of straight lines are obtained which follow the general equation,

$$\log_{10} P_{\text{mm}} = -A/T(K) + B .$$

The values obtained for the Constants A and B in this equation and the heats of solution for the various compositions are given in Table I-1. It is seen that there is a general increase in the heat of solution with hydrogen composition up to a 0.4 hydrogen-to-niobium ratio. At higher compositions there appears to be very little, if any, change in the heat of solution.

TABLE I-1. HEATS OF SOLUTION FOR THE NIOBIUM-HYDROGEN SYSTEM

Hydrogen-to-Niobium Ratio	Constants for log $P = A/T + B$		Heat of Solution, cal per mole
	A	B	
0.01	-3480	4.83	15,900 ± 200
0.02	-3590	5.49	16,400 ± 130
0.05	-3570	6.17	16,400 ± 110
0.10	-3690	6.80	16,900 ± 120
0.20	-3950	7.55	18,090 ± 42
0.30	-4150	8.03	18,990 ± 47
0.40	-4300	8.40	19,700 ± 120
0.50	-4420	8.77	20,200 ± 140
0.60	-4320	8.90	19,800 ± 290
0.70	-4180	9.30	19,100 ± 1900

FIGURE I-1. LOGARITHMIC PLOT OF ISOTHERMS IN THE NIOBIUM-HYDROGEN SYSTEM

FIGURE I-2. PHASE DIAGRAM FOR THE NIOBIUM-HYDROGEN SYSTEM SHOWING REPRESENTATIVE ISOBARS.

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Migration of Hydrogen in Zirconium Hydride

W. M. Albrecht, J. J. Ward, and H. H. Krause

The experimental portion of the program to study the migration of hydrogen in zirconium and zirconium hydride has consisted of the determination of isothermal diffusion coefficients for hydrogen. The results to date are summarized in this subsection.

A theoretical analysis of the factors affecting the diffusion of gases in metals has been continued, but no further effort on this program is scheduled for the remainder of Fiscal Year 1958.

Diffusion Coefficients

The diffusion coefficients for hydrogen in the delta zirconium hydride were determined using a moving-boundary technique as described by Jost.* In this technique the rate of movement of the hydride-metal boundary into the hydrogen-saturated core is measured. The movement of the boundary at a given temperature is described by the equation,

$$\xi = 2 \gamma \sqrt{Dt} , \quad (I-1)$$

where

ξ = displacement of the boundary

D = diffusion coefficient

t = time

γ = dimensionless parameter depending upon the distribution of hydrogen in the system.

The parameter γ is obtained from the relationship,

$$(C_s - C_{II,I}) / (C_{II,I} - C_0) = \sqrt{\pi} \gamma e^{\gamma^2} \operatorname{erf}(\gamma) . \quad (I-2)$$

For the zirconium-hydrogen system, C_s is the concentration of hydrogen at the surface of the specimen, $C_{II,I}$ is the concentration at the alpha plus delta/delta boundary, and C_0 is the initial average hydrogen concentration.

*Jost, W., Diffusion in Solids, Liquids, and Gases, Academic Press Inc., New York (1952).

Experimental rates of diffusion of hydrogen in delta zirconium hydride were determined in the range 200 to 700 C. The temperature dependence of the diffusion coefficient, D , determined from these data is expressed by the equation,

$$D = 0.40 \exp [(-20,700 \pm 750)/RT] , \quad (I-3)$$

where 20,700 cal/(mole)(C) is the activation energy.

Initial experiments to determine the diffusion coefficient of hydrogen in beta zirconium were made using the concentration-gradient technique.* Attempts were made to produce hydrogen-concentration gradients in beta-phase zirconium cylinders by dissolving thin hydride films for 20 and 30 min at 600 and 700 C, respectively. Analysis of layers machined radially showed that the hydrogen was distributed uniformly throughout the specimen, indicating that the heating times were too long. However, from this observation the diffusion rate for hydrogen in beta zirconium was estimated to be of the order of 10^{-4} cm² per sec at 600 to 700 C, since only about 1 min would be required to produce a suitable concentration gradient. Such time is not experimentally feasible in the gradient technique. The moving-boundary, sorption-rate, and desorption-rate technique were then considered as means to determine the rate of diffusion. Because diffusion is very rapid, the diffusion coefficient could not be determined with any degree of certainty using any of these techniques. Further consideration has indicated that a permeability technique should prove more suitable for determining the high diffusion coefficients that were estimated for hydrogen in beta zirconium.

Theoretical Analysis

Further consideration of the problem of the migration of hydrogen in the zirconium hydride system has been based on a thermodynamic viewpoint. The equation commonly written for thermal diffusion that describes the Soret effect is

$$J_H = -D \frac{\partial C_H}{\partial x} - D' C_H \frac{\partial T}{\partial x} , \quad (I-4)$$

where

- J_H = the hydrogen "current", or flow
- D = the ordinary diffusion constant
- D' = the thermal diffusion constant
- C_H = concentration of hydrogen
- x = distance of the gradient
- T = temperature.

D and D' may be functions of composition and temperature.

*Mallett, M. W., Baroody, E. M., Nelson, H. R., and Papp, C. A., J. Electrochem. Soc., 100, 103-06 (1953).

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At the steady state, J_H is zero and Equation (I-4) becomes

$$-\frac{\partial \ln C_H}{\partial T} = \frac{D'}{D} = S_T ,$$

where S_T is the Soret coefficient.

A viewpoint of the Soret effect developed along thermodynamic lines of reasoning can be given as follows: the thermal gradient term in Equation (I-4) can be expressed in terms of free energy,

$$-D'C_H \frac{\partial T}{\partial x} = -\frac{\partial \bar{F}_H}{\partial x} B_H ,$$

where $\bar{F}_H$ is the partial molar free energy of hydrogen, and whose gradient is the driving force of thermal diffusion. B_H is a mobility factor, and it may be a function of composition, pressure, and temperature. It may be regarded as a resistance term in thermal diffusion.

Since $\bar{F}_H = RT \ln a_H$, where R is the universal gas constant and a_H is the activity of hydrogen in the metal, and $a_H = C_H \gamma_H$, where C_H is the mole fraction concentration of hydrogen, and γ_H is the activity coefficient of hydrogen, it can be shown for an ideal solution that

$$-DC_H \frac{\partial T}{\partial x} = -R(\ln C_H) B_H \frac{\partial T}{\partial x} .$$

For a nonideal solution such as the zirconium hydride-hydrogen system, it can be shown that

$$-DC_H \frac{\partial T}{\partial x} = -B_H \left\{ \left(\frac{\partial \ln a_H}{\partial T} \right) \frac{dT}{dx} + \left(\frac{\partial \ln a_H}{\partial P} \right) \frac{dP}{dx} + R \ln a_H \frac{dT}{dx} \right\} ,$$

where

$$\frac{\partial \ln a_H}{\partial T} = \frac{H_H^\circ - \bar{H}_H}{RT^2} ,$$

where

H_H° = molar heat content of pure hydrogen

$\bar{H}_H$ = partial molar heat content of hydrogen,

and

$$\frac{\partial \ln a_H}{\partial P} = \frac{\bar{V}_H}{RT} ,$$

where

$\bar{V}_H$ = partial molar volume of hydrogen and

$$a_H = C_H \gamma_H.$$

It is seen that the driving force of thermal diffusion may be related to the partial molar thermodynamic properties and the activity coefficient as deviations from ideal solution behavior.

On the basis of this viewpoint, it is recommended that the thermal-diffusion data of the zirconium hydride system be correlated with the thermodynamic properties of the system. Gulbransen and Andrew* have given a treatment of the system which could be extended to give the necessary thermodynamic data for such a correlation.

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 been continued.

Titanium continues to have merit for use as a material of construction for the Darex dissolver and feed-adjustment tank.

The evaluation of Nionel for use in constructing the dissolver vessel for the Sulfex-Thorex process continues to give promising results. Type 329 stainless steel will not withstand the Sulfex solutions. Carpenter 20 Cb suffers severe cracking around surface deformations upon exposure to Sulfex solutions. The several remedies for this, such as heat treatment, cathodic protection, and inhibition by metal ions, are now under study. Other candidate materials of construction are being screened.

INOR-8 had a lower corrosion rate than INOR-1 during their first 500-hr exposure period of immersion in the molten, equimolar NaF-ZrF₄ mixture at 650 C. Much lower potentials were measured between the specimens and containers than on previous runs and crystal deposition did not occur. Vapor-phase specimens have been included during the second 500-hr exposure period for these metals.

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

The flowing dissolver has been kept in operation and, by the end of May, a total of over 1900 hr of exposure had been accumulated in this apparatus. Examination of the specimens is not scheduled until the 2000-hr mark has been reached. Stressed titanium specimens containing transverse weldments have been inserted for evaluation.

FAT Studies With Titanium

No examination of the titanium specimens exposed to boiling solutions representing the initial conditions of the feed-adjustment tank was made during May.

The titanium steam tube exposed to Initial FAT solution and the three tubes exposed to Final FAT solutions were removed, examined, and returned to exposure in fresh solutions. No apparent corrosion was detected.

The crystalline deposit which forms on the liquid-phase portion of the tubes exposed to Final FAT solutions is being analyzed. Preliminary study shows that removal of this deposit may not be difficult.

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 steel-clad fuel elements of throrium 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 $\text{Th}(\text{NO}_3)_4$.

After 20 periods of cycling specimens of Carpenter 20 Cb between Sulfex and Thorex solutions, a microscopic examination of the surfaces of the specimens was made. A large number of transgranular cracks were observed on surfaces which had been heavily deformed by stamping and on sheared edges. These occurred only on specimens exposed in sulfuric acid, either cyclically or alone, and appeared to be more numerous and pronounced in the more concentrated (6 M) acid. A few of the specimens showed signs of cracking along the surface of the material next to the weldments and in the weldments themselves. Intergranular attack was observed along some of the weldments of specimens exposed to the Thorex solutions.

Heat treatment of the specimens for 45 min at 2050 F in a nitrogen atmosphere, followed by water quenching, appeared to completely inhibit any cracking around surface deformations upon subsequent exposure to boiling sulfuric acid solutions. However, purposely deforming the surface following heat treatment resulted in cracking around the deformations upon a similar acid exposure. It appears that heat treating could be used to remedy cracking around surface deformations, but on several of the specimens minute cracks were observed which seemed to be unconnected in any way with prior surface deformation.

Cracks do not develop as readily in 1/8-in.-thick specimens as in the 1/16-in. sheet material used in most of the studies. It has been shown that an addition equivalent to 35 g per liter of dissolved Type 304 L stainless steel reduces the corrosion rate of Carpenter 20 Cb specimens tenfold in boiling 6 M H_2SO_4 and completely eliminates cracking around surface deformations. Presently the lowest concentration of dissolved stainless steel which will inhibit cracking is being determined in the hope that it will be sufficiently low that no passivating effect on the further dissolution of stainless steel would be exerted if it were preadded to the dissolver.

Experiments, uncompleted as yet, seem to be indicating that a beneficial cathodic protection of a Carpenter 20 Cb dissolver against cracking would be afforded by stainless steel-clad elements contacting the vessel walls during dissolution and acting as sacrificial anodes. However, other observations during these experiments lead to the

J-3 and J-4

belief that the vapors evolved during actual dissolution may be much more corrosive to unprotected Carpenter 20 Cb specimens than the vapors above a similar acid solution during a reflux-type corrosion study. Studies of the use of externally impressed potentials are envisioned if Carpenter 20 Cb continues to be favored.

Further scouting-type experiments have continued to illustrate the probable merit of Nionel as a material of construction for the dissolver. This was not true for Type 329 stainless steel, which showed fairly low corrosion rates in Thorex solutions but dissolved rapidly in sulfuric acid.

Several other materials are being evaluated in Sulfex and Thorex solutions by means of scouting experiments. These include the Hastelloys C and F, Incoloy 804, Illium R, and zirconium. Welded specimens of Nionel have been fabricated in preparation for cyclic experiments of the type conducted with Carpenter 20 Cb.

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 molten bath of NaF-ZrF_4 with a stream of HF. A material of construction for this hydrofluorinator is being sought.

Exposure periods of 500 hr have been completed with INOR-1 and INOR-8 specimens immersed in the molten equimolar NaF-ZrF_4 mixture at 650 C in the new Hastelloy B melt containers. The INOR-1 specimen was heavily etched and some intergranular attack may have occurred in various areas. A noticeable metal loss occurred as indicated by grooving of the lower edge at the point where the HF stream impinged upon it. An over-all penetration rate of 2.1 mils per month was calculated from the total weight loss.

The INOR-8 specimen was etched but showed no appearance of intergranular attack. The over-all penetration was calculated to be 1.2 mils per month.

No deposition of metallic crystals occurred on either of these specimens. Potential measurements between the specimens and the Hastelloy B containers were much lower than between similar specimens and Inconel containers at times when crystal deposition did occur. Perhaps the deposition is more a result of galvanic action than mass transfer.

Specimens of the same composition were placed in the vapor phase during the second 500-hr exposure period of these specimens. The second exposure period for INOR-8 has already been completed, uneventfully. Data for both materials will be discussed next month.

The problem of blockage of exhaust tubes and arms of the melt container by "snow" accumulation during the course of the runs seems to have been solved by placement of a baffle a few inches above the surface of the molten salt.

K-1

K. POSTIRRADIATION STUDIES OF SRE,
OMRE, AND OMR FUEL MATERIALS

J. E. Gates

In connection with the SRE fuel program, a six-specimen capsule train, NAA-15-7, has been opened and a postirradiation evaluation initiated. This study will be continued next month.

An irradiated OMRE fuel element will arrive at the Battelle hot-cell facility in July for a postirradiation evaluation. Equipment has been modified and standardized to accommodate this element, and the evaluation should be initiated upon arrival of the element.

A dummy element containing OMR nonfueled corrosion specimens will be available for study early in July. Planning for handling this evaluation is under way. However, no evaluation will be performed during the fiscal year.

SRE Fuel Material

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

The postirradiation evaluation of the NAA-15-7 capsule train is in progress. This train contained six thorium-11 w/o uranium specimens in individual NaK-filled thermocoupled capsules. This train was irradiated in the MTR to an estimated burnup of 1 total a/o and a temperature of approximately 1000 F. These estimates are based on MTR flux data and initial temperature readings. They are subject to revision on completion of radiochemical analyses.

The capsule train has been separated into capsules, three capsules sampled for fission gas, and all capsules opened. The specimens are identified by letters A through F, starting at the bottom of the train.

The visual examination indicated that Specimens B, E, and F were slightly bowed, although all three specimens had a clean surface free of surface defects. Specimen A was badly swollen on the end opposite the thermocouple; however, the remainder of the specimen appeared in good condition. Specimen C could not be removed from the capsule. This specimen had swollen until it had filled the capsule. A stereomacroscopic examination of the end of the specimen stuck in the capsule indicated large voids near the specimen axis. Only a small section of one of the thermal barriers around the specimen was visible.

Approximately one-third of the length of Specimen D on the thermocouple end was in poor condition. The surface was blackened and rough, and small pieces crumbled easily from this region which invalidated any estimate of swelling. The remainder of the specimen appeared clean and bright.

K-2

Dimensional surveys and density measurements will be made on specimens where possible, and selected specimens sectioned for metallography and radiochemical burnup analyses. Analyses will be made to determine the amount of cesium-137 released to the NaK from Specimens A and F.

The second train of capsules containing specimens of a thorium-uranium alloy, NAA-15-6, is expected to arrive at the hot cell for evaluation in June. Postirradiation evaluation will be initiated as soon as possible upon arrival.

OMRE Fuel Elements

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

The effects of radiation and corrosion on a series of OMRE fuel elements will be evaluated. These fuel elements consist of 16 plates of 25 w/o enriched UO_2 dispersed in a matrix of Type 304 stainless steel and clad with Type 304 stainless steel. The plates are assembled inside a box fabricated of stainless steel.

The examination of the elements, after irradiation in the OMRE, will involve inspection for corrosion, measurements of plate spacing and distortion, and a gamma-activity scan along the length of the element and several individual plates. Each element will then be cut apart and each plate examined individually for corrosion and distortion. Specimens for radiochemical burnup analysis will be punched from selected plates. The data obtained from the radiochemical analyses will be correlated to the gamma-scan data to provide information on relative burnup along the length of the element. It is also planned to machine tensile, impact, and bend-test specimens from the fuel plates from selected fuel elements, and to perform subject tests. Stereomacrographs will be made to provide a pictorial record of the condition of the elements.

Several pieces of equipment are now being designed, assembled, or readied for use in the remote examination of the ORE fuel elements. These include a device for measuring plate spacing, milling equipment to cut open the elements, and equipment to be used in unloading the large fuel-element shipping cask. A dummy fuel element has been received to facilitate design work and for use in checking remote-handling procedures.

It is planned to continue the design and assembly of remote equipment during June, 1958. The assembly of the most of this equipment will be completed and a checkout of remote-handling techniques will be initiated.

OMR Fuel and Structural Materials

Examination of irradiated OMR structural materials or fuel elements will not be initiated during Fiscal Year 1958.

L-1

L. STUDIES OF SODIUM-TANTALUM COMPATIBILITY AT ELEVATED TEMPERATURES

J. H. Stang

This section covers investigations of the creep behavior of tantalum in sodium and in an inert atmosphere (helium) and a study of factors concerned with the welding of tantalum. The supporting agency, Los Alamos Scientific Laboratory, is engaged in the LAMPRE development program.

It was reported previously that specimens of arc-cast tantalum were exhibiting higher strength in 1200 F flowing sodium than would be predicted on the basis of available inert-atmosphere data. However, it was recently determined that the specimens being used in the sodium experiments are not fully annealed. Thus, since annealed specimens were used in the inert-atmosphere tests, a direct correlation of data is not permissible.

Thermally degassed tantalum has been rolled into strip and subsequently annealed to provide a fine-grained high-purity material for inert-atmosphere creep experiments at 1200 F. Tests with this material will start early in June.

Evidence that carbon level in commercial tantalum affects weld soundness is mounting. However, difficulties in introducing carbon into tantalum have prevented an evaluation of the welding characteristics of specimens having a precisely known amount of carbon distributed evenly throughout the matrix. The study of grain refinement in tantalum welds by ultrasonic excitation is currently concerned with the application of ultrasonics to weldments held at an elevated temperature. Furnace apparatus to obtain the experimental conditions desired is being constructed. Studies to appraise resistance welding for tantalum tube-to-header joint fabrication have been completed. While the method has been proven feasible for certain applications, it has shortcomings which would render it unsuitable for others.

Tantalum-Sodium Compatibility Studies

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

The effects of sodium exposure on the creep behavior of tantalum are being investigated. Specimens are simultaneously stressed and exposed to 1200 F flowing sodium in polythermal loop systems. Each system is provided with facilities for monitoring creep.

Five experiments with arc-cast tantalum reported in BMI-1262 were continued; specimen loads in these tests ranged from 20,000 to 26,000 psi. As reported in BMI-1262, the creep rates in this test series have been lower than expected on the basis of prior experiments with annealed arc-cast material in an inert atmosphere. For example, the creep rate for a sodium-immersed specimen loaded to 26,000 psi was about 0.0004 per cent per hr (after 787 hr); this is about equivalent to the rate expected in helium with a load in the neighborhood of 20,000 psi.

L-2

At the present time, the difference is attributed to incomplete annealing of the gage sections used for the sodium-exposure specimens; evidence on which this is based was obtained from recent metallographic examinations of a control specimen. This annealing difficulty apparently stemmed from a specimen modification adopted to prevent grip-portion deformation during loading. In the induction-annealing furnace, these grips, which were strengthened by spot welding tantalum strips on each face and, hence, were relatively massive, evidently reached the annealing temperature (about 2700 F) while the relatively slender gage sections did not. As a result of this situation, a pair of inert-atmosphere experiments with specimens from this annealing lot will be conducted to obtain directly comparable information. Such experiments will start during June.

Also, an experiment was started in which the test specimen is being given a 2-week soak in 1200 F sodium prior to application of load. The purpose of this experiment is to obtain an indication of the effect of a reduction in oxygen content in tantalum on its initial in-sodium creep behavior. Prior corrosion-loop experiments have demonstrated that exposure of commercial tantalum to high-purity sodium will effectively lower its oxygen level.

High-Temperature Mechanical Properties of Tantalum

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

Experiments with the recently modified vacuum-induction furnace have provided a sufficient stock of thermally degassed tantalum for cold rolling to strip, machining into creep specimens and recrystallizing by annealing to produce a fine-grained material. Creep tests of this thermally degassed, fine-grained tantalum at 1200 F in a helium atmosphere will be initiated at loads around 10,000 psi. The purpose of these tests is to obtain an indication of the stress necessary to produce a minimum creep rate of 0.00005 per cent per hour.

Creep tests on annealed arc-cast tantalum sheet specimens at 900 and 1500 F have been completed after about 1725 hr of exposure. The results indicate that, at 900 F, creep is negligible at loads of 20,000 and 25,000 psi; loadings of 30,000 and 40,000 psi produced minimum creep rates of about 0.00004 and 0.00048 per cent per hr, respectively. At 1500 F, creep was negligible at loads of 5,000, 6,000, and 8,000 psi, while a loading of 10,000 psi caused a minimum rate of about 0.00003 per cent per hr.

The chemical analysis of a tantalum ingot, produced from capacitor-grade scrap by the electron-beam melting technique has been completed. The interstitial content is as follows:

	<u>PPM</u>
Oxygen	16
Nitrogen	10
Carbon	30
Hydrogen	1.4

The hardness of the ingot, 83 VHN, is approximately in line with these levels.

L-3

Weldability of Tantalum for High-Temperature Systems

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

Studies to date have indicated that the carbon content in tantalum has a direct relationship to the porosity of welds made in commercial tantalum. Efforts to increase the carbon content of an unweldable lot of tantalum by subjecting it to methane gas at high temperatures with subsequent homogenizing treatments at 1000 C have not been successful. Although a gain in weight from carbon was recorded (from 60 to 880 ppm carbon) the resulting welds were porous throughout, a result which indicated that the carbon or carbide had not been diffused by the homogenizing treatment. In an attempt to study the problem further, welds were made on a sample which was subjected to the methane gas atmosphere but not homogenized. The surfaces of these welds were free of porosity indicating that surface carbon was present and had a beneficial effect. Evidently, the material treated at 1000 C lost its surface carbon through reaction with minute traces of oxygen present in the inert-gas blanket.

Because of the difficulty in diffusing carbon into solid tantalum, it was decided to add carbon to unweldable tantalum while in molten state. For this work, two samples with known additions of carbon (100 and 200 ppm) will be arc melted. The arc-melted samples will then be fabricated to sheet for further welding studies.

Studies on the effect of ultrasonics on grain size have shown that vibrations during welding of tantalum have reduced the grain size of the weld. A series of tests was made with a new design of the acoustical horn (ultrasonic source). From these studies, it was found that the amount of grain refinement during welding was dependent on the horn displacement since the greater the horn displacement the finer the grain size. However, the amount of grain refinement was limited by the level of strain which the tantalum specimen could withstand without fracturing in the weld zone. At this limit grains produced in the weld zone are approximately one-third the size found in unexcited tantalum.

A study is in progress to evaluate the effects of grain refinement by applying ultrasonics to weldments which have been heated to approximately 1100 C. If this process is successful, a small assembly can be welded, heated to an elevated temperature, and subjected to ultrasonic vibrations. Such a scheme would be more practical than applying vibrations during welding. At present, a method of heating tantalum weldments to the elevated temperatures is being investigated. Induction heating is being used; however, some difficulty is being encountered in obtaining satisfactory coupling. The major problem is arcing between the coil and specimen as a result of the low-dielectric strength of the inert-gas blanket.

Studies on stored-energy resistance welding have been completed and have shown that two types of joints can be obtained between tantalum tubes and header stock. These are:

- (1) Welds accompanied by upsetting of the tube wall immediately adjacent to the tube-to-header interface
- (2) Welds accompanied by violent expulsion of molten metal from the interface and very little upsetting of the tube wall.

L-4

Both methods have provided leaktight joints as determined by helium leak tests. Tension tests have indicated that joints in which violent expulsion occurred have lower strength than joints in which the tube upset occurred. Strength data obtained from a number of samples are given in Table L-1.

TABLE L-1. STRENGTH DATA FOR RESISTANCE-WELDED TUBE-TO-HEADER JOINTS

Joint Type	Breaking Load, lb	Breaking Strength ^(a) , psi	Number of Samples
1/64 by 3/16-in. tantalum tubing	850	105,000	1
Upset only	225-660	26,700-78,000	11
Expulsion	175-220	20,800-26,000	2

(a) Based on 0.015-in. wall by 0.1875-in. -diameter tantalum tubing.

Hardness surveys indicated that the hardness was higher in joints in which expulsion occurred probably because of contamination of molten tantalum by the atmosphere. Both joining techniques produced shortening of the tubes which would be troublesome if tubes were joined individually in a multiple-tube assembly.

M-1

M. DEVELOPMENTAL STUDIES FOR THE PWR

R. W. Dayton

Studies of mixing in a flow model of the PWR have continued. Improved mixing has been obtained, without large increases of pressure drop, by adjustment of the angle of the swirl vanes in the lower plenum.

The evaluation of the X-ray photometer has been completed, and data obtained on its sensitivity for void and inhomogeneity detection.

Studies of the panoramic camera have been resumed to improve its ability to distinguish between corrosion and other surface imperfections. Apparatus is now being assembled.

Best results are obtained in pressure bonding oxide fuel plates when a pressure of 10,000 psi, a temperature of 1450 to 1500 F, and abraded rather than pickled surfaces are used. Such bonds, after beta treating, have been strong and ductile, and exhibit grain growth across the original interface. Work is continuing on an evaluation of the consistency of the process under the best conditions, using a Neolube coating to prevent a zirconium- UO_2 reaction.

Reactor-Flow Studies

L. J. Flanigan and H. R. Hazard

Air-flow studies in a quarter-scale model of the PWR with Core 2 are being conducted to determine the effect of lower plenum geometry on mixing and flow distribution in the reactor core. Previously reported work includes installation of a perforated flow baffle simulating a 7-1/2-ft core design and studies of mixing with five different lower plenum configurations.

In May, mixing in six additional lower plenum configurations was studied to determine the effect of both thermal-shield flow and angular position of swirl vanes, which extend from the flow baffle to the bottom dome, on mixing results.

Decreasing the vane angle reduced lower plenum pressure drop with a slight decrease in the amount of mixing obtained.

Thermal-shield coolant flow increased the maximum concentration of air from the traced inlet found in the core, as the highest concentrations from the shield channels entered the core in the area which received the highest concentration from the lower plenum.

With vanes positioned at an angle of 30 deg and with a shield coolant flow rate of 19.6 per cent of model flow, the fluid from the traced inlet was well distributed over the core with a maximum concentration of 62 per cent. The drop in total pressure from

M-2

an inlet to the downstream side of the flow baffle corresponded to 7.7 psi for the prototype as compared with 5.4 psi for Core 1.

Experiments will be concluded early in June, and a topical report summarizing flow and mixing studies with the 7-1/2-ft core design will be prepared.

X-Ray Photometric Examination of Fuel Elements

J. B. Schroeder and C. M. Schwartz

The X-ray photometer developed for the inspection of fuel elements has been completed. Tests on a 0.425-in. Zircaloy 2 rod with artificial defects showed that a void volume equivalent to a spherical void 0.010 in. in diameter could be detected at a circumferential speed of 1/2 rpm, or a 0.015-in. void at 2-1/2 rpm. This indicates the potential capability of the instrument for void and porosity detection in circumferential end welds of PWR-type fuel pins. Steps were machined in fuel-alloy filler plates nominally 0.050 and 0.140 in. thick. From photometry tests on these step plates at a scanning rate of 1/2 in. per min, the sensitivity of the photometer was calculated as follows: for clad 0.050-in. fuel elements, 0.006 g per cm²; and for 0.140-in. filler plate, 0.02 g per cm². A composite plate, composed of a series of uranium and niobium strips, carefully ground to dimensions computed so that adjacent areas of the composite would simulate small differences in composition of filler plate, was tested. This test indicated a sensitivity of 0.015 g per cm².

This completes the work on this project.

Improved Techniques for Corrosion Detection in Subassemblies and Clusters

G. G. Cocks and C. M. Schwartz

A special panoramic camera, designed for inspecting the walls of the water channels of fuel-element assemblies for corrosion, is in service at BAPD. This camera has proven to be overly sensitive to surface features other than corrosion; thus, it has been difficult to interpret the photographs accurately and to identify and distinguish corrosion from these other surface features. It is the purpose of this work to study the factors which affect the interpretation of the photographs and to improve the apparatus so that unambiguous measurements of the degree of corrosion can be made.

Two approaches have been adopted: (1) the use of a non-image-forming device based on diffuse reflectance of light introduced into the channel by means of a "light pipe" of Lucite, and (2) modification of the illuminating system of the panoramic camera.

A number of light pipes have been devised and tested. It has been found that with the particular light pipes tested, it is difficult to identify corroded spots, although scratches or painted lines show quite well.

M-3

In order to study modifications of the illuminating system of the camera, it has been necessary to construct a small model of the camera, which is now ready for use. During June, the camera will be run with various arrangements of the illuminating system. Different types of film will also be tested.

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

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

Flat-plate Zircaloy 2-clad fuel elements containing compartmented uranium dioxide fuel are being considered for use in PWR Core 2. A gas-pressure-bonding process is being evaluated for the preparation of the experimental flat-plate elements. The process consists of pressure bonding at temperatures of 1400 to 1550 F followed by an 1850 F heat treatment in a salt bath. The additional heat treatment accompanied by a phase transformation promotes increased grain growth that may not have been complete during the pressure-bonding cycle.

A series of flat-plate specimens, consisting of two Zircaloy 2 plates, has been bonded at various conditions to determine the optimum bonding conditions. Specimens were bonded at 5,000 to 10,000 psi for 4 hr at temperatures ranging from 1200 to 1500 F. The as-bonded specimens were subjected to an additional 5-min heat treatment at 1850 F. Three types of surface-preparation methods were also studied, namely: belt abraded, pickled with a hydrofluoric acid solution followed by a sulfuric acid pickle, and pickled with a hydrofluoric acid solution followed by a treatment in boiling sodium hydroxide.

The results of the metallographic examination of this series disclosed, as anticipated, that, as the bonding temperature was decreased, the consistency of the metallurgical bonds obtained also decreased. A similar effect was noted upon decreasing bonding pressure. The data obtained clearly showed that the belt-abraded surfaces produced the most consistent bonds for all pressure-bonding conditions tested. It was also apparent that the 1450 and 1500 F temperatures and the 10,000-psi pressure produced the best bonding for all three surface preparations. Excellent bonds were obtained at these conditions using belt-abraded surfaces. The sodium hydroxide-treated specimens bonded at 1400, 1450, and 1500 F appeared promising; however, the bonding results were not as consistent as the bonds obtained with belt-abraded specimens. The sulfuric acid treatment in some cases greatly improved the bond normally obtained with hydrofluoric acid-solution-treated specimens; however, the pickling action was difficult to control and the bonding results were somewhat erratic. Evaluation of all of the bonded specimens revealed that it is desirable to place the major emphasis on belt-abraded surfaces.

This series of flat-plate specimens were also subjected to destructive tests to determine if cladding embrittlement during pressure bonding or beta treating is a problem. Tensile tests, bend tests, and chisel tests were made on all of the flat-plate specimens to determine the effect of the bonding conditions on bond strength and the ductility of the cladding. The tensile tests are not completed; however, all of the

M-4

specimens tested to date have been ductile after pressure bonding and beta treating. They exhibited yield strengths of 51,100 to 53,900 psi with elongations of 21 to 28 per cent. All of the fractures during tensile testing appeared ductile. The bend tests have been completed, and all of the specimens displayed reasonable ductility during bend testing.

The bonds prepared at temperatures of 1200 F failed during chisel testing, and the bonds in some instances failed during chisel testing of the specimens bonded at 1350 F. From the results obtained with this series of flat-plate specimens, it was determined that future specimens should be bonded at 1500 F for 4 hr at 10,000 psi. In addition, the surface preparation used for all future specimens will be belt abraded.

All of the flat-plate specimens were subjected to an additional study by electron microscopy to determine the nature of the bond interface obtained during pressure bonding. The dots of contamination, or what was assumed to be contamination, along the bond interface appear to be small voids as viewed by electron microscopy. Additional metallographic studies indicate that the voids are small and localized. The voids do not appear to have any appreciable depth and only in the case of poorly bonded specimens do these voids form a sufficient plane of weakness to cause failure along the bond interface during destructive testing.

Previously, six specimens, with vapor-blasted and pickled components, containing natural uranium oxide cores were edge welded by Bettis and pressure bonded at Battelle. Examination of the specimens revealed that even though the surface preparation used was not optimum, the specimens bonded reasonably well without a protective can. As a result of the encouraging results obtained with these specimens, it was decided to prepare five additional edge-welded specimens with belt-abraded components and subject these specimens to the pressure-bonding cycle. These specimens have been pressure bonded and will be used to determine if it is feasible to prepare fuel elements without a protective can. If this process is feasible, the economy of the pressure-bonding process would be improved.

Investigation of the core-to-cladding reaction introduced during bonding and heat treating revealed that chromium and Neolube barriers are satisfactory. Although a series of irradiation specimens and some experimental specimens were prepared with chromium-plated cores, all future specimens will be prepared with graphite-coated cores. The latter coating is much more economically applied than the chromium plating. Specimens prepared with Neolube coatings have exhibited excellent corrosion behavior in 680 F water. This information, coupled with the Bettis evaluation of specimens pressure bonded without benefit of a barrier, definitely establishes the need for a barrier layer and graphite coatings applied in the form of Aquadag or Neolube appears to be the most suitable barrier layer.

Additional irradiation specimens for the WAPD-29-17 loop have been prepared. The specimens were all prepared with chromium-plated cores. In all cases, the pressure-bonding conditions used were 4 hr at 1500 F at 10,000 psi followed by a 5-min 1850 F heat treatment. A similar series of specimens will be prepared with graphite coated cores and will be compared to the chromium-plated specimens during irradiation testing.

M-5 and M-6

The feasibility of pressure bonding as a technique for preparing PWR-type fuel plates is being evaluated by the critical testing of 75 large-size specimens. These specimens are being pressure bonded primarily with graphite-coated cores at 1500 F for 4 hr at 10,000 psi. The bonded specimens will all be subjected to a series of tests including metallographic examination, corrosion tests, water-logging tests, burst tests of compartments, intercompartmental pressure tests, chisel tests, bend tests, and tests to determine if a fuel-to-cladding reaction occurred during the bonding and subsequent heat-treating cycle. In addition, it is planned to evaluate part of these specimens after pressure bonding to determine if the beta treatment is really necessary in the bonding cycle to obtain satisfactory specimens.

N-1

N. EVALUATION OF URANIUM MONOCARBIDE AS A REACTOR FUEL

F. A. Rough

This program is intended to provide irradiation and physical-property data on uranium monocarbide for potential use in the SRE. It is proceeding at an accelerated pace in order to get data as soon as feasible. The first capsule is tentatively scheduled for insertion in the MTR in July. A second experiment intended to provide data on the fission-gas retention characteristics of the monocarbide is planned for the Battelle Research Reactor. Meanwhile, research on the preparation and physical properties of cast uranium monocarbide is in progress.

Casting Techniques for the Preparation of Uranium Monocarbide

Emphasis is currently directed toward the preparation of enriched samples for various irradiation experiments. However, research on the physical properties of the monocarbide is continuing.

Two enriched UC specimens to be irradiated in the MTR have been prepared. These specimens have been cast and ground to size, and will be loaded into a NaK-filled capsule and sent to the MTR. The two specimens, finish-machined to 3/8 in. in diameter by 2 in. long, were made using uranium enriched with 8.4 w/o uranium-235. Three additional specimens were prepared as controls. Twelve additional enriched UC castings will be prepared for irradiation in the Battelle Research Reactor. Twelve specimens (1/4 in. in diameter by 1/2 in. long) and six control specimens are required for this test. The enrichment used for these specimens will be the same as that for the MTR specimens. Following this series, the original schedule for preparation and encapsulation of ten enriched specimens in five capsules for irradiation at MTR will be resumed.

Studies relating to the physical properties of cast uranium monocarbide are also in progress. Tentative linear-thermal-expansion coefficients of cast uranium monocarbide were found to be similar to values reported for UO_2 over the temperature range of 20 to 950 C. The electrical resistivity of cast uranium monocarbide measured at 28 C was 99 $\mu\text{ohm-cm}$. Thermal-cycling, thermal-shock, and heat-treatment studies of cast and ground specimens are now in progress.

In addition to the preparation of enriched specimens for the MTR, future plans include a study of the effect of varying composition upon hardness, castability, and microstructure of uranium monocarbide. Heat-treatment studies will be continued also.

Irradiation-Capsule Design for Uranium Monocarbide

R. B. Price, R. H. Barnes, and E. M. Chandler

The basic designs for the MTR and BRR irradiation capsules have been completed. The major components for the MTR capsules have been fabricated and the inner basket assemblies are being completed. The BRR capsule will be fabricated shortly.

The long-term NaK compatibility tests at 1100 F are continuing. A series of thermal-shock tests has been completed. Specimens, in NaK, were thermal cycled ten times from 1300 F to room temperature in a few minutes. Radiographs after one, five, and ten cycles indicated the specimens were still intact. These capsules are stored, unopened, awaiting a decision to conduct further cycling or to cut open and examine specimens more closely.

Physical drop tests of specimens encapsulated in a manner similar to that to be used for irradiation were conducted. Acetone was used to simulate NaK since its density and viscosity at room temperature are quite similar to those of NaK at 800 F, the average NaK temperature during irradiation. The capsule was dropped both in a horizontal and a vertical position from heights of 1, 2, and 3 ft onto concrete. After each drop, the capsule was opened and the specimens examined. The specimens, one drilled and one solid, remained intact throughout these tests.

Several specimens that were previously used in NaK compatibility tests were submitted to an ultrasonic through-transmission test and a Zyglo-penetrant test. Indications of material or structural differences or cracks were found in several specimens. Some correlation was observed between the ultrasonic and Zyglo tests. There was little correlation with radiographs of specimens.

Compatibility tests of UC specimens with kerosene and Zyglo were conducted for 1, 3, and 5 hr. No weight change was observed with kerosene. A small gain of less than 1 mg occurred in the Zyglo test. The weights returned to normal after cleaning in CCl₄.

Target dates for the loading of specimens into capsules for the MTR irradiations are:

MTR Capsule 1	First week of June
MTR Capsules 2 and 3	First week of July
MTR Capsules 4, 5, and 6	First week of August.

MTR insertion dates are still highly dependent upon MTR scheduling and the proposed experimental plutonium core test now tentatively scheduled to begin in June, Cycle 106.

N-3 and N-4

The target date for insertion of the BRR capsule is also June. Preliminary discussions have been made regarding the use of Core Position 56 for the BRR irradiation. Flux measurements are being made and a memorandum to the Safeguards Committee is being prepared. Tentative plans for the fission-gas-release measurements have been discussed with hot-cell and radioisotope-laboratory personnel. Operating temperatures, burnups, and fission-gas production have been calculated.

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O-1

O. DEVELOPMENTS FOR NMSR

A. W. Hare and R. F. Dickerson

The progress of the research and development concerned with the Nuclear Merchant Ship Reactor Program is reported in this section. The required UO_2 fuel pellets and final fuel-pin assemblies for the B&W test loop at the MTR are presently being fabricated and should be completed by the end of June. Capsule-irradiation studies have been initiated.

Fabrication of Urania Fuel Pellets for Loop-Test Studies

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

The objective of this program is to prepare UO_2 specimens containing 3 to 7-1/2 per cent U^{235}O_2 for irradiation testing for the NMSR program. Bulk densities of about 91 per cent of theoretical are desired.

Processing conditions suitable for fabricating pellets having the required densities were determined previously. During this report period, specimen fabrication was continued. About 60 per cent of the required specimens have been fabricated. These include 50 pellets 1/4 in. in diameter and 1/4 in. long, and 200 pellets 1/2 in. in diameter and 1/2 in. long.

Fabrication of Loop-Test Fuel Pins

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

The measurements of length, diameter, and apparent densities have been completed on the UO_2 -containing fuel pins for the first loading at the MTR. These specimens have been assembled into their respective test holders and have been shipped.

Current work is now being directed to the completion of the necessary out-of-pile loop specimens and control specimens for the first loading and the in-pile, out-of-pile, and control specimens for the second loading of the test loop.

Preirradiation MeasurementsR. B. Price, R. H. Barnes, J. C. Smith, D. B. Hamilton,
and E. M. ChandlerIn-Pile Loop Program

Two groups of fuel pins, UO_2 pellets in stainless steel tubes, have been marked for identification, and preirradiation measurements were made. The first group of

O-2

six short fuel pins were assembled and numbered as follows:

Assembly	Pin
BWC-301-304-1	756 ^{X(a)} , 753
BWC-301-304-2	751, 755 ^{X(a)}
BWC-301-304-3	752, 754

(a) Extended-end-cap pin.

Assembly numbers were stamped on the assembly lifting bars. Pin numbers were stamped on both ends of the individual pins, either on the flat end or on the beveled shoulder near the end.

Measurements included densities, diameters, lengths, and over-all assembly lengths. The preirradiating data are recorded in the standard Battelle preirradiation check list.

Fuel-Capsule-Irradiation Program

A series of twelve capsules, each containing one specimen, is tentatively planned for irradiation in the MTR. The general details of the program are given in Table O-1, which lists the variables covered by each capsule. The fuel specimens under consideration consist of a Type 304 stainless steel tube sealed at each end with solid plugs and filled with UO₂ ceramic pellets. Helium or lead is used as a thermal bonding agent between the pellets and tubes. The over-all length of each specimen is about 3.27 in. Except for being shorter in length, the specimens are identical to the proposed reactor fuel elements. During irradiation the surface temperature of the specimens should operate at 535 ± 90 F with a surface heat flux of 278,500 Btu/(hr)(ft²). The temperature of 535 F and the designated heat flux match the estimated conditions to which elements in the actual reactor will be exposed.

TABLE O-1. OUTLINE OF CAPSULE PROGRAM

Capsule (Number Arbitrarily Assigned)	Fuel Diameter, in.	Specimen Diameter, in.	Burnable Poison in Cladding (530 PPM Boron)	Heat-Transfer Bonding Agent	Enrichment in Uranium-235, w/o	Irradiation Time, MTR cycles
1	0.4425	0.500	None	He	3	2
2	0.4425	0.500	None	He	3	2
3	0.4425	0.500	None	Pb	3	2
4	0.4425	0.500	Yes	He	3	2
5	0.4425	0.500	Yes	He	3	2
6	0.4425	0.500	Yes	Pb	3	2
7	0.2675	0.3125	Yes	He	6	18
8	0.2675	0.3125	Yes	He	6	18
9	0.2675	0.3125	Yes	Pb	6	18
10	0.2675	0.3125	None	He	6	18
11	0.2675	0.3125	None	He	6	18
12	0.2675	0.3125	None	Pb	6	18

O-3 and O-4

Preliminary heat-generation calculations and heat-transfer studies have been made to determine the necessary capsule characteristics to satisfy the irradiation conditions. The capsule design will be completed during June. Fabrication of the capsule components will be initiated early in July. Specimens should be available in about 6 weeks for preirradiation examination.

RWD:CR T/all