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PROGRESS RELATING TO CIVILIAN APPLICATIONS
DURING AUGUST, 1960

by

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REPORTS RELATING TO CIVILIAN APPLICATIONS
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- BMI-1432 "Development of a Fuel-Element Leak-Detection System Based on the Principle of Isotopic Exchange", by James E. Howes, Jr., Thomas S. Elleman, and Duane N. Sunderman.
- BMI-1448 "Progress Relating to Civilian Applications During June, 1960", by (Rev.) Russell W. Dayton and Clyde R. Tipton, Jr.
- BMI-1451 "The Bonding of Molybdenum- and Niobium-Clad Fuel Elements", by Stan J. Paprocki, Edwin S. Hodge, and Paul J. Gripshover.
- BMI-1452 "Irradiation Effects on Massive Uranium Monocarbide", by Alan W. Hare and Frank A. Rough.
- BMI-1453 "Apparatus for the Study of Fission-Gas Release From Fuels During Postirradiation Heating at Temperatures up to 1600 C", by Russell H. Barnes and Duane N. Sunderman.
- BMI-1454 "Preparation and Evaluation of Aluminum-35 w/o Uranium Alloys Containing up to 3 w/o Tin or Zirconium", by Norman E. Daniel, Ellis L. Foster, Jr., and Ronald F. Dickerson.
- BMI-1455 "Progress Relating to Civilian Applications During July, 1960", by Russell W. Dayton and Clyde R. Tipton, Jr.
- BMI-1457 "Creep and Corrosion Properties of Zircaloy-2 in Steam at 750 F", by Edward J. Jablonowski and Frederic R. Shober.

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REACTOR MATERIALS AND COMPONENTS (AEC-DRD)

R. F. Dickerson

As a portion of the study concerned with stabilization of UO_2 , "oxidized" solid solutions of $\text{UO}_{2+x}\text{-La}_2\text{O}_3$ have been examined. After air firing for 20 hr at 1700 C the results indicate that the oxygen/uranium ratio increases with increased La_2O_3 content. The lattice parameters of these materials were less than those reported by Hund for solid solutions of $\text{U}_{2.67}$ and La_2O_3 . The Hund data indicate a much greater degree of oxidation than that obtained in the current work. The results of these studies also show that the stoichiometry of these solid solutions is not associated with their stability. With these experiments the work on this phase of the program has been completed.

The study of the effects of ultrahigh pressure and high temperature on the reactions of uranium oxides with other oxides is continuing. Results indicate that the reaction between UO_2 and Sc_2O_3 previously reported was due to oxidation rather than the effects of pressure. On the other hand, reaction between U_3O_8 and Al_2O_3 is induced by pressure. Attempts will be made to define the new phases formed with respect to composition and pressure-temperature existence region.

Specimens of Type 347 stainless steel have received estimated fast-neutron exposures of up to 7.615×10^{21} nvt in the ETR. Results from this program have pointed up the uncertainties involved in determining the total fast-neutron flux to which specimens are subjected. Nickel wires are not satisfactory as fast-flux indicators; therefore attempts have been initiated to develop a suitable indicator. The use of pure iron wire is being studied in some detail.

Niobium-base alloys are being studied as potential cladding materials for the Experimental Breeder Reactor. Annealed alloys of niobium-2.37 w/o chromium, -3.34 w/o zirconium, and -5.21 w/o vanadium and niobium-1.56 w/o zirconium-1.09 w/o chromium are being tested for both creep and tensile properties. Alloys of the same compositions have been prepared by consumable-electrode arc melting and canned in stainless steel for rod rolling. The specimens obtained from the resulting rod will be used for physical-property determinations.

The completion of the capsule designed to perform in-pile-creep experiments on Zircaloy-2 has been delayed until the difference between the estimated and the apparent thermal conductivity of the MgO insulation has been resolved. Tests indicate that the density of the MgO particles used in the thermal mock-up capsule was so low that the required thermal conductivity of 1.5 Btu/(hr)(ft)(F) could not be attained. The use of fused particles should provide the conductivity required.

Powders of HfB_2 (55 per cent enriched in boron-10) and natural HfB_2 have been irradiated in the ETR to boron-10 burnups of about 50 to 60 and about 90 per cent, respectively. Preirradiation and postirradiation metallographic examinations of the powders have been completed, and the starting materials have been analyzed by chemical and spectrographic means. X-ray diffraction studies are in progress, and preliminary results indicate that free carbon is present in the natural HfB_2 and that a second phase, believed to be nitride, is present in the enriched material.

The design of a capsule for the irradiation of fueled MgO has been initiated. Ceramic bodies consisting of a dispersion of UO_2 in BeO , Al_2O_3 , or MgO are being prepared in order to determine the optimum process for the preparation of actual test specimens, and appropriate reactor test space is being obtained.

In an attempt to determine the presence of small amounts of oxygen in sodium, mass spectrometry has received emphasis. Sodium samples containing about 30 ppm oxygen were extruded into a synthetic sapphire oven within the mass spectrometer, the free sodium was distilled, and the residue in the oven was heated to 720 C to decompose the sodium oxide while the mass spectrum was scanned for oxygen. Further refinements of this technique will be attempted. A careful study of the drop-time technique indicated that rapid determination of saturable impurity in the <10-ppm range may not be feasible. Additional study is being given to this possible method of analysis.

Friction tests have been completed between a ball and plate of Stellite No. 3 with and without molten sodium, and the data obtained from these tests are being analyzed. Electron microscopy is being used to study wear tracks in a number of materials for conditions of both sodium lubrication and no lubrication. A theoretical study of the thermodynamics of reactions involving oxides of sodium and molybdenum show that five possible reactions are thermodynamically favorable, which indicates that a Na_2MoO_4 film is readily formed.

Valence Effects of Oxide Additions to Uranium Dioxide

W. B. Wilson, A. F. Gerds, and C. M. Schwartz

The final results of the study of the "oxidized" solid solutions of $\text{UO}_{2+x}\text{-La}_2\text{O}_3$ have been obtained. The objectives of this phase of the investigation were (1) to confirm, if possible, the X-ray lattice-parameter data of Hund and Peetz* for solid solutions of $\text{UO}_{2.67}\text{-La}_2\text{O}_3$, and (2) to establish the oxidation state of the uranium to determine if dissociation of the uranium oxide (U_3O_8) occurred at the higher temperatures.

A determination was made, using microbalance techniques, of the oxygen/uranium ratio for solid solutions of UO_{2+x} containing varying concentrations of La_2O_3 . These materials were air fired at 1700 C for 20 hr. The results, given in Table A-1, show that the oxygen/uranium ratio increased from $\text{UO}_{2.36}$ at 40 mole per cent La_2O_3 to $\text{UO}_{2.72}$ for 75 mole per cent La_2O_3 . Several determinations for $\text{UO}_2\text{-ThO}_2$ and $\text{UO}_2\text{-Y}_2\text{O}_3$ yielded results which were in agreement with those obtained from the La_2O_3 solid solutions. An extrapolation of these results would tend to suggest that at very high La_2O_3 contents the uranium would be incorporated as essentially UO_3 .

The X-ray results of Hund and Peetz were not confirmed in the current studies even when reasonable precautions were taken to duplicate the methods by which they produced the solid solutions. Their solid solutions were produced by evaporation of nitrate solutions of $\text{U}_3\text{O}_8\text{-La}_2\text{O}_3$ followed by final calcination at 1200 C. The lattice parameters were, in all instances, smaller than those obtained in the present work. A comparison of lattice parameters is given in Table A-2 for $\text{UO}_2\text{-La}_2\text{O}_3$, Hund and Peetz's $\text{UO}_{2.67}\text{-La}_2\text{O}_3$, and the results of the study of the "oxidized" solid solutions in which the oxygen/uranium ratio is dependent upon La_2O_3 content.

*Hund, Von F., and Peetz, U., "Investigation of the Systems La_2O_3 , Nd_2O_3 , Sm_2O_3 , Yb_2O_3 , Sc_2O_3 , with U_3O_8 ", Z. anorg. u. allgem. Chem., 271, 6 (1952-53).

TABLE A-1. MICROBALANCE DETERMINATION OF THE OXYGEN/URANIUM RATIO FOR SOLID SOLUTIONS OF $\text{UO}_{2+x}\text{-La}_2\text{O}_3$ AIR FIRED AT 1700 C

Sample	Composition, mole per cent				Analyzed Oxygen/Uranium Ratio
	UO_2	ThO_2	La_2O_3	Y_2O_3	
206	50	50	--	--	2.46
207	25	--	75	--	2.72
208	50	--	50	--	2.58 ^(a)
209	50	--	--	50	2.50
210	45	--	55	--	2.53
211	50	--	--	50	2.50
183	50	--	50	--	2.46
184	45	--	55	--	2.49
185	60	--	40	--	2.36

(a) Value appears slightly high by comparison with the other determinations.

TABLE A-2. LATTICE PARAMETERS OF THE $\text{UO}_{2+x}\text{-La}_2\text{O}_3$ SOLID SOLUTIONS

Composition, mole per cent		Lattice Parameter, A		
UO_{2+x}	La_2O_3	$\text{UO}_2\text{-La}_2\text{O}_3$ ^(a)	Hund and Peetz ^(b) $\text{UO}_{2.67}\text{-La}_2\text{O}_3$	"Oxidized" ^(c) $\text{UO}_2\text{-La}_2\text{O}_3$
70	30	5.565	5.473 + U_3O_8	5.502
60	40	5.586	5.485	5.530 ^(d)
50	50	5.628	5.506	5.562 ^(d)
45	55	5.64	5.518	5.580 ^(d)
40	60	5.64	5.530	5.595
25	75	5.64 + La_2O_3	5.586	5.64 ^(d)

(a) Sintered at 1760 C in vacuum.

(b) Evaporated nitrate solution fired at 1200 C in air.

(c) Air-fired (1760 C) $\text{UO}_{2+x}\text{-La}_2\text{O}_3$ solid solutions of present study.

(d) Analyzed oxygen/uranium ratio is given in Table A-1.

Assuming the validity of the Hund and Peetz lattice parameters, their solid solutions indicate a much greater degree of oxidation than that obtained in the current work. Since they did not analyze for oxygen, it is not possible to establish the oxidation state of their materials.

On the basis of the present studies the "oxidized" solid solutions showed only a very slight variation in lattice parameter when run in air at 1350 C to 1760 C and in oxygen at 1475 C. This somewhat surprising result, suggesting very limited temperature and oxygen pressure dependency on dissociation, is probably due to the fact that the solid solutions were furnace cooled in air and deviated somewhat from their actual composition at high temperatures.

The direct air sintering at 1200 C and at 1370 C of the 60 mole per cent La_2O_3 composition yielded a noncubic phase, which bears some structural similarity to the rhombohedral structure of $\text{Ca}(\text{UO}_2)_2\text{O}_2$. The firing of this material at 1760 C in air for 1, 5, and 20 hr yielded cubic fluorite-structure solid solutions. This phase was not observed in the 75 mole per cent composition which was vacuum sintered at 1650 C and air oxidized at 1475 C in oxygen.

Analysis of the data obtained to date show that the stoichiometry of these solid solutions is not associated with their stability. The weight-loss and vapor-pressure measurements have shown a marked lowering of the volatility at and above the 50 mole per cent La_2O_3 composition. The only observable change which appears operative at this composition is the change in sign of the thermoelectric power from p-type to n-type which occurs for compositions of greater than 50 mole per cent La_2O_3 .

High-Pressure High-Temperature Solid-State Studies

W. B. Wilson and C. M. Schwartz

An investigation is being conducted on the effects of ultrahigh pressure and high temperature on the reactions of uranium oxides with various mixed oxides. In previous work, results were obtained which indicated the possibility of pressure induced reactions to occur between UO_2 and Sc_2O_3 and U_3O_8 and Al_2O_3 ; neither reaction has been observed to occur at normal pressure.

The reaction between UO_2 and Sc_2O_3 was investigated to determine if pressure was responsible for the observed reaction or, alternatively, whether the UO_2 was oxidized during the course of the high-pressure experiments, thus permitting the observed reaction to occur. A reaction between U_3O_8 and Sc_2O_3 has previously been observed to occur at normal pressure with the formation of solid solutions of fluorite structure.

Microbalance analyses of samples of UO_2 -50 mole per cent Sc_2O_3 , subjected to 70,000 atm at temperatures to 1000 C, have shown that oxidation of the uranium occurred. The resulting oxidation state of the uranium component was equivalent to $\text{UO}_{2.46}$, which is nearly identical to the composition analyzed for other solid solutions containing 50 mole per cent additive when fired in air at 1700 C. The close similarity of results strongly indicates that the oxidation occurred during the course of the high-pressure experimentation, and serves to establish what the "atmosphere" of the internally heated ultrahigh-pressure apparatus represents.

In contrast to the results for $\text{UO}_2\text{-Sc}_2\text{O}_3$, a reaction is observed between U_3O_8 and Al_2O_3 under pressure. At ambient pressure, no reaction has been observed to occur between any of the oxides of uranium and Al_2O_3 . (In each instance to date, attempts to react U_3O_8 with Al_2O_3 by direct sintering, evaporation of nitrate solutions, or coprecipitation techniques have resulted in only free U_3O_8 and Al_2O_3 being detected after air firing of the products at 800 C.) The pressure reaction between U_3O_8 and Al_2O_3 is apparently reversible, and samples fired at 800 C in air revert to free U_3O_8 and Al_2O_3 .

In view of the results for Sc_2O_3 , more emphasis will be placed upon the $\text{U}_3\text{O}_8\text{-Al}_2\text{O}_3$ reaction under pressure. Numerous experiments performed to date indicate that the new phases formed should be defined with respect to composition and their pressure-temperature existence region. Limited work has also been done to determine if pressure affects the reaction of TiO_2 with UO_2 and U_3O_8 .

Modifications to the press facility are being investigated to determine if the experimental conditions of pressure and temperature can be maintained for longer periods of time. The use of catalysts and higher temperatures will also be introduced in future work in an attempt to increase the rate of reaction between such refractory oxides.

Irradiation-Surveillance Program on Type 347 Stainless Steel

W. E. Murr, F. R. Shober, R. L. Ritzman, and J. F. Lagedrost

The objective of the surveillance program is to investigate the effects of fast-neutron irradiation (neutrons having energies greater than 1 Mev) upon the mechanical properties of Type 347 stainless steel. The program, which is in support of pressure tubes currently operating in the ETR, consists of the irradiation and subsequent post-irradiation testing of subsize tensile, fatigue, and impact specimens from approximately 14 capsules. The effects of two irradiation temperatures, process water and 600 F, will be studied on specimens irradiated to fast-neutron exposures of about 5.0×10^{21} to greater than 1.5×10^{22} nvt.

Ten capsules containing test specimens are being irradiated in the ETR, and two capsules are being irradiated in the WTR, with a third WTR capsule planned at a later date. The total estimated fast-neutron-flux exposures of all 12 operating capsules as of the end of ETR Cycle 30 (July 31, 1960) are shown in Table A-3. The ETR is currently operating in Cycle 31; the WTR, which has been closed down, is expected to resume operation in September.

A major uncertainty in carrying out the program lies in the determination of the total fast-neutron flux to which the specimens are subjected. Data have been collected on nickel dosimeter wires exposed adjacent to test holes for various lengths of time. The data indicate that if flux wires are retained in the reactor for multiple-cycle irradiations, the values obtained will be lower than for single-cycle insertions. Further, the flux values obtained from nickel wires exposed only during N_L runs are approximately 30 per cent greater than when the wires are irradiated for a complete cycle.

All data to date indicate that nickel dosimeters have serious drawbacks when used as fast-flux indicators. Consequently, BMI has initiated a limited investigation to

TABLE A-3. CAPSULES PREPARED FOR THE TYPE 347 STAINLESS STEEL IRRADIATION-SURVEILLANCE PROGRAM

Capsule	Type of Specimen in Capsule	Proposed Irradiation Temperature, F	Total Exposure as of July 31, 1960, nvt		Location	Remarks
			Top	Bottom		
BMI-24-2	Tensile and fatigue	120	5.853×10^{21}	6.426×10^{21}	ETR K-8-NE	Being irradiated
BMI-24-3	Tensile and fatigue	600	--	--	BMI	To be irradiated in WTR
BMI-24-4	Tensile and fatigue	120	5.451×10^{21}	6.550×10^{21}	ETR K-8-SE	Being irradiated
BMI-24-6	Tensile and fatigue	120	6.789×10^{21}	5.660×10^{21}	ETR K-8-NE	Being irradiated
BMI-24-8	Tensile and fatigue	120	5.550×10^{21}	6.366×10^{21}	ETR K-8-SE	Being irradiated
BMI-24-10	Tensile and fatigue	120	6.239×10^{21}	5.365×10^{21}	ETR K-8-SE	Being irradiated
BMI-24-12	Tensile and fatigue	120	7.615×10^{21}	7.218×10^{21}	ETR L-8-SE	Being irradiated
BMI-24-14	Impact	120	5.736×10^{21}	5.811×10^{21}	ETR K-8-NW	Removed for testing at BMI
BMI-24-15 ^(a)	Impact	600	3.5×10^{20}	1.28×10^{20}	WTR L-7-6	Being irradiated in WTR
BMI-24-16	Impact	120	6.455×10^{21}	5.372×10^{21}	ETR K-8-NW	Being irradiated
BMI-24-18	Tensile and fatigue	120	1.992×10^{20}	3.056×10^{21}	ETR K-8-NW	Being irradiated for postirradiation anneal- ing studies
BMI-24-19 ^(a, b)	Tensile and fatigue	600	4.53×10^{20}	4.33×10^{20}	WTR L-7-6	Fabricated to replace BMI-24-17; being irradiated in WTR
BMI-24-20	Tensile and fatigue	120	2.086×10^{21}	3.424×10^{21}	ETR K-8-NE	Being irradiated for postirradiation anneal- ing studies
BMI-24-22	Tensile and fatigue	120	2.660×10^{21}	3.886×10^{21}	ETR L-8-SE	Being irradiated for postirradiation anneal- ing studies

(a) Flux is reported as greater than 0.821 Mev; all other fluxes are greater than 1.0 Mev.

(b) Thermocouple-lead capsule.

determine other suitable fast-flux indicators. One method showing promise involves the irradiation of pure iron wire and subsequent determination of the amount of manganese-54 produced in the iron by an (n,p) reaction. Another method being studied consists of determining thermal flux in a test position and applying a correction factor based on a known fast-to-thermal flux ratio. Once a suitable dosimetry technique has been established, correction factors based on the best available knowledge will be applied to determine neutron-flux exposures for the BMI-24 series of capsules.

Development of High-Strength Niobium-Base Alloys

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

Operation of the sodium-cooled Experimental Breeder Reactor at temperatures above 725 C is dependent upon the development of a new cladding material. A eutectic reaction between iron and uranium at 725 C prevents the use of stainless steel. Niobium-base alloys are of interest as replacement cladding materials since no eutectic exists between niobium and uranium at 1000 C, and niobium can be alloyed to produce material with high elevated-temperature strength. In a program (BMI-1417) conducted to study selected niobium-base alloys, it was shown that the addition of chromium, vanadium, and zirconium improves the elevated-temperature strength of niobium. Even though the high melting points of these alloys indicate that they should retain their strength above 1000 C, experimental studies are needed to confirm this.

The current program includes preparation of alloys with the following nominal composition: niobium-2.0 w/o chromium, niobium-5.3 w/o vanadium, niobium-3.0 w/o zirconium, and niobium-2.0 w/o zirconium-1.0 w/o chromium.

Property determinations on the wrought alloys will include the following:

- (1) The recrystallization temperature (parameters: 1 hr anneal and 95 per cent cold work) will be determined for all alloys being studied. Mechanical and physical properties will be determined on recrystallized material.
- (2) The tensile strength of all alloys will be determined at 650, 800, and 1000 C.
- (3) Creep tests will be performed at 650, 800, and 1000 C at stress levels which will produce creep rates of approximately 0.1, 0.01, and 0.001 per cent per hr.
- (4) The thermal conductivity and thermal expansion will be determined from room temperature to 1000 C.

Ingots of niobium-2.37 w/o chromium, -3.34 w/o zirconium, and -5.21 w/o vanadium and niobium-1.56 w/o zirconium-1.09 w/o chromium alloys were double melted in a consumable-electrode arc-melting furnace. These 3 to 5-lb ingots have been forged to 1/2-in.-thick slabs at 540 C and rolled to 0.040-in. sheet at the same temperature. Specimens have been machined for creep- and tensile-strength determinations. These specimens received a 1-hr anneal at 1450 C. Both creep and tensile tests are in progress.

Examination of the annealed specimens (1450 C) by metallographic techniques indicates that all four alloys have completely recrystallized structures.

Ingots weighing approximately 4 lb each of niobium-2.0 w/o chromium, -3.0 w/o zirconium, and -5.3 w/o vanadium and niobium-2.0 w/o zirconium-1.0 w/o chromium alloys have been double melted in a consumable-electrode arc-melting furnace. These have had chips removed from each end of the ingot for chemical analyses, and have been canned in stainless steel for fabrication. The can assemblies will be rod rolled at 1200 C to 1-in.-diameter rod. A portion of this rod will then be shipped to ANL for extrusion study. The remaining rod will be further reduced by rod rolling for physical-property measurements.

Investigation of the Creep Properties of Zircaloy-2 During Irradiation at Elevated Temperatures

F. R. Shober, P. B. Shumaker, A. P. Young, and C. K. Franklin

A study of the creep behavior of Zircaloy-2 at elevated temperatures during irradiation has been undertaken. A comparison is to be made of the total creep deformation obtained in a reactor environment with that occurring in tests out of the reactor. Thermal tests of a mock-up capsule have been postponed until the thermal conductivity of variously prepared MgO has been resolved. Studies of strain aging in Zircaloy-2 are being continued.

The thermal analysis of the in-reactor creep capsule containing MgO has been interrupted to study in greater detail the thermal properties of fused MgO. Apparently, the density of the MgO particles used originally was so low that it was extremely difficult to obtain a sufficiently high packing fraction to reach the required thermal conductivity of 1.5 Btu/(hr)(ft)(F). Studies with the fused MgO particles of higher density indicate that higher conductivity values can be obtained. Thermal tests of the mock-up capsule will be made in September, and, if satisfactory, the creep capsule will be assembled.

The creep specimens have been fabricated and annealed to remove stresses induced by the final grinding. These specimens will be scribed to demark the individual gage lengths.

No additional tensile tests or electron-microscopy studies were made during August. The tensile-testing technique will be refined in order to define more accurately the presence of a yield point. These studies will be continued during September.

Radiation Stability of Boron-Containing Powders

R. A. Ewing and D. N. Sunderman

The objective of this program is to investigate the suitability of hafnium diboride as an alternate control material or burnable poison. Samples of natural HfB₂ powder and HfB₂ powder 55 per cent enriched in boron-10 were irradiated at the ETR to boron-10 burnups of about 90 and 50 to 60 per cent, respectively.

Preirradiation and postirradiation metallographic examinations of the powders have been completed. Chemical and spectrographic analyses of the starting materials indicate that they had the following compositions:

	<u>Analyzed Composition, w/o</u>				
	<u>Hf</u>	<u>Zr</u>	<u>B</u>	<u>C</u>	<u>Total</u>
HfB ₂ (natural)	81.5	1.05	9.87	6.05	98.5
HfB ₂ (enriched)	76.5	12.45	9.36	0.22	98.5

X-ray diffraction studies of the preirradiated powders are now in progress. Though not complete, preliminary results indicate that the carbon in the natural HfB₂ is present as free carbon rather than as a carbide, and also that there is a second phase present in the enriched HfB₂, believed to be a nitride. Lattice parameters of the preirradiated specimens are now being determined. X-ray diffraction studies of the irradiated specimens will begin upon completion of these determinations.

Evaluation of MgO as a Fuel Matrix

M. C. Brockway, A. K. Smalley, and W. H. Duckworth

The aim of this research is to compare the behavior of fueled MgO in an irradiation environment with that of Al₂O₃ and BeO, and also to compare the fission-product retention of fueled MgO ceramics with that of similar bodies based on BeO.

Work is now in progress on preparation of ceramic bodies consisting of a dispersion of UO₂ in BeO, Al₂O₃, or MgO. Processing conditions which will yield similar microstructures in the three types of bodies are being determined. Natural UO₂ is being used in these experiments. When suitable processing conditions have been established, samples containing enriched UO₂ will be prepared for encapsulation and irradiation.

Heat-transfer calculations leading to the design of the irradiation capsule are in progress, and the design should be completed during September.

Determination of Oxygen in Sodium at Concentrations Below 10 PPM

D. R. Grieser, E. H. Hall, J. W. Kissel, and J. McCallum

Continued interest in the use of beryllium, niobium, and zirconium in sodium-cooled reactor systems has placed a premium upon sensitive monitoring of the sodium because of the extreme oxygen susceptibility of these materials. The present program is aimed toward the development of a continuous oxygen monitor capable of ± 1 -ppm sensitivity at concentrations below 10 ppm and applicable to large-scale sodium-cooled reactors.

During August, work continued toward the attainment of a polarogram with liquid sodium drops. A polarographic cell for use with fused salts was designed, and a sodium-purification loop now is under construction. Fused salts selected for first study are phenoxides, caustic soda, and chloride eutectics. Electrochemical and a chlorine purge methods have been chosen for dehydration and purification of the selected salts, and the necessary apparatus has been prepared. During September, the preparation or assembly of equipment, purified salts, and auxiliary apparatus will be continued.

In the mass spectrometric study of oxygen in sodium, a sample of sodium containing about 30 ppm oxygen was extruded into a synthetic sapphire oven within the mass spectrometer. The oven was heated to about 325 C to distill the free sodium with a special condenser positioned to collect most of the sodium vapor. The residue in the sapphire oven was then heated to 720 C to decompose the sodium oxide while the mass spectrum was scanned for oxygen. A second sample of sodium was subsequently carried through the same procedure. The oxygen peak observed during the first run increased as the oven temperature was raised. However, other background peaks also increased; hence, the increase in the oxygen peak cannot be definitely ascribed to oxygen in the original sodium sample. In the second run the background peaks remained small and no significant increase in the oxygen peak occurred. In subsequent runs larger sodium samples will be employed in an attempt to increase the size of the oxygen peak relative to the background.

Analyses of sodium cold trapped at differing temperature levels are being made to determine the reliability and sensitivity of a modified vacuum-distillation apparatus. Some preliminary results from the test series have been examined. A relatively large divergence of results from anticipated values and a high degree of scatter have been noted thus far. The series will be completed during the next report period, and the technique will be reviewed when the results have been compiled.

The paper study of the drop-time technique has indicated that rapid determination of saturable impurity in the <10-ppm range may not be feasible. Accordingly, a review of the method is being made to establish the value of proceeding to the laboratory-experimentation phase.

The design of a system for the production of sodium containing predetermined levels of oxygen in the 0 to 10-ppm range is essentially complete. Component parts have been or are under construction. The main effort at present is the design of the direct coupling between the production system and the analytical equipment.

Friction and Wear of Sliding Surfaces in Sodium Environments

J. W. Kissel, W. A. Glaeser, and W. H. Goldthwaite

The slow-speed friction and wear behavior between metallic or cermet specimen pairs has been shown to be altered by the presence of molten sodium. For both molybdenum and tungsten specimens, the friction and wear were reduced after molten sodium had been applied. In the first case sodium molybdate was found adhering to the surfaces after sliding contact between specimens and this material, artificially applied to stainless steel specimens, was subsequently shown to provide lubricity. Attempts at identification of a similar film on tungsten specimens have not yet been successful.

During August friction tests were completed between a ball and plate of Stellite No. 3, with and without molten sodium. The data from these runs are being analyzed. The following friction specimen materials have been examined by electron microscopy: tungsten, tungsten carbide (K-91), titanium carbide (K-138A), Type 347 stainless steel, and stainless steel coated with Na_2MoO_4 . Wear tracks are being studied for conditions of both sodium lubrication and no lubrication.

A theoretical study of the thermodynamics of reactions involving oxides of sodium and molybdenum was also completed during August. Each of five possible reactions was considered at 1000 K, and the free-energy change was calculated. The results are as follows:

Reaction	ΔF° , cal per g mole
(1) $4\text{Na}_2\text{O} + \text{Mo} \rightleftharpoons \text{Na}_2\text{MoO}_4 + 6\text{Na}$	-19,000
(2) $2\text{Na}_2\text{O} + \text{MoO}_2 \rightleftharpoons \text{Na}_2\text{MoO}_4 + 2\text{Na}$	-53,000
(3) $2\text{Na} + 2\text{MoO}_2 \rightleftharpoons \text{Na}_2\text{MoO}_4 + \text{Mo}$	-87,000
(4) $\text{Na}_2\text{O} + \text{MoO}_3 \rightleftharpoons \text{Na}_2\text{MoO}_4$	-93,000
(5) $2\text{Na} + 4/3 \text{ MoO}_3 \rightleftharpoons \text{Na}_2\text{MoO}_4 + 1/3 \text{ Mo}$	-120,000

It is apparent that all these reactions are thermodynamically favorable with Reaction 5 being the most favorable and Reaction 1 the least. This study suggests that a Na_2MoO_4 film is readily formed, is quite stable in sodium, and even in systems where the Na_2O content is quite low the film should tend to be self-healing if it is rubbed off. A literature search is being undertaken regarding the kinetics of these reactions, with the hope of defining the specific temperature-time conditions required to promote the film formation.

STUDIES OF FUEL ALLOYS (AEC-DRD)

R. F. Dickerson

The study of niobium-uranium alloys is continuing with the major effort being devoted to the fabrication of irradiation specimens and the necessary capsules. Since the conditions available in the GETR best suit the requirements of the test, negotiations are now under way for suitable space in that reactor.

Thorium compounds including Th_2Al , ThBe_{13} , ThB_4 , ThC , ThC_2 , ThN , Th_3P_4 , Th_3Si_2 , ThSi , and ThS have been prepared by arc melting. Only the carbides have been successfully cast into cylindrical molds; however, further attempts to cast the other compounds will be made with solidification cooling rates reduced by appropriate mold design. The results of 1-week corrosion tests in 350 C Santowax show that the addition of 10 w/o uranium carbide increases the corrosion resistance of thorium carbide by a factor of more than 2. Complete evaluation of ThS prepared by induction melting thorium and sulfur in BeO crucibles indicates that attack on the crucible is severe. Further attempts to prepare massive ThS will be made by sintering and/or arc melting compacted ThS powder.

Niobium-base binary alloys containing 1, 3, 5, and 10 w/o plutonium have been prepared by arc-melting and drop-casting techniques. These alloys are being studied by metallographic techniques to determine the extent of plutonium solubility and/or the distribution of plutonium in the niobium. The preparation of thorium-base plutonium alloys has been interrupted because of a fractured glove-box window. Upon replacement of the window, thorium-5, -10, and -30 w/o plutonium alloys will be prepared. The compatibility study of PuO_2 with various metals and ceramics is being continued.

Development of Niobium-Uranium Alloys

J. A. DeMastry, B. D. Hamilton, A. A. Bauer,
and R. F. Dickerson

Utilization of niobium-uranium alloys as a reactor fuel requires that information concerning the irradiation stability of these alloys be available. These alloys, especially the niobium-20 w/o uranium alloy, have properties (reported in BMI-1400) which indicate their potential as high-temperature fuels. Therefore, an irradiation program has been planned to determine the irradiation stability of selected alloys at temperatures of approximately 1600 and 1800 F.

The physical properties, short-time tensile strength, and corrosion behavior of wrought niobium-10 and -20 w/o uranium alloys have been determined (BMI-1400). A study of the creep strength of these alloys will be undertaken when specimens are available. Difficulties encountered in fabricating alloys containing more than 30 w/o uranium have caused a temporary suspension of property studies of these alloys. Further studies are being made of the casting and fabrication characteristics of the above-mentioned alloys.

Examination of areas adjacent to cracks which occurred during fabrication of the higher uranium alloys indicated that these are segregated areas which are uranium-rich in composition. The cracks seem to form and propagate from these segregated areas. Therefore, two approaches are being taken in an effort to eliminate or alleviate the segregation problem. The first approach involves varying the casting technique. Ingots are to be cast into slab form from a skull-melting furnace; this technique should induce higher cooling rates during solidification than are possible by conventional arc casting. The effects of ultrasonic vibration during cooling of ingots will also be investigated. The structures and fabricability of ingots produced by the above methods will be investigated. A second approach consists of annealing castings for varying times at elevated temperatures in an attempt to reduce macrosegregation.

Samples for irradiation studies of niobium-10, -20, and -30 w/o uranium, with and without zirconium additions, are being prepared. The uranium enrichments are being varied to keep the uranium-235 atom density as uniform as possible for uniform heat generation during irradiation and to simplify capsule design.

Enriched specimens of the niobium-10, -20, and -30 w/o uranium alloys have been cast and fabricated and are being machined into pins 1 in. long by 0.150 in. in diameter. Difficulties were encountered in fabricating the 30 w/o alloy, and only lengths about 1 in. long were recovered. Alloys of niobium-20 w/o uranium with additions of 10 and 20 w/o zirconium are also being prepared.

Fabrication of components for two irradiation capsules is nearly complete. Each capsule will contain 15 fuel pins for irradiation at temperatures of 1600 and 1800 F.

It is planned to conduct the irradiation at the GETR, and negotiations are now under way for suitable space. Capsule assembly is expected to be completed in September. It is expected that capsules can be inserted into the reactor during the shutdown for Cycle 18 in November. The irradiations are to be conducted to 1 a/o total burnup and will require 5 GETR cycles.

Development of Uranium-Containing Thorium Compounds

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

Thorium and thorium-uranium compounds are being investigated with the aim of developing a high-temperature nuclear fuel suitable for use in a thermal-breeder reactor. Low thermal-neutron cross section, high melting point, and high thorium (uranium) density were properties considered when choosing the compounds to be studied. Under investigation are Th_2Al , ThBe_{13} , ThB_4 , ThC , ThC_2 , ThN , Th_3P_4 , Th_3Si_2 , ThSi , and ThS . These same compounds with a 10 a/o addition of uranium also will be investigated.

Arc-cast buttons of the above compounds have been prepared. These buttons are brittle and usually exhibit cracks resulting from cooling stresses; in some cases they fracture. Only the carbides have been successfully drop cast into cylindrical molds. Further attempts to cast these compounds will be made with cooling rates during solidification reduced by appropriate mold design.

Thorium and thorium-uranium carbides have been corrosion tested in 350 C Santowax for 1 week. The addition of 10 w/o uranium carbide to the thorium carbides

increases the corrosion resistance by a factor of more than 2. The ThC and ThC₂ corroded at rates of about 0.3 and 0.5 mg/(cm²)(day), respectively. However, no definite trend in corrosion resistance with carbon content, in alloy compositions between ThC and ThC₂, was observed.

Chemical analyses of ThS (12.1 w/o Sulfur), prepared by induction melting thorium and adding sulfur, indicate that only 6.3 w/o sulfur was retained in the melt and that 5 w/o beryllium was present as a result of attack of the BeO crucible. Further attempts to prepare massive ThS will be made by sintering and/or arc melting compacted ThS powder.

The thorium and thorium-uranium compounds which are prepared in suitable form will be corrosion tested in boiling water, air, CO₂, NaK, and Santowax; density and hot-hardness measurements will be made and thermal-shock resistance will be evaluated. The thermal-expansion and thermal-conductivity characteristics of the most promising compounds will be determined.

A Study of Plutonium-Containing Fuel Alloys

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

An investigation to select irradiation-resistant plutonium-containing fuel alloys is being conducted. Selection of alloy systems for preliminary studies was based on previous experience with similar uranium systems. Initially, binary niobium and thorium and ternary uranium-molybdenum and uranium-niobium alloys will be investigated.

Niobium-1, -3, -5, and -10 w/o plutonium alloys have been prepared by an arc-melting and drop-casting technique. Specimens of these alloys are being studied metallographically to determine the solubility and/or distribution of plutonium in the niobium. Should solid solubility exist, these alloys would be expected to exhibit outstanding high-temperature properties. If plutonium proves to be completely insoluble in niobium, but is present as discrete particles, the system may still be useful as a liquid-dispersion-type fuel element. Some niobium-base ternary alloys are also to be prepared. These include niobium-10 w/o zirconium-10 w/o plutonium and niobium-30 w/o zirconium-10 w/o plutonium alloys, which should be essentially solid-solution alloys, and a niobium-plutonium-silicon alloy which should consist of 30 volume per cent PuSi₂ dispersed in niobium.

The preparation of thorium-base alloys has been interrupted by the fracture of a glove-box window. Upon replacement of the window, thorium-5, -10, and -30 w/o plutonium and thorium-10 w/o plutonium-10 w/o zirconium alloys will be prepared. These alloys are to be fabricated, heat treated, and studied using metallography, hardness testing, and, perhaps, tensile testing. Uranium-10 and -15 w/o molybdenum-5, -10, -15, and -20 w/o plutonium will be similarly prepared and studied.

Future work on these systems depends on preliminary results. Any promising alloys will be studied in greater detail. Other systems being considered for future study include the titanium-plutonium and zirconium-plutonium systems.

Compatibility of PuO₂

S. J. Paprocki, D. L. Keller, and W. M. Pardue

The compatibility of PuO₂ with various ceramics and metals will be determined by following both analytically and metallographically the appearance of new phases upon heat treating powder mixtures and abutted surfaces of PuO₂ and the material in question.

Because of safety and criticality restrictions, only 100 g of PuO₂ received from Hanford was introduced into the powder glove-box train. Visual observation showed the oxide to be a subsieve size, thus precluding a standard screen analysis. In addition, several larger particles with slightly different appearance were noted. Because of these factors, and to more completely characterize the oxide, preparations are being made to submit samples of the material for petrographic and spectroscopic examination. In addition, X-ray diffraction studies of the material will be made to determine a base-line lattice parameter for future reference.

Work to date has been restricted to pure PuO₂ compacts in order to become familiar with the characteristics and sinterability of this material. A series of three compacts is being prepared with a compacting pressure of 60 tsi. These specimens contain: (1) no binder, (2) 0.05 w/o stearic acid, and (3) 0.05 w/o camphor-alcohol solution, respectively. The specimen containing no binder appeared fairly sound after being ejected from a die with the exception of a radial crack, but it crumbled upon handling. The specimen containing stearic acid appeared sound after pressing. The specimen containing camphor-alcohol binder is in process.

Each of the compacts in this series will be sintered at 1750 C in an argon atmosphere to determine the extent of densification.

FISSION-GAS RELEASE FROM REFRACTORY FUELS (AEC-DRD)

J. B. Melehan, R. H. Barnes, R. L. Ritzman,
S. D. Beck, M. Kangilaski, and F. A. Rough

Studies are being conducted to understand the fission-gas release from UO_2 , and data on diffusion of xenon in single crystals, on characterization of UO_2 , and on in-pile release of fission products are being obtained.

Diffusion in UO_2

During August, postirradiation release of xenon-133 from four pellets of sintered uranium dioxide was studied. Two of the pellets had densities that were 78 per cent of theoretical and the other two had densities 90 per cent of theoretical. One pellet of each density was heated at 1400 C for 5 hr. The second pellet of each density was heated for 5 hr at 1200 C, followed by 5 hr at 1400 C. Analysis of the data is in progress.

Measurements of fission-gas release from sintered uranium dioxide having a theoretical density of 95 per cent are also scheduled. In addition, similar specimens of sintered UO_2 are being retreated in dry hydrogen to determine whether the D/a^2 values can be altered by this procedure.

In most of the earlier experiments on UO_2 crystal plates, the plates were embedded in UO_2 powder during irradiation in order to maintain a uniform concentration gradient or prevent recoil losses at the surfaces. In addition, experiments have been conducted where surface depletion was allowed to occur during irradiation. Analysis of diffusion release from a semi-infinite plate for the case where surface depletion occurs is given below:

$$R/C_0 = \frac{1}{t} \left[\frac{1}{2} \sqrt{\frac{\tau}{\pi}} + \tau/2\nu \operatorname{erf} \frac{\nu}{2\sqrt{\tau}} \right] , \quad (1)$$

where

R = release rate

C_0 = initial interior concentration

t = time

$\tau = Dt/h^2$

$\nu = \epsilon/h$

ϵ = recoil range

$2h$ = plate thickness

D = diffusion coefficient.

Characterization of UO_2 and Model of Gas Release

The conditions under which one might expect idealized representations of fission-product release by diffusion to differ significantly from real releases are being considered. In preliminary analyses, the equations for in-pile equilibrium diffusion release of radioactive fission products were compared for plates and spheres having identical surface-to-volume ratios. The maximum difference in release rates, about

14 per cent, was found to occur when $\frac{a^2\lambda}{D}$ was approximately 10, where a = sphere

radius, λ = decay constant, and D = diffusion coefficient. If $D/a^2 = 10^{-10}$ per sec, this degree of difference would apply only for the longest lived gaseous isotope, krypton-85. If $D/a^2 = 10^{-7}$ to 10^{-6} per sec, the calculated releases of several isotopes would differ by 10 to 14 per cent due to the geometry of plates and spheres. Further consideration is being given to this problem.

The study of pore structure of a series of groups of UO_2 specimens ranging in density from 78 to 98 per cent of theoretical is in progress in order to better understand the nature and amount of surface that may release fission gases during diffusion. Studies include the determination of pore-surface areas by quantitative metallography and by gas adsorption. Pore surfaces are being determined by counting the pore-surface intercepts along a straight line. The relationship being used is

$$2 N_L = S ,$$

where

N_L = number of intercepts per centimeter

S = surface area in cm^2 per cm^3 .

In-Pile Fission-Product Release

Quantitative data have been obtained at temperatures of about 265 to 1800 F for the in-pile release of fission products from a 78 per cent dense UO_2 specimen. The results indicate little temperature dependence of release rates in this range. The experiment was conducted under conditions where surface recoils were entrapped in surrounding tantalum components. The lack of temperature dependence of release, up to 1800 F, indicates that the observed release is an irradiation effect which might result from an effect of irradiation upon diffusion or by a surface-vaporization mechanism.

Preliminary interpretations of the diffusion coefficients for the fission-gas releases from this 78 per cent dense specimen are given below and apply approximately for the range 265 to 1800 F.

<u>Isotope</u>	<u>$D/a^2, \text{sec}^{-1}$</u>
Xenon-133	8×10^{-10}
Xenon-133m	11.1×10^{-9}
Xenon-135	8×10^{-10}

These values, coupled with estimates of "a" (the equivalent sphere radius) based upon surface-area determinations, indicate that the apparent diffusion coefficient for these isotopes was about 1×10^{-17} cm² per sec.

Additional experiments are in progress on a 90 per cent dense specimen, and data are being obtained for the release of both krypton and xenon. Short-time runs have been made up to 2500 F, and preparations are being made for future extended runs at these higher temperatures.

GENERAL FUEL-ELEMENT DEVELOPMENT (AEC-DRD)

S. J. Paprocki

The irradiation testing of cermet fuels containing 60 to 90 volume per cent of UO_2 combined with a refractory metal matrix is planned. Fabrication techniques have been developed, and physical and mechanical properties have been determined. Based on these data, the cermet fuels appear to be superior to ceramic UO_2 and possess excellent potential for use in applications where a relatively high fuel loading is required.

The development of techniques for the fabrication of cermet fuels embodying UN has been initiated. Compatibility studies are being conducted to select the most promising matrix materials.

A study has been initiated with the objective of investigating methods of fabricating tubular fuel elements that will produce a quality product at a minimum of cost. The Allis-Chalmers superheater fuel design has been selected for this study. Various potential fabrication techniques will be explored and ultimately the most promising will be selected for development.

Studies are being conducted to obtain a basic understanding of the kinetics involved in a solid-phase bonding process utilizing temperature and pressure. The current investigation is concerned with determining the effects of thermal cycling through an allotropic transformation on a partial bond produced by pressure bonding.

Irradiation of Cermet Fuel Elements

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

A program of developing 60 to 90 volume per cent UO_2 cermets and evaluating them as a potential reactor fuel by microstructures and physical and mechanical properties has been completed. Methods investigated for densification included sintering, hot-press forging, and gas-pressure bonding. Gas-pressure bonding was found to be the best method of producing cermets of 90 per cent of theoretical density or greater having homogeneous microstructures. Other methods of fabrication resulted in inhomogeneous microstructures and densities lower than 90 per cent of theoretical.

Of the matrix materials used (chromium, molybdenum, niobium, and stainless steel) molybdenum was found to give the largest increase in thermal conductivity over that of pure UO_2 . Cermets of 80 volume per cent UO_2 -molybdenum resulted in a 7.6-fold increase in thermal conductivity at 800 C over thermal conductivities reported for bulk UO_2 at 800 C. Other matrix materials resulted in increases of 3.1 to 6 times the reported thermal conductivity of UO_2 at 800 C when added in the proportions of 80 volume per cent UO_2 -20 volume per cent metal. Thermal cycling of the UO_2 cermets to obtain mean linear-thermal-expansion measurements indicated structural stability in all cases. Room-temperature modulus-of-rupture values of 80 volume per cent UO_2 cermets ranged from 12,000 to 22,500 psi for all the matrix metals. Densities of the test specimens ranged from 90.7 to 97 per cent of theoretical. Other property measurements show that UO_2 cermets are equal to or superior to UO_2 , indicating

that UO_2 cermet s are attractive as a reactor fuel to overcome high central temperatures and poor fission-gas retention experienced with bulk UO_2 .

Irradiation plans are being made for the UO_2 cermet s during this fiscal year. Selection of the uranium loading and matrix materials is being made at this time.

Fabrication of Cermet Fuel Elements

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

Because of the improvements in physical and mechanical properties experienced by the addition of metal to UO_2 in concentrations up to 40 volume per cent, a program has been initiated to investigate the fabrication and to determine the properties of UN-base cermet s. Uranium nitride offers the advantage over UO_2 of a higher fuel loading with the same proportion of matrix metal.

Fabrication techniques will be evaluated for producing UN cermet s with densities 90 per cent of theoretical or greater. Currently UN powder is being prepared by reacting uranium metal with nitrogen. To select promising matrix metals for UN cermet s, a check of available thermodynamic data is currently being made by sintering UN powder with chromium, iron, molybdenum, niobium, tantalum, titanium, tungsten, and zirconium metal powder at 2250 F. Microscopic examinations of the cores will be conducted in order to select the most promising matrix materials. In addition to the UN work, a cursory evaluation of fabrication techniques for producing high-density UC cermet s having a UC loading of 60 volume per cent or greater will be conducted.

Development of Low-Cost Tubular Fuel Elements

S. J. Paprocki, R. J. Carlson, and E. G. Smith

The tubular fuel-element configuration has long been recognized as an ideal shape for many applications because of its inherent structural and thermal characteristics. While several fabrication techniques have been developed for the production of fuel elements of this shape, they suffer the disadvantage of relatively high cost or they produce elements of inconsistent quality. The objective of this program is to develop feasible fabrication techniques for this geometry which are both relatively low in cost and reliable.

In order to insure that the program will be realistic in the choice of dimensional and material variables, it has been decided to aim at the production of an actual fuel-element design, in this case the outer element of the Allis-Chalmers superheater fuel-element assembly. The design of this assembly consists of two concentric tubular elements together with a central control rod, all separated with spacers to form the water channels. The outer element, which is 0.789 in. in diameter, has a 0.006-in. cladding on the ID and OD and a core thickness of 0.015 in. The over-all length of the assembly is slightly over 77 in. The cladding has been designated as Type 316 stainless steel tubing and the core as 20 w/o UO_2 in a Type 316 stainless steel matrix. All studies will be planned in such a manner that any process techniques can be adapted to other material systems with a minimum of additional development.

While the initial investigations will cover the feasibility of several approaches, it is anticipated that the effort can be rather quickly narrowed to a few possibilities showing the most promise. Initially, various methods will be investigated for assembling the components which make up the element. These will include slip casting and direct pouring of stainless steel- UO_2 powder dispersions into an annulus formed by the concentric stainless steel cladding tubes. Subsequent to loading the annulus, explosive forming or isostatic pressing will be used to produce the finished element. Since these two operations are performed at essentially room temperature, it may be necessary to subject the elements to a heat treatment after their fabrication to enhance the quality of or to create a metallurgical bond between core and cladding.

Some effort will be made to load the powder core dispersion using vibratory-packing methods. However, this will come later in the program after other loading methods have been investigated.

Another approach will involve the development of techniques for fabrication of tubular fuel elements by expansion of roll-bonded flat plates using low-pressure gas. Picture-frame assemblies will be used to stack the fuel and cladding components into place. A parting compound will be used to prevent bonding in certain areas when the packs are subsequently roll bonded to the desired thickness. The rolled composite will be then expanded by the use of gas pressure to form a tubular shape.

Materials for this program were procured, and fabrication studies have begun.

Factors Affecting Pressure Bonding

G. W. Cunningham and J. W. Spretnak

The effect of the allotropic transformation of iron on the mechanism of pressure bonding is being investigated. Cycling about the allotropic transformation is known to cause irreversible plastic deformation. This procedure is also believed by some workers to enhance diffusion. The irreversible plastic deformation as related to pressure bonding has been studied by cycling Armco ingot iron and high-purity zone-refined iron in hydrogen and in vacuo.

Specimens with a surface roughness of $500\text{ }\mu\text{in.}$, rms, were clamped into position on surfaces of $8\text{ }\mu\text{in.}$, rms, and were cycled ten times from 1500 to 1700 F. The erratic results obtained were possibly due to different clamping pressures on the individual jigs, but the specimens all bonded at the tips of the asperities. The specimens were again run without benefit of clamps. They were heated at a rate of 50 F per min and cooled at a rate of 100 F per min for five cycles. The total decrease in thickness of both Armco iron specimens was $300\text{ }\mu\text{in.}$ and the total decrease in thickness for the zone-refined iron was $500\text{ }\mu\text{in.}$ Additional specimens are being prepared to investigate the effect of cooling rate.

GAS-PRESSURE BONDING OF CERAMIC, CERMET, AND DISPERSION FUEL ELEMENTS (AEC-FUEL CYCLE)

S. W. Porembka

The objective of this program is to refine and further develop the gas-pressure bonding process as a method for fabricating low-cost ceramic, cermet, and dispersion fuel elements. This technique permits the simultaneous densification and cladding of fuel, and, as a result, appears to be a promising method for maintaining or improving fuel-element quality while reducing fabricating costs. To date the major emphasis has been placed on the uranium dioxide fuel system since it offers the greatest potential for achieving a high burnup over a range of temperatures. The techniques developed for this system are expected to be applicable to the cermet and dispersion systems with a minimum of further development.

In past studies the cold-compaction and pressure-bonding characteristics of seven commercial powders and various mixtures were established. The behavior of Type 304 stainless steel-clad basic fuel-element shapes on pressure bonding has also been investigated. Current efforts are directed toward defining preliminary process specifications on the more promising fuel-geometry combinations, including refinement of uranium dioxide compacting and binder-removal techniques.

Uranium Dioxide Compaction Studies

S. J. Paprocki, D. L. Keller, and J. B. Fox

Uranium dioxide powder-compaction studies have been directed toward attaining high-green-density fuel cores which will achieve a range of desired densities on subsequent pressure bonding. Present studies concern the stoichiometry of the initial oxide and the final oxygen content and its relation to the final density. Particular emphasis has been placed on the fused oxides and fused oxide-ceramic oxide mixtures.

A batch of fused oxide was fired for 5 min at 850 F yielding an oxygen/uranium ratio of 2.19, which was higher than anticipated from previous uranium dioxide roasting experiments. After pressure bonding for 3 hr at 2100 F and a pressure of 10,000 psi, the oxygen/uranium ratio for the stainless steel-clad core was 2.03. Presently, a series of ten tubes is being tamped; these will be used to define the pressure-bonding conditions required for yielding stoichiometric oxide.

Flat partial-density Type 304 stainless steel sheet stock prepared by powder metallurgy has been produced for use in evaluating the feasibility of pressure bonding compartmented flat plates from powder-metallurgy components. These sheets measuring 2.0 by 10.0 by 0.150 in. were initially compacted at 27 tsi to a green density of approximately 70 per cent of theoretical. Sintering at 2000 and 2200 F yielded densities of 72 and 74 per cent of theoretical, with subsequent cold rolling producing further densification to 84 per cent of theoretical. It is anticipated that densities within this range will be required to match the densification of pressed oxide cores on pressure bonding.

The effectiveness of using buffer materials in the explosive compaction of uranium dioxide powder as contained in thin-walled stainless steel tubes has been demonstrated. Mild steel buffers which are readily removed by pickling eliminated the splitting experienced in earlier tests. In this approach sheet explosive is wrapped around the mild steel-jacketed tube and then detonated to produce the compacting forces. Preliminary measurements indicate bulk densities in excess of 75 per cent of theoretical have been achieved. Presently more precise density measurements are being taken.

Gas-Pressure-Bonding Studies

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

The fabrication of Type 304 stainless steel-clad UO_2 rod, tube, and flat-plate elements and flat-plate assemblies has been accomplished by the gas-pressure-bonding technique. Dimensional control of the bonded element was found dependent on the initial oxide density and method of compaction. Excellent dimensional tolerances were achieved when high-density cores were used. Investigations are being continued to develop different basic fuel-element shapes using high-density green-pressed oxide cores which will give similar results. Methods of minimizing densification effects are being studied.

Flat plates of Type 304L stainless steel powder cold pressed and sintered to densities of 71.5, 74.0, 77.0, 82.0, and 83.0 per cent of theoretical are being machined into frame components. These frames will be utilized in making flat-plate elements employing green-pressed oxide cores. In such assemblies simultaneous densification of the core and frame components will be achieved. Methods of containing these components while pressure bonding are also being investigated, and it appears that the stainless steel container can serve as the cover-cladding for these specimens.

Thermal-conductivity measurements have been completed on the second of the four UO_2 specimens submitted for evaluation over a range of 200 to 800 C. This specimen consisted of an oxide mixture of 75 w/o special dense and 25 w/o ceramic as-received UO_2 . It was pressure bonded at 2100 F for 3 hr at 10,000 psi and rerun at 2300 F for 3 hr at 10,000 psi, which yielded a final density of 95 per cent of theoretical. The sample measured 0.625 in. in diameter by 2.242 in. long, and final analysis showed an oxygen/uranium ratio of 2.02. The data from the two specimens run thus far show good agreement; however, the values for the second specimen are about 10 per cent higher than for the first. This could be due to the difference in starting oxide material and the higher density of the second specimen. When the data from these specimens were plotted along with the values for sintered oxides reported in the literature, the slopes of the curves of the pressure bonded-specimens showed a greater increase with temperature than the literature values. At 800 C the values for the current oxide were 30 per cent higher than the comparison values. Though the results on the pressure-bonded specimens were reproducible, there is some question as to why the pressure-bonded UO_2 should exhibit this difference in behavior. Other specimens are being planned to further the evaluation of pressure-bonded UO_2 .

DEVELOPMENT OF URANIUM CARBIDE (AEC-FUEL CYCLE)

F. A. Rough and W. Chubb

Uranium monocarbide and its alloys are being developed for the AEC as a part of the Fuel-Cycle Development Program. This research and development program includes: (1) studies of the synthesis and consolidation of uranium carbide powders, (2) development of melting and casting techniques for carbides, (3) determination of the mechanical, physical, and chemical properties of uranium carbides and their alloys, (4) measurement of the rates of diffusion of uranium and carbon in uranium carbides, and (5) attempts to obtain basic information on the nature of radiation-damage processes in uranium carbide.

During August, additional metallographic and X-ray studies were made on alloys of uranium containing approximately 5, 7, and 9 w/o carbon. These alloys were heat treated at temperatures from 1100 to 1800 C. The UC_2 needles present in the uranium-5 w/o carbon alloy were observed to transform to U_2C_3 at 1200 and 1400 C. The intimate eutectoid mixture of UC and UC_2 present in the uranium-7 w/o carbon alloy was observed to transform to U_2C_3 at 1200, 1400, and 1600 C. Specimens of these alloys heat treated for 1000 hr at 1100 C remain to be examined.

Studies of the effects of additions of other refractory carbides to uranium carbides have continued. Additions of 10 to 15 w/o of Mo_2C , NbC, TiC, VC, or ZrC generally improve the strength and corrosion resistance of UC. Extreme variability in behavior from sample to sample of both UC and its alloys makes it impossible to say at the present time that one alloying element (among those listed) is better than another. Much more detailed knowledge of the factors controlling the corrosion resistance of UC is required.

Constitutional-diagram data have been obtained by metallographic examination of ternary alloys of uranium, carbon, and a third element. Chromium, iron, and nickel appear as the uncombined elements in alloys with UC and UC_2 . Molybdenum, titanium, tungsten, and vanadium appear as carbides in alloys with UC and UC_2 . The solubilities of these carbides in UC and UC_2 are low or negligible. Niobium, tantalum, and zirconium also appear as carbides in alloys with UC and UC_2 . The solubility of these carbides in UC is 100 per cent; the solubility of these carbides in UC_2 is low.

A preliminary calculation of the activation energy for self-diffusion of uranium in uranium monocarbide discloses that it is identical for all practical purposes with the activation energy for interdiffusion of uranium and carbon. Diffusion rates are quite different, however. This result suggests that the mechanism by which uranium and carbon atoms diffuse is the same (i. e., substitutional vacancies, for example), but that the rate at which they diffuse is different.

Alternate Fabrication Methods for UC

S. J. Paprocki, D. L. Keller, D. E. Kizer, and J. M. Fackelmann

A study is being made of economical powder metallurgical methods for producing useful uranium carbide components.

Chromium additions to uranium carbide have served to accelerate sintering, but have introduced an undesirable side effect, namely the formation of very large spherical voids. There are several possible explanations for these large voids. First, impurities associated with the chromium powder used could be creating gas pressure in the compact. Another source of internal pressure would be the vapor pressure of the chromium itself. Still another possible mechanism for the formation of the voids is related to the relative surface-tension values for the chromium-gas interface and the chromium-UC interface. In any sintering process the driving force is due to surface tension and the rearrangement and elimination of voids so as to produce a component with a minimal surface energy. In this particular system chromium wets UC, and it would thus be expected that the chromium-to-UC surface tension is lower than that for chromium to gas. If the difference in surface tensions is sufficiently large, UC particles may actually be pulled along on the film of liquid chromium and be rearranged to give large voids with a chromium-rich phase. Such a situation would represent the lowest surface energy except for a system where no voids exist.

If the voids resulted from internal pressure the pressure in the sintering environment should influence the situation. Therefore, an argon atmosphere was employed in sintering a specially prepared UC-chromium mixture. To minimize possible impurities from the chromium powder the chromium was arc melted into a uranium-carbon-chromium button. The button was then crushed to an average particle diameter of approximately 25μ and green pressed to a density of approximately 9.1 g per cm^3 . Chemical analysis of the powder showed 4.97 w/o carbon and 1.26 w/o chromium. The specimens were sintered for 4 hr at 1700°C under argon. Average density after sintering was 10.5 g per cm^3 . This value is similar to what has been obtained in vacuum. Metallographic examination of these compacts is in progress.

Additional UC-10 w/o Mo_2C alloy buttons have been ball milled to a fine particle size along with several types of commercial UC that were recently obtained. Sintering studies on these materials are in progress.

Hydrostatic hot-pressing experiments on several types of UC are also in progress. Ten capsules containing pellets made from several types of commercial UC powder, a UC-5 w/o NbC alloy powder, and powder produced at Battelle were prepared for these experiments.

Special precautions were taken during the preparation of the capsules to prevent oxidation of the UC pellets, which did occur during previous hot pressing experiments. The pellets were sealed in evacuated niobium tubing and hydrostatically pressed at 2600°F for a period of 2.5 hr. Results of these experiments will be reported next month.

Melting and Casting Techniques for Uranium-Carbon Alloys

B. C. Boesser, E. L. Foster, and R. F. Dickerson

In the interest of developing techniques for the production of high-quality cast shapes, efforts are being devoted to the problem of purity and composition control for large volumes of uranium carbide arc melted and cast by skull techniques.

It has been established that splattering of the UC melt onto the graphite electrode tip is a major cause of poor composition control. This splashing is caused by outgassing of the melt, and even under supposedly identical melting conditions this is not the same from melt to melt. Recent research indicated that when very pure carbon (spectrographically pure grade) was used as an alloy addition outgassing was significantly reduced.

As a result, a program to determine the effects of different grades of carbon used in preparing electrode tips and as alloy addition has been started. Several castings have been made under the following conditions:

Starting materials	Ingot-grade uranium and 4 w/o commercial-grade graphite (AGR or AGX)
Power level	3600 amp at 22 v
Furnace atmosphere	Dynamic vacuum ($<25\mu$)
Melting time	35 to 40 min

Analyses of the castings were from 5.09 to 5.50 w/o carbon. Analyses of the skull after casting showed that the skull composition was substantially the same as the casting, indicating that no pickup of carbon from this source occurred during the melting cycle. Therefore, as much as 1.5 w/o carbon was picked up by the molten UC by electrode-tip erosion. The weight lost by the electrode tip agreed well with the increase in carbon content of the casting over the charge composition.

Next month AGOT reactor-grade graphite and vacuum-outgassed commercial graphite will be used as the alloy addition and to make the electrode tip. Fluctuations in carbon analyses will be evaluated using the same power level, charge composition, atmosphere, and melting time as used for the castings made this month.

Mechanical and Physical Properties of Uranium Monocarbide

G. G. Chesmar, R. W. Endebrock, E. L. Foster, and R. F. Dickerson

The potential offered by UC as an economical high-temperature fuel necessitates that such properties as strength, hardness, microstructure, and corrosion properties be determined. The effect of various alloy additions and heat treatments on the properties are also of considerable interest.

Heat treatments, in addition to those reported in BMI-1455, have been conducted, and the resulting structures have been examined. In summary, specimens containing nominal contents of 5, 7, and 9 w/o carbon were heat treated for 2 hr at 1800 C, 2 hr at 1600 C, 2 and 15 hr at 1400 C, 10 and 100 hr at 1200 C, and 100 and 1000 hr at 1100 C. In evaluations made during this report period, the 5 w/o carbon specimens exposed for 2 hr at 1400 C and 100 hr at 1200 C were noted to contain a grain-boundary phase that appeared to be U_2C_3 . Examination by X-ray diffraction confirmed this interpretation.

A compilation of the results of these heat treatments and resistivity and hardness measurements are given in Table F-1. The U_2C_3 was found to be considerably harder than UC or UC_2 , having a hardness of ~1000 KHN. The hardness of the UC and UC_2 was about 500 KHN, while the eutectoid mixture of UC and UC_2 had a hardness of about 700 KHN.

TABLE F-1. EFFECT OF HEAT TREATMENT ON THE PROPERTIES OF URANIUM-CARBON ALLOYS

Nominal Composition, w/o	Heat-Treatment		Resistivity, microhm-cm	Hardness, KHN	Microstructure
	Temperature, C	Time, hr			
U-5 C	As cast	--	40	567	UC plus UC_2 needles
	As cast	--	--	441	UC plus UC_2 needles
	1100	100	36	570	UC plus UC_2 needles
	1100	1000	--	--	Not yet examined
	1200	10	35	508	UC plus UC_2 needles
	1200	100	37	--	UC plus UC_2 needles plus U_2C_3 in grain boundaries at edge ^(a)
	1400	2	37	--	UC plus UC_2 needles plus U_2C_3 in grain boundaries at edge ^(a)
	1400	15	34	552	UC plus UC_2 needles
	1600	2	33	561	UC plus UC_2 needles
	1800	2	38	623	UC plus UC_2 needles
	As cast	--	55	722	UC plus UC_2 eutectoid
	As cast	--	--	780	UC plus UC_2 eutectoid
	1100	100	198	744	UC plus UC_2 eutectoid
	1100	1000	--	--	Not yet examined
U-7 C	1200	10	--	752	UC plus UC_2 eutectoid
	1200	100	--	--	U_2C_3 plus UC trace ^(a)
	1400	2	208	1064	U_2C_3
	1400	15	--	1077	U_2C_3 plus UC trace
	1600	2	195	1065	U_2C_3 ^(a)
	1800	2	132	--	UC plus UC_2 eutectoid, coarse
	As cast	--	100	469	UC_2
	As cast	--	--	535	UC_2
	1100	100	92	548	UC_2
	1100	1000	--	--	Not yet examined
U-9 C	1200	10	120	426	UC_2
	1200	100	73	591	UC_2
	1400	2	77	453	UC_2
	1400	15	85	516	UC_2
	1600	2	77	441	UC_2
	1800	2	80	485	UC_2

(a) Confirmed by X-ray analysis.

Various alloys of UC were prepared to determine flexure strength, hardness, and corrosion resistance and to characterize the microstructure. Flexure strengths of the various alloys are shown in Table F-2. The UC-NbC and UC-VC alloys show good strength. As can be seen in Table F-2, the corrosion resistance of most of the alloys showed improvement over unalloyed UC. The corrosion rates were generally $< -1 \text{ mg}/(\text{cm}^2)(\text{day})$.

TABLE F-2. EFFECT OF ALLOY ADDITIONS ON THE MECHANICAL PROPERTIES AND CORROSION RESISTANCE OF URANIUM-CARBON ALLOYS

Composition, w/o	Corrosion Rate in Santowax R at 350 C, $\text{mg}/(\text{cm}^2)(\text{day})$	Flexure Strength, psi	Hardness, KHN
UC	-1.0 (approximately)	9,000	550
UC-5 ZrC-5 Mo ₂ C	-0.42	22,400	1212
UC-5 ZrC-5 VC	-3.5	36,300	1420
UC-10 ZrC-5 Mo ₂ C	--	--	1222
UC-10 ZrC-5 VC	--	--	1365
UC-2.5 Mo ₂ C	--	--	804
UC-7.5 Mo ₂ C	-0.89	17,400	--
UC-15 Mo ₂ C	-0.64	17,400	861
UC-2.5 VC	--	--	1130
UC-7.5 VC	-0.59	26,400	1087
UC-15 VC	-0.39	21,000	--
UC-2.5 TiC	-1.30	1,700	940
UC-15 TiC	-0.23	12,200	--
UC-2.5 NbC	--	3,900	881
UC-7.5 NbC	-0.15	25,100	--
UC-15 NbC	+0.007	25,100	--

The effect of various alloying elements on the microstructure of uranium carbides is as follows:

- (1) As-cast samples of UC₂ plus additions of 5 and 10 w/o chromium, iron, or nickel plus sufficient carbon to form carbides possess microstructures showing metallic chromium, iron, or nickel plus UC₂ and graphite. The solubility of these metals in UC₂ and vice versa is negligible.

- (2) The pseudobinarys of UC and UC₂ with Mo₂C, TiC, VC, and WC involve eutectics.

The eutectic in the UC-Mo₂C pseudobinary system is estimated to be near 25 w/o Mo₂C and below 2300 C. The eutectic in the UC₂-Mo₂C system is estimated to be near 1 w/o Mo₂C and below 2300 C. Terminal solid solubilities in UC, UC₂, and Mo₂C are negligible.

The eutectic in the UC-TiC pseudobinary system is near 10 w/o TiC. The solubility of TiC in UC is less than 1/2 w/o. The eutectic in the UC₂-TiC pseudobinary system is at UC₂, and terminal solid solubilities are negligible.

The eutectic in the UC-VC pseudobinary system is estimated to be near 25 w/o VC and below 1800 C. The solubility of VC in UC is between 1 and 2.5 w/o VC at the eutectic temperature. The eutectic in the UC₂-VC pseudobinary system is estimated to be near 20 w/o VC. The solubility of VC and UC₂ is negligible.

The eutectic in the UC₂-WC system is near 25 w/o WC, and terminal solubilities are negligible.

- (3) The pseudobinary systems of UC with NbC, TaC, and ZrC are solid-solution systems, but castings of these alloys are subject to severe coring.

The pseudobinary systems of UC₂ with NbC, TaC, and ZrC are apparently eutectic-type systems with the eutectics generally near UC₂. The solubility of NbC, TaC, and ZrC in UC₂ is much less than 5 w/o.

Future research will be devoted to the completion of the heat-treatment evaluations. In addition, efforts have been initiated to prepare specimens for a study of the elevated-temperature properties of the uranium carbides. This will include physical and mechanical tests. Further alloying of the uranium carbides is planned, and tests to evaluate the effects of these additions on corrosion and strength will be conducted.

Uranium Monocarbide Diffusion Studies

W. Chubb, R. W. Getz, and F. A. Rough

The rate of self-diffusion of uranium in uranium monocarbide is being investigated using a tracer technique. Diffusion rates in uranium monocarbide are useful in predicting the elevated-temperature behavior of this material and for understanding the atomic structure of this material. Initially, the rate of interdiffusion of uranium and carbon in uranium carbides was determined between 1600 and 2000 C using graphite and liquid uranium saturated with carbon. Currently, the rate of self-diffusion of uranium in uranium monocarbide is being investigated using a diffusion couple consisting of an enriched uranium foil sandwiched between two lengths of depleted uranium monocarbide rod.

Using the values of the self-diffusion coefficients determined thus far in this study, a preliminary calculation was made to determine the activation energy and the diffusion constant for uranium in uranium monocarbide. The result of this calculation is the following equation:

$$D = 0.23 e^{\frac{-82,000}{RT}},$$

where 0.23 is the diffusion constant in cm^2 per sec, 82,000 is the activation energy in cal per mole, and T is the temperature in deg K. The activation energy is very high when compared with that of most metals, but it is reasonable for a material such as uranium monocarbide which has a melting point of approximately 2400 C.

A diffusion couple was bonded for 4 hr at 1400 C and is being diffusion annealed for 3840 min at 1700 C. This couple will be sampled and analyzed for uranium-235 content using the gross-gamma counting method of analysis for enrichment.

Future work will include the preparation and analysis of additional diffusion couples.

Effects of Radiation on UC

C. M. Schwartz, A. E. Austin, D. G. Freas,
J. E. Gates, and R. F. Dickerson

Natural-Uranium Studies

An investigation of radiation-induced changes in the structural and physical properties of UC is in progress. Specimens of natural uranium-5 w/o carbon have been irradiated in two NaK-filled capsules in the BRR to burnups of 0.01 and 0.03 a/o of the uranium. These specimens experienced irradiation temperatures of less than 500 F. The postirradiation examinations of these specimens have been completed, and the results were reported in earlier progress reports. The only major property change detected in these specimens was a considerable increase in the electrical resistivity. Results obtained by analysis of the X-ray diffraction line breadths indicate that internal lattice strains were the main cause of line broadening for the specimens subjected to 0.01 a/o burnup. These data also suggest that both crystal fragmentation and lattice strain contributed to the line broadening observed in the specimens subjected to 0.03 a/o burnup. Metallographic examinations of one specimen from each level of burnup failed to reveal any structural changes. An electron-microscopic examination of these irradiated specimens is in progress.

Enriched-Uranium Studies

Specimens of uranium-5 w/o carbon, uranium-6.7 w/o carbon, and uranium-8.5 w/o carbon alloys have been prepared from 10 per cent enriched uranium, and their physical properties have been measured. These specimens are to be irradiated in four NaK-filled capsules containing six specimens each. The specimens in three of the

capsules will be irradiated at 1400 to 1600 F to target burnups ranging from 0.1 to 0.7 a/o of the uranium. The specimens in the fourth capsule will be irradiated to a target burnup of 0.1 uranium a/o at an irradiation temperature not to exceed 600 F.

Heat treatments, determined from thermal-stability studies on specimens of natural uranium, have been performed on some of these irradiation specimens. The heat treatment for the uranium-5 w/o carbon specimens was 2650 F for 5 hr for the purpose of relieving casting stresses. Specimens of this composition in the as-cast condition will also be included in the irradiation experiments. The specimens of uranium-6.7 w/o carbon were heat treated at 2550 F for 15 hr to transform the specimens to an essentially equilibrium mixture of U_2C_3 with some UC present. The specimens of uranium-8.5 w/o carbon were also heat treated at 2550 F for 15 hr to relieve casting stresses.

These specimens are being encapsulated for irradiation. The components of these capsules, as well as the NaK to be used in them, have undergone purification treatments to reduce oxygen contents. These capsules are to be inserted into the BRR early in September.

PHYSICAL RESEARCH (AEC-DR)

F. A. Rough

Attempts to grow single crystals by a variety of methods have been carried out. Vapor growth and precipitation from a fused-salt bath are being discarded in favor of emphasis on fusion methods.

Growth of UO_2 Crystals From the Vapor Phase

C. A. Alexander and R. B. Filbert, Jr.

Work was continued this month in determining the purity of the UO_2 crystals grown from solvent evaporation of a KF solvent and by vapor-phase reaction of UF_4 with H_2O .

X-ray determinations indicated the presence of a foreign substance in the crystals grown by solvent evaporation in sufficient amounts to indicate that this method will not produce crystals of the desired purity. Lattice-parameter determinations on single crystals produced from the vapor-phase reaction indicated that pure crystalline UO_2 was produced with an oxygen/uranium ratio of 2.013.

Due to the promise of vapor-phase reactions, a different reaction was investigated. Some UO_2 powder was placed in a Vycor boat inside a Vycor tube and maintained at 600 C. Chlorine gas was then passed over the UO_2 powder to form UO_2Cl_2 . The gas flow caused the UO_2Cl_2 to be transported along the Vycor tube into a hotter region (1000 C) at which temperature the UO_2Cl_2 would decompose back to UO_2 and chlorine. Crystals of a few millimeters length and about 1 mm square were grown, but were U_4O_9 and U_3O_8 rather than UO_2 . An attempt was made to remove the oxygen from the U_3O_8 crystals by heating in hydrogen, but the crystals were badly deformed and cracked in the process. The cause of the formation of the higher oxides can be due to either the reaction $\text{UO}_2 + 2\text{Cl}_2 \rightarrow \text{UCl}_4 + \text{O}_2$, or to oxygen present in the chlorine gas employed in the experiment. An oxygen partial pressure of several millimeters of mercury would be necessary for formation of the higher oxides at the temperatures employed in the experiment.

If the difficulty is due to oxygen impurity in the chlorine cylinder, then this reaction may be of some promise in that Vycor may be used so that the crystal growth can be observed and that a higher partial pressure of uranium in the vapor can be realized at lower temperatures than with the $\text{UF}_4\text{-H}_2\text{O}$ reaction. Corrosion problems become a major factor in the $\text{UF}_4\text{-H}_2\text{O}$ reaction at temperatures of 1000 C or higher.

Study of these vapor and fused-salt methods of growing crystals is essentially complete, and little additional research is planned. Instead, fusion methods reported in the next section will be emphasized.

Fusion Methods to Prepare Single Crystals of UO_2

J. D. Jackson

The growth of boules of UO_2 using the Verneuil technique and a plasma-jet as the heat source was investigated during August. Several fused boules were produced, ranging up to 28 g in weight and 5/8 in. in length from feed material of minus 200 plus 325-mesh UO_2 shot, of which 20 to 50 per cent was recovered in the boule. X-ray diffraction study of one sample indicates nearly stoichiometric UO_2 ($\text{UO}_{2.01}$) with no observable second phase.

The boule has a fused top surface, while the sides are rough from partly fused feed material and as the result of vapor growth. The UO_2 produced tends to be polycrystalline, with long and narrow crystals in the direction of growth. In one boule, several large crystals were grown as shown by the difference in reflection from the crystal faces. Transgranular cracks were also noted. This cracking apparently results from thermal shock caused by the temperature gradient across the boule, which may amount to as much as 1600 C in a 1/4-in. -thick boule grown on a tungsten pedestal. Adding radiation shields and insulation around the sample and growing the boule on a piece of sintered UO_2 rod reduced the formation of cracks somewhat. Additional refinements are required, however, such as adding heating elements to the pedestal to reduce the thermal shock further.

These experiments show the feasibility of growing large crystallites of UO_2 by fusion in a plasma-jet and also show that the vapor pressure of UO_2 at melting does not seriously limit this process. For this reason, it would be advantageous to investigate other fusion methods in which the vapor pressure can be controlled by enclosing the UO_2 in a crucible. Either induction or resistance heating could be used to grow crystals by the Bridgman drop technique or the Czochralski crystal-pulling technique.

It is planned that investigation of the Verneuil technique using the plasma-jet to grow single crystals of UO_2 will be continued. Induction- and resistance-heating methods will also be investigated in attempts to pull crystals from the melt.

RADIOISOTOPE AND RADIATION APPLICATIONS (AEC-OID)

D. N. Sunderman

Work is continuing for the Office of Isotopes Development on two research programs in the areas of intrinsic tracers for process control and radiation-induced graft polymerization, while a third concerning the development of radiotracer quality-control systems was concluded this month.

Improvements in the method for removal of silica and the use of larger samples have increased the accuracy of the radiometric method for the determination of sulfate in portland cement. Experiments in which sulfur-35 was used as a tracer indicate that losses of sulfate in sample-preparation steps is negligible. Because higher precision and accuracy were possible in relatively clean sulfate solutions than in actual cement samples, it is suggested that careful standardization be done prior to the use of method in complex systems. Experimental work has been concluded, and a final report is being prepared.

The use of intrinsic tracers in the process-model system for iron removal has been shown capable of indicating accurately the efficiency of the precipitation process under a variety of experimental conditions. Following the solution of problems arising from a minor radioactive impurity in the iron-59 radiotracer, a series of experiments with varying pH was conducted. The results allowed comparison of radiotracer measurements with spectrophotometric analyses of the filter effluent.

Free-radical decay data have been obtained for irradiated normal-, iso-, and sec-buthylmethacrylate polymers. Experimental evidence for two distinct types of radical sites and the relation of the number of sites to the degree of branching of the ester confirm earlier results. Further work is under way on the n-propyl- and n-amylnmethacrylates and on three polymethylmethacrylates of different molecular weights.

Development of Radioactive-Tracer Quality-Control Systems

R. Lieberman, R. A. Ewing, and D. N. Sunderman

Development of a radiometric method based on the precipitation of $\text{Sr}^{89}\text{SO}_4$ for the determination of sulfate in portland cement continued during the month, with the principal emphasis on improving the accuracy of the method.

Sulfate values from previous experiments had tended to be low. Improvements which resulted in essentially quantitative recovery of sulfate included increasing the size of sample and more thorough elimination of silica by a preliminary dehydration of the sample after its initial dissolution with HCl. Recovery of sulfate was demonstrated by a tracer test using sulfur-35, which showed losses to be insignificant.

The sulfate content of a sample of a typical portland cement was determined to be 2.13 w/o SO_3 , based on the average of 11 determinations. Chemical analysis of the same cement gave 2.1 w/o SO_3 . However, values obtained by the radiometric procedure varied from 1.86 to 2.34 w/o, an error of ± 11 per cent.

The precision attainable by the proposed radiometric procedure, even after refinement, appears to be inferior to that of the standard BaSO_4 gravimetric method. Experimental work on the method has been concluded. Results of the work will be included in a topical report now being prepared.

Use of Intrinsic Radioactive Tracers for Process Control

J. L. McFarling, P. Gluck, J. F. Kircher, and D. N. Sunderman

During the month, experiments were conducted using the process-model system for iron removal as previously described. These experiments were performed under various conditions of iron concentration, total salt concentration, and pH of precipitation.

In general, the results showed good agreement between the radioactivity reduction ratios and the iron-reduction ratios determined by chemical analysis. However, the quantitative agreement was poor in some cases, particularly during the later experiments. The results of these experiments are given in Table H-1.

TABLE H-1. DATA FROM TYPICAL EXPERIMENTAL RUNS WITH THE PROCESS-MODEL SYSTEM FOR IRON REMOVAL

Time Since Start of Run, min	pH of Iron Precipitation	Count Rate in Feed Solution, cpm	Count Rate in Filtrate, cpm $\pm \sigma$	Radioactivity Reduction Ratio	Iron Reduction Ratio Shown by Analysis
105	4.5	29,000 $\pm$ 1000	940 $\pm$ 14	31 $\pm$ 1	25
180	4.5	29,000 $\pm$ 1000	820 $\pm$ 13	35 $\pm$ 1	30
240	4.5	29,000 $\pm$ 1000	730 $\pm$ 12	40 $\pm$ 1	34
285	4.5	29,000 $\pm$ 1000	540 $\pm$ 12	54 $\pm$ 1	49
315	4.5	29,000 $\pm$ 1000	460 $\pm$ 11	63 $\pm$ 1	60
105	5.0	13,200 $\pm$ 300	81 $\pm$ 9	163 $\pm$ 18	175
195	5.0	13,200 $\pm$ 300	109 $\pm$ 10	121 $\pm$ 12	110
225	5.0	13,200 $\pm$ 300	55 $\pm$ 9	240 $\pm$ 40	300
270	5.0	13,200 $\pm$ 300	61 $\pm$ 9	216 $\pm$ 30	210
135	5.5	40,000 $\pm$ 1000	90 $\pm$ 10	435 $\pm$ 44	530
165	5.5	40,000 $\pm$ 1000	78 $\pm$ 10	513 $\pm$ 51	610
225	5.5	40,000 $\pm$ 1000	98 $\pm$ 10	408 $\pm$ 41	460
300	5.5	40,000 $\pm$ 1000	104 $\pm$ 10	384 $\pm$ 38	610
345	5.5	40,000 $\pm$ 1000	307 $\pm$ 11	130 $\pm$ 5	60
360	5.5	40,000 $\pm$ 1000	109 $\pm$ 10	366 $\pm$ 37	1800

The cause of the poor agreement observed in some cases has now been determined to be the presence of soluble radioisotope impurities in the iron-59 tracer, in quantities considerably less than 1 per cent of the total radioactivity. Manganese-54 has been tentatively identified as one of the impurity activities.

This problem has been solved by a preliminary radiotracer purification step, and further experimental work is now under way. In the course of purifying iron-59 tracer

solutions, the impurity activities will be concentrated, and further attempts will be made to identify them.

The survey of unit operations as intrinsic radiotracer applications is nearly complete. A total of 14 different unit operations have been covered in this study.

Next month, the bulk of the experimental work on the iron-removal application will be completed. The unit-operations survey will be completed early in September.

Graft-Polymerization Studies

I. S. Ungar, F. A. Sliemers, W. B. Gager,
J. F. Kircher, and R. I. Leininger

An examination of the free-radical decay data for the normal-, iso-, and sec-butylmethacrylate polymers confirms earlier findings that (1) at least two distinct types of free-radical sites are formed during irradiation, and (2) the total number of sites formed increases with increased branching of the hydrocarbon tail of the ester side chain. This work is being extended to include the tertiary-butylmethacrylate polymer.

Curves of free-radical sites versus total dose are being obtained for the n-propyl- and n-amylmethacrylate polymers. The results from this study should be available during the first week of September. The data will be used to establish the validity of an earlier study of the efficiency of free-radical-site formation. The G-values indicated that an odd-even type relationship exists among the methacrylates. The values obtained were considerably higher for the normal hydrocarbon chains having odd numbers of carbon atoms.

In addition to the work described above, three polymethylmethacrylates of different molecular weights have been prepared. These materials will be further fractionated, and the effect of molecular weight on the efficiency of free-radical-site formation will be determined. It is hoped that a number of fractions having molecular weights in the 5 to 100,000 range can be isolated for this study. Earlier work had indicated little or no effect of molecular weight in the 5 to 15,000 range.

VOID-DISTRIBUTION AND HEAT-TRANSFER STUDIES (AEC-EURATOM)

D. V. Grillot, F. G. Peter, R. O. Wooton, H. M. Epstein, and J. W. Chastain

Battelle is conducting a program to evaluate steam void content, heat transfer, and slip ratios in the subcooled and bulk boiling regions of simulated reactor-coolant channels.

During August further attempts to correlate the data were made. A correlation for slip ratios proposed by Bankoff* is at present being studied. Bankoff derived the following equation for the ratio of the mean velocities of vapor and liquid, μ''/μ' , in terms of the mean local void fraction, α :

$$\frac{\mu''}{\mu'} = \frac{1 - \alpha}{K - \alpha} .$$

At present the flow parameter K is being studied as a function of pressure, quality, and mass flow rate. Tentatively, it can be stated that K is almost independent of pressure but appears to be dependent on mass flow rate.

As a first approximation K can be related to the flow rate, W, by the following equation:

$$K = 0.725 + 0.497 \left| 1 - \frac{W}{850} \right| .$$

In the range of parameters now being studied, K has values between 0.725 and 0.90. These values are in good agreement with the values of K predicted analytically by Bankoff. During the coming month correlations based on Bankoff's model will be continued, and attempts will be made to derive an equation for K as a function of the significant parameters.

During August revisions in the design of the test section were made. These revisions were begun in an attempt to increase the lifetime of the test section, and several designs now under consideration appear promising. During the coming month tests will be continued using new test sections.

*Bankoff, S. G., "A Variable Density Single-Fluid Model for Two-Phase Flow With Particular Reference to Steam-Water Flow", ASME-AIChE Heat Transfer Conference, Storrs, Conn., 1959, Paper No. 59-HT-7.

MATERIALS DEVELOPMENT AND EVALUATION (HAPO)

F. R. Shober

The electrical and thermal properties of UO_2 are being measured before and after irradiation. Preliminary thermal-conductivity measurements to 1025 C have been made on a UO_2 disk specimen. In an investigation of the creep properties of Zircaloy-2 at elevated temperatures, tests on cold-worked material and on annealed materials are continuing under several test conditions.

Work has been concluded on the development of corrosion-resistant welding alloys for use with Hastelloy F to contain decladding solutions. A topical report is in preparation.

A program directed toward the development of a metallic container for the fused chloride-electrolytic fuel-recovery process has continued with the corrosion experiments on nickel and nickel alloys.

Thermal Conductivity and Electrical Conductivity of UO_2

C. F. Lucks and H. W. Deem

Preliminary measurements have been made on the first UO_2 disk specimen, identified as Fabrication Specimen A. This specimen was prepared by cold pressing micronized PWR-grade UO_2 , mixed with Carbowax binder, in a 300-ton press and then sintering the compact in hydrogen at 1650 C. Table J-1 gives tentative measured thermal-conductivity values. The measured density for this specimen is 95.3 per cent of theoretical.

Continuing the program on the effect of fabrication on the thermal conductivity of UO_2 , HAPO has prepared a vibrationally compacted UO_2 specimen. During August preparations were made for drilling the specimen for thermocouples.

No experimental work was done on irradiated specimens this month.

TABLE J-1. TENTATIVE THERMAL-CONDUCTIVITY VALUES OF UO_2 FABRICATION SPECIMEN A, ADJUSTED LINEARLY TO 100 PER CENT DENSITY

Temperature, C	Thermal Conductivity, w/(cm)(C)
401	0.047 ₃
604	0.039 ₆
808	0.032 ₉
884	0.034 ₀
918	0.029 ₂
1035	0.025 ₉
1062	0.025 ₁

Mechanical Properties of Zirconium Alloys

L. P. Rice and J. A. VanECHO

The program of evaluating the long-time creep properties of Zircaloy-2 sheet is continuing as outlined in BMI-1455. The program consists of four phases. Briefly, these four phases include study of: (1) long-time creep of cold-worked sheet, (2) long-time creep of annealed sheet, (3) effect of temperature cycle on the creep properties of both annealed and cold-worked material, and (4) effect of temperature and stress cycle on the creep properties of annealed material. Temperatures of interest range from 290 to 400 C (550 to 750 F). The long-time tests are continuing to as long as 20,000 to 25,000 hr and the cyclic tests for 3000 hr. Twenty creep machines are being utilized in this work.

Since most of the tests now in progress are of long duration there is very little change from one month to the next. However, as indicated in earlier reports, there is a definite pattern concerning the relationship of the cycled to the noncycled tests. In all cases to date, the temperature-cycled tests produce the highest creep rate, the non-cycled tests the lowest, and the stress- and temperature-cycled tests an intermediate creep rate. The latter tests are still in the initial stages, and the pattern may change as additional data are accumulated.

The 20 tests now in progress are expected to continue during the next month. Major emphasis will shift more and more to creep testing of annealed Zircaloy-2 and determining the effect of cycling both stress and temperature on the creep properties of this material.

Materials of Construction for the Fused Chloride-Electrolytic UO₂ Process

E. L. White, P. D. Miller, C. L. Peterson, and W. K. Boyd

This research program is being directed toward developing a metallic container for the HAPO fused chloride-electrolytic fuel-recovery process. The process consists of chlorinating uranium oxide (U₃O₈) with chlorine or hydrogen chloride in an equimolar melt of sodium and potassium chlorides to form uranyl chloride. In a second step, uranium dioxide (UO₂) is deposited at the cathode by electrolysis of the molten salt solution. The high temperature, the active gases, and the molten salt produce a highly corrosive condition.

In the first series of corrosion experiments, chlorine is being used as the chlorinating agent. The materials being evaluated are exposed to the molten salt, containing U₃O₈, while dry chlorine plus oxygen is bubbled through the melt to simulate the first step of the process. The second step is then simulated by bubbling only chlorine through the melt. This cycle can be repeated for the desired exposure period. The materials have been chosen from commercially available alloys to indicate the effects of various alloying elements in different alloy systems.

The first two experiments in the nickel-alloy system have indicated that both pure nickel and Hastelloy B are attacked very rapidly under these conditions, and also that extensive localized attack occurs in the vapor-liquid interface zone.

The present series of experiments with chlorine will be continued, using other nickel alloys and other alloy systems. A second series of experiments similar to the chloride series will be conducted using hydrogen chloride as the chlorinating agent.

REFINING THE GRAIN SIZE OF URANIUM BY VIBRATIONAL ENERGY (SRL)

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

The application of vibrating energy to the refinement of the microstructure in cast metals has received considerable attention and has proved useful in producing relatively fine-grained material. However, during subsequent fabrication, grain growth may occur which will negate the benefits derived from the prior vibrational treatment. Therefore, it would be desirable if the vibrational energy could be applied to the metal in the solid state, after the grain-coarsening fabrication, and still yield a relatively fine, uniform grain size in the metal. This is particularly true in the case of uranium which is made in blanks for subsequent coextrusion. In this case, large grains or large groups of similarly oriented grains may result in irregular core-to-cladding interfaces and add to the problems associated with attaining a uniform, thin, cladding thickness. Therefore, a program of research concerned with the feasibility of refining the grain size in unalloyed uranium through the application of vibratory energy in conjunction with thermal energy has been initiated.

In other materials, the frequency and the power level of the vibratory energy may affect the ultimate grain size. Therefore, at least two frequencies and two or more power levels are to be investigated. The furnace which is to be used in the experiments consists of an evacuated quartz tube with an induction coil for heating the uranium specimens. Coupling of the vibratory source to the specimens is to be achieved by a water-cooled threaded adaptor of a length designed to impart the maximum vibratory energy to the center of the specimen. The central portions of the 8-in. -long specimens will be heated to either the recrystallization temperature or to about 30 C above the alpha-to-beta transformation temperature. At this temperature the specimen will be subjected to the vibrational energy. The temperature gradients that will be present in each specimen will allow the qualitative evaluation of a number of temperatures with each test piece. Specimens of uranium in the hot-alpha-worked, in the hot-alpha-worked and beta-quenched, and in the gamma-annealed conditions are to be examined by metallographic techniques. Of these, the hot-alpha-worked and the gamma-annealed uranium will be subjected to vibration.

Future work will be concerned with completing the metallographic examination of the above-mentioned specimens, the design of the furnace and its construction, and the heat treatment of 14 uranium specimens which are to be gamma annealed to furnish material for the studies.

REFLECTIVE-INSULATION STUDIES (KAISER)

L. J. Flanigan and H. R. Hazard

Studies are being conducted at Battelle to determine the characteristics of reflective insulation materials suitable for use in the EGCR. Suitability of the insulation is being determined by subjecting it to repetitive thermal cycling and to rapid decompression, and by measuring heat flow at prototype operating conditions. Work previously reported includes completion of studies of one sample of insulation, and completion of decompression studies and initiation of thermal-cycling studies for two other samples.

In August, thermal-cycling studies of all samples were completed.

On August 8, a heat-transfer specimen was received from the Solar Aircraft Company. It was immediately installed in the heat-transfer equipment, and studies to determine conductance were started. Several equipment failures have delayed completion of the studies, but conductance data have been obtained at atmospheric pressure and at 300 psi. The studies will be resumed the first week in September.

On August 10, a heat-transfer specimen was received from the Johns-Manville Corporation.

In September, the conductance studies will be completed and the heat-transfer equipment will be calibrated.

A topical report covering all the decompression, thermal-cycling, and heat-transfer studies will be prepared at the conclusion of experimental studies.

PROBLEMS ASSOCIATED WITH THE RECOVERY OF SPENT REACTOR FUEL ELEMENTS (ORNL)

R. A. Ewing and C. L. Peterson

Rates of dissolution of irradiated 91 w/o ThO_2 -9 w/o UO_2 Consolidated Edison fuel were higher for uranium burnups above 1 a/o than below. Both were appreciably higher than those obtained with unirradiated fuel. Fission-product activity was greater in Darex than in Sulfex decladding solutions, as were thorium and uranium losses. The major portion of the radioactivity (75 to 90 per cent) in either Sulfex or Darex decladding solutions was, however, induced activity resulting from cobalt-60.

Study of the Effects of Irradiation on Cladding- and Core-Dissolution Processes

R. A. Ewing, H. B. Brugger, and D. N. Sunderman

Dissolutions of Irradiated CETR Fuel Pins

Chemical and radiochemical analyses are in progress on the solutions from previous dissolution experiments on five CETR Consolidated Edison prototype fuel pins. Thorium and uranium losses to the decladding solutions, based on core weights, have been reported previously.

Core-dissolver solutions were sampled at 1, 3, and 5 hr during the first pass and at 2 and 5 hr during the second pass. Analyses of these samples indicate that there was no selectivity of dissolution, as both the thorium and the uranium dissolved at essentially the same rate. There were some indications that the chemical reactivity of the cores increased with increased burnup. At uranium burnups in the 1.02 to 1.37 a/o range, from 90 to 99 per cent of the core dissolved in the first 5-hr pass, and the balance in a second 5-hr pass. The specimen with lowest burnup (0.86 a/o) dissolved only to the extent of about 83 per cent in the first pass, and only this specimen had a measurable residue (~0.5 per cent) remaining after the second pass. In comparison, characteristically only about 60 and 94 per cent of duplicate unirradiated cores were dissolved by the same procedure.

Some qualitative and semiquantitative radiochemical studies have been made on cladding and core solutions using gamma-ray spectrometry. Cladding solutions were fractionated by ion exchange. Core solutions were fractionated by solvent extraction with TTA. Gross gamma activities of the cladding solutions were in the 1 to 2×10^8 cpm/ml range, about one-tenth of the value obtained on the previously examined ORR-irradiated fuel pins. Examination of the fractions indicated that, as before, 75 to 90 per cent of the gamma activity resulted from cobalt-60. On these 14-month-cooled specimens, the only other major activities observed in cladding solutions were cerium-144 and zirconium-95 and niobium-95. These accounted for most of the remainder of the activity. The amount of cerium-144 and zirconium-95 and niobium-95 activity increased with increasing thorium and uranium losses. In Sulfex Run 3 (uranium loss = 0.07 per cent) these radioisotopes accounted for about 10 per cent of the radioactivity, whereas in Darex Run 2 (uranium loss = 0.8 per cent) they accounted for about 20 per cent of the total.

M-2

Analysis of an NaOH solution from the off-gas scrubber by gamma spectrometry indicated that the principal activity present was radioniobium, confirming similar earlier observations at ORNL. Since a common scrub solution was used for Runs 3, 4, and 5, the source of the radioniobium cannot be definitely stated. This will be investigated further in the next series of dissolutions of irradiated fuel pins.

Dissolution experiments on high-burnup (12,000 to 25,000 MWD/T) Consolidated Edison fuel pins are scheduled to begin next month.

VARIABLE-MODERATOR REACTOR CRITICAL-
ASSEMBLY STUDIES (ATL-A)

R. A. Egen, L. H. Bettenhausen, W. S. Hogan,
D. A. Dingee, and J. W. Chastain

A critical-assembly research program is being conducted for Advanced Technology Laboratories to aid in the development of the Variable-Moderator Reactor concept.

Difficulties have been encountered in fabricating lutetium foils. A fourth core configuration was constructed, and testing was initiated until these foils can be obtained to measure the neutron spectral index. The fourth core configuration is composed of 37 fuel elements, with 61 pins arranged in a hexagonal array on a 0.703-in. pitch, but with a 6.34-in. element pitch. Disadvantage factors, resonance absorption in uranium-238, and fast-fission effect in uranium-238 and uranium-235 were measured for both water and furfuryl alcohol coolant. Reactivity data were also obtained.

Lutetium foil was fabricated during the month and preparations were made for its use in the third core configuration (fuel elements as described above, but with a 5.95-in. triangular element pitch) for thermal-neutron energy-spectrum measurements. Obtaining these spectrum measurements will conclude the major portion of the present critical-assembly program. Preparation of the final report is expected to begin during September.

URANIUM CARBIDE AS A REACTOR FUEL (AI)

E. L. Foster

Atomics International has been supporting research on the irradiation and evaluation of UC as a fuel material for operation at high temperature. In this research, six capsules, each containing two uranium monocarbide specimens 3/8 in. in diameter and about 2 in. long, were irradiated to burnups of from 1400 to 25,000 MWD/T.

In the examination of specimens in the sixth and last capsule, the general appearance of the UC was good, indicating that only slight surface or volume changes had occurred. The detail examination of areas on the surface and of areas adjacent to the defective thermocouple is in progress.

Based on dosimetry results, burnups of from 12,500 to 25,400 MWD/T have been calculated.

Development of Techniques for Preparing Cast Shapes
of UC in Large Quantities

Work on this program has been recessed temporarily.

Postirradiation Examination of Uranium Monocarbide

A. W. Hare and R. F. Dickerson

Six irradiation capsules, each of which contained two cylindrical specimens of uranium carbide 3/8 in. in diameter and about 2 in. long, were irradiated in the Materials Testing Reactor. The burnups ranged from 1400 MWD/T of uranium to an estimated maximum of about 25,000 MWD/T of uranium. Core and surface temperatures ranged from 880 to 1600 F and 540 to 915 F, respectively.

The irradiation data on the first five capsules have already been reported as has the initial postirradiation data on the last capsule. The last capsule contained specimens of nominal uranium-5 w/o carbon alloy and was irradiated to a burnup of about 25,000 MWD/T of uranium.

A metallographic examination of a section of the top specimen, adjacent to the thermocouple hole, is now in progress to confirm the results of the examination which was performed on a section of the top specimen below the thermocouple hole. In this initial examination the depletion of UC₂ evident in specimens in two of the other capsules was not observed. Also in progress is a metallographic examination of the failed thermocouple tip.

The spiral dosimeter wire which was wrapped around the stainless steel basket which contained the test specimens was analyzed to determine the total exposure. The burnup results are listed in Table O-1.

It has been found that, generally, the burnup values based on dosimetry results are usually low as compared with values obtained by radiochemical isotopic analyses for burnup. Unfortunately, the radiochemical and isotopic analyses for this capsule have not yet been completed. However, they will be available and reported next month.

TABLE O-1. PRELIMINARY BURNUP DATA FOR CAPSULE BMI-23-4

Dosimeter Wire	Based on Bradley Lewis Curve ^(a)			Based on Taraba-Payne Curve ^(c)		
	Integrated Effective Flux, 10 ¹³ nvt	Uranium Burnup		Integrated Effective Flux, 10 ¹³ nvt	Uranium Burnup	
		a/o	MWD/T ^(b)		a/o	MWD/T ^(b)
Section 1	1.31	26.5	16,800	1.95	37	23,400
Section 2	0.93	19.7	12,500	1.39	28	17,700
Section 3	1.43	28.5	18,100	2.14	40	25,400
Section 4	1.35	27.4	17,400	2.02	38	24,100

(a) Based on Y (perturbation factor of Bradley Lewis) = 0.47.

(b) Based on 195 Mev per fission and 2200-lb ton.

(c) Based on Y (perturbation factor of Taraba-Payne) = 0.7.

PEBBLE-BED REACTOR MATERIALS (S&P)

W. S. Diethorn

In an assistance program to Sanderson & Porter, Battelle is evaluating spherical fueled-graphite fuel elements for the helium-cooled Pebble-Bed Reactor. The fission-product-release problem is receiving the chief emphasis in the fuel-element development program. Good fission-product retention at temperatures up to 2000 F is required. Two approaches to the problem have been studied over the last 18 months. One approach is to coat the spheres with a dense ceramic coating. A silicon carbide coating is an excellent fission-product barrier, but when a single crack in the coating occurs fission-gas release is very high. A second approach receiving major attention in recent months is to coat the individual fuel particles. Carbon coatings on UC_2 and Al_2O_3 coatings on UO_2 are being investigated.

The fabrication of carbon-coated natural UC_2 particles has been terminated pending the evaluation of a graphite sphere fueled with these coated particles. Fabrication of enriched material will begin when this sphere is proven to be satisfactory for a low-flux capsule experiment. Several small batches of Al_2O_3 -coated UO_2 particles are being prepared for future evaluation in high-flux capsule experiments. These latter capsule experiments will supplement the SP-5 sweep-capsule experiment now being conducted with fully enriched Al_2O_3 -coated UO_2 particles dispersed in a graphite sphere. The Al_2O_3 coatings are showing the first evidence of fission-gas leakage. The rate of release is increasing slowly with in-pile time.

FABRICATION DEVELOPMENT OF COATED FUEL PARTICLESCarbon-Coated UO_2

A. C. Secrest, J. H. Oxley, and J. M. Blocher

Fabrication of carbon-coated uranium carbide has been terminated. A graphite sphere containing natural 149 to 250- μUC_2 coated with 80 μ of carbon will be evaluated in out-of-pile tests before fabrication of material is resumed. It is planned to irradiate this sphere in a low-flux sweep capsule in a study of the effect of temperature on fission-gas release.

 Al_2O_3 -Coated UO_2

M. Browning and J. M. Blocher

Several batches of enriched Al_2O_3 -coated UO_2 particles are being prepared for irradiation evaluation in the next few months. Laboratory evaluations consist of air-oxidation and thermal-cycling tests, metallography, and determination of uranium contamination in the coating. A low rate of effort in the fabrication and laboratory evaluation is expected during the next few months.

IN-PILE-CAPSULE EXPERIMENTS

D. Stahl, G. E. Raines, R. J. Burian, and W. H. Goldthwaite

In this phase of the program, candidate fuel elements for the Pebble-Bed Reactor are irradiated in low-flux and high-flux sweep capsules. Fission-gas release from natural fueled-graphite spheres is studied at temperatures up to 1900 F in the low-flux capsules. The effect of burnup on fission-gas release is studied at a single temperature (~ 1500 F) with fully enriched spheres in the high-flux capsules.

SP-5

Sweep-Capsule SP-5 contains four static specimens and two sweep specimens. Helium is passed over the sweep specimens during irradiation and analyzed for krypton-85 m, krypton-87, krypton-88, xenon-133, and xenon-135. The specimens are described in Table P-1.

TABLE P-1. DESCRIPTION OF SP-5 SPECIMENS

Specimen	Type	Fuel	Coating ^(a)		Capsule Compartment
		Particle Diameter, μ	Type	Thickness	
FA-22, No. 470	UO ₂	100-150	Al ₂ O ₃	40 μ	Static
FA-20, No. 338	UC ₂	100-150	Carbon	2 mils	Static
FA-22, No. 471	UO ₂	100-150	Al ₂ O ₃	40 μ	Sweep
FA-23, No. E8-7	UC	100-150	SiC	8 mils	Sweep
FA-23, No. E8-12	UC	100-150	SiC	10 mils	Static
FA-20, No. 345	UC ₂	100-150	Carbon	2 mils	Static

(a) Sphere coatings are expressed in mils.

No further sampling of sweep helium from the leaky FA-23 sphere is planned.

Specimen FA-22, No. 471, containing 500,000 particles of fully enriched UO₂ each coated with 40 μ of Al₂O₃ was operated with helium flow during August. Sphere center and surface temperatures are running at 1400 and 1250 F, about 100 F lower than last month. As of August 31, this sphere has been irradiated 109 days for an estimated uranium-235 fission burnup of 4.5 a/o. Two gas samples were collected during August (Table P-2).

The R/B values in Table P-2 are about ten times higher than the R/B values reported in BMI-1455 for Samples 10, 11, and 12. The coatings are leaking, but it is not clear whether this leakage is the result of occasional failure of individual coatings or a gradual change in coating structure leading to enhanced diffusion rates for the fission gases. The R/B values depend strongly on the half-life of the fission-gas species. The

R/B for xenon-133 (half-life = 5.2 days) is about 1000 times higher than the R/B for krypton-87 (half-life = 78 min). Little change in the relationship between R/B and half-life has occurred during the entire in-pile period.

TABLE P-2. FISSION-GAS RELEASE FROM FA-22, NO. 471

Gas-Sample Collection Date	Sample	R/B(a)				
		Krypton-85 m	Krypton-87	Krypton-88	Xenon-133	Xenon-135
8/3/60	13	8.2×10^{-6}	2.9×10^{-7}	1.0×10^{-6}	1.2×10^{-3}	1.5×10^{-6}
8/18/60	14	1.6×10^{-5}	1.1×10^{-6}	3.2×10^{-6}	1.1×10^{-3}	4.2×10^{-6}

(a) R = rate of release from the sphere, atoms per sec.
B = rate of production in the sphere, atoms per sec.

SPF-3

The low-flux sweep-capsule experiment, SPF-3, was resumed in August. This capsule contains natural Al_2O_3 -coated UO_2 in a graphite sphere. Several gas samples will be collected over the temperature range 300 to 1900 F to determine the effect of temperature on fission-gas release. Low-temperature results will be available next month.

TANTALUM AND TANTALUM ALLOYS FOR LAMPRE APPLICATIONS (LASL)

J. H. Stang

Two investigations dealing with tantalum and binary alloys of tantalum are being carried out for Los Alamos Scientific Laboratory in connection with the LAMPRE program. In the first study, tantalum specimens, containing selected elements as alloying additions, are being prepared, studied for certain metallurgical characteristics, and forwarded to LASL for plutonium-fuel-mixture corrosion exposures. Progress in this program was impeded during August because of a failure of the furnace used to perform the demanding annealing operations that form a part of the specimen-preparation sequence.

The second study has the objective of developing information that can aid LASL in interpreting the corrosion behavior of tantalum subject to plutonium-fuel-mixture corrosion. During August, limits of solid solubility of nitrogen and oxygen in tantalum annealed at 1000 C and quenched were tentatively established. Corresponding data for tantalum annealed at 1500 C have been reported previously in this section.

Development of Container Materials for LAMPRE Applications

D. C. Drennan, C. J. Slunder, J. G. Dunleavy, and A. M. Hall

A breakdown of a high-vacuum furnace employed routinely in this program for the annealing of tantalum-base alloys prevented the completion of several specimen-preparation operations scheduled for August. The following steps are now being implemented to minimize further delays while the furnace system is being repaired:

- (1) A small vacuum furnace formerly used for tantalum annealing studies is being reassembled and is expected to be in operation early in September.
- (2) As-rolled 30-mil strip of unalloyed tantalum and of alloys containing additions of iron, thorium, and yttrium has been sent to Los Alamos for final annealing.
- (3) As-rolled 150-mil strip of materials designated as Group 2 alloys in BMI-1455 has also been sent to Los Alamos, in this case for intermediate annealing. These materials are to be returned to Battelle for final processing.

During August, the microstructures of several of the Group 2 alloys in the as-cast condition were studied. General observations are as follows:

- (1) One specimen alloyed with a nominal 3 w/o yttrium was segregated and contained an intragranular and intergranular phase. A companion specimen similarly alloyed had a single-phase structure.

- (2) Boron-containing alloys which were completely unfabricable consisted of a complete dendritic structure.
- (3) A tantalum-1 w/o beryllium alloy contained a small, spherical intragranular and intergranular phase.
- (4) Tantalum-1 w/o lanthanum and tantalum-1 w/o scandium materials had a large amount of needlelike phase in the matrix. A depleted zone was present near the grain boundaries and a slight amount of precipitate could be seen in the boundaries proper.
- (5) A tantalum-1 w/o uranium material exhibited several grain boundaries and matrix areas that contained a second phase, although most of the structure was a single phase.
- (6) Single-phase microstructures were found in tantalum-aluminum (0.5 and 1.0 w/o nominal additions), tantalum-cerium (1.0 w/o nominal addition), and tantalum-titanium (1.0 and 3.0 w/o nominal additions).

Precipitate-Phase Identification and Interstitial-Type
Solid Solubility in Tantalum

D. A. Vaughan and C. M. Schwartz

Previous reports have described the solubility limits of the interstitials carbon, nitrogen, and oxygen in tantalum at 1500 C, based on measurements of abrupt changes in room-temperature lattice parameter versus composition and on metallographic examinations. The carbon solubility was found to be of the order of 0.2 a/o at 1500 C, which did not significantly change the lattice parameter of the pure tantalum. In higher tantalum-carbon alloys Ta_2C was identified as the precipitate phase. In the tantalum-nitrogen and tantalum-oxygen system, however, the solubility at 1500 C is considerably greater, 3.6 and approximately 4.0 a/o, respectively. The initial precipitate phases in these systems have not been identified as yet.

During the past month, selected specimens from the tantalum-nitrogen and tantalum-oxygen preparations were annealed at 1000 C and quenched. Lattice-parameter measurements were obtained, metallographic studies were initiated, and tentative solubility limits were obtained from these studies. Solid-solubility limits of approximately 3 a/o at 1000 C were found, tentatively, for both nitrogen and oxygen, from the lattice-parameter data. Although incomplete, metallographic data appear to be essentially in agreement with these results. The above solubility limits are based upon compositional data prior to the 1000 C anneal. Because of some scatter in the results, lattice-parameter determinations on some specimens are being repeated, and the compositions are being checked. It is expected that these and the metallographic studies on the 1000 C annealed specimens will be completed during the next period. Also, specimens will be annealed at 500 C for solubility studies at this temperature.

FLOW STUDIES FOR THE PWR (BAPD)

R. W. Dayton

The two flow studies are still in the apparatus-construction stage. The PWR-2 model is expected to be completed in September, and preliminary testing will be begun.

Reactor Flow Studies

H. R. Hazard and L. J. Flanigan

Studies of air flow in a 0.4-scale model of PWR-2 are being conducted at Battelle to provide design data for the prototype. Work previously reported includes design of the model, installation of plastic model parts in the test facility, and initiation of construction of model core parts.

In August installation of the air-supply piping was completed.

An existing 360-tube water manometer, which will be used to measure pressures throughout the model, was revised and readied for use.

Machining of the aluminum core tubes, which simulate the fuel assemblies in the prototype, was completed. Machining of the thick aluminum core tube sheets is nearly completed.

In September, the model core will be completed and installed in the model, and preliminary flow studies to verify that prototype reference design is satisfactory will be run.

PWR-2 Fuel-Assembly Flow Studies

L. J. Flanigan and H. R. Hazard

Air-flow studies to determine the magnitude of flow maldistribution between channels within seed and blanket fuel assemblies that occur as a result of fuel-assembly entrance geometry are being conducted at Battelle. The studies will be carried out in models approximately 3.5 times the prototype size. Work previously reported includes revision of the piping for the four blowers to provide air at flow rates up to 18 lb per sec at 2 psig and completion of model design work.

In August, the model design was revised because of a change in the prototype design.

Drawings of the revised plastic fuel-assembly entrance sections were sent out for bids, and a purchase order was placed with the low bidder.

Materials have been ordered for the fuel-plate sections of the models and for connecting the models to the air supply.

In September, model construction will be started and the air-supply piping will be completed.

DEVELOPMENT OF MATERIALS FOR THE MGCR AND HTGR (GA)

W. H. Goldthwaite

Research on core materials for the MGCR and HTGR is in progress.

Radiation-effects studies of potential MGCR fuels consisting of UO_2 dispersed in BeO and Al_2O_3 and UC and UC_2 dispersed in graphite are under way. Initial runs of equipment designed to measure compressive creep of BeO in high-temperature helium have been made.

HTGR research is continuing on techniques for coating uranium and thorium carbide powder with carbon by vapor deposition.

DEVELOPMENT AND EVALUATION OF FUEL ELEMENTS FOR THE MGCRRadiation-Effects Study of Potential Fuels

W. E. Murr, N. E. Miller, J. E. Gates, and R. F. Dickerson

Ceramic-type fuels composed of about 20 volume per cent of 30 per cent enriched uranium dioxide in beryllium oxide, 20 volume per cent of 30 per cent enriched uranium monocarbide in graphite, and 20 volume per cent of 30 per cent enriched uranium dicarbide in graphite are being investigated for use as potential fuels for the MGCR. A program designed to study the stability of these fuels over a range of burnups of 1 to 25 uranium-235 a/o at specimen-surface temperatures of about 1500 F is in progress. Four capsules, each containing two specimens of each fuel composition, make up the program. The irradiation of two capsules has been completed. Results of the postirradiation examination of the initial capsule (which was irradiated in the BRR) were reported in BMI-1366 and BMI-1377. The postirradiation examination of six specimens in the second capsule is being performed at the General Atomic laboratories.

The two remaining capsules have been operating satisfactorily in the MTR since the beginning of Cycle 125 (July 24, 1959). Now in their nineteenth cycle of operation, they are operating with only two of the six original thermocouples functioning. Near the end of July, one of the three heaters in Capsule BMI-31-2 failed, causing an over-all temperature drop of about 50 to 80 F in specimen-surface temperatures. Specimen temperatures and heater-power requirements as of the middle of Cycle 144 are shown in Table S-1.

Capsule BMI-31-2 will be discharged from the MTR on September 12, with an estimated specimen fission burnup of about 15-20 uranium-235 a/o. The capsule will be sent to the General Atomic laboratories for opening and subsequent specimen examination. The final capsule of the series, BMI-31-1, will be discharged from the reactor in March, 1961.

TABLE S-1. TEMPERATURE AND HEATER-POWER CONSUMPTION FOR CAPSULES
BMI-31-1 AND BMI-31-2 AT THE MIDDLE OF CYCLE 144

Capsule	Heater-Power Consumption, watts	Thermocouple Reading ^(a) , F					
		No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
BMI-31-1	850	-	-	-	1500 ^(b)	1185	-
BMI-31-2	2230	1420	-	-	-	-	1400 ^(b)

(a) Specimen-surface temperatures are approximately 30 F higher.

(b) Controlling couple.

Experimental Capsule Studies

N. E. Miller, P. M. Steinback, and J. H. Stang

Pellets of BeO containing 30 volume per cent of 12 per cent enriched UO_2 and enclosed in Hastelloy X cans are to be encapsulated in a double-wall instrumented (equipped with thermocouples and auxiliary electrical heaters) capsule for an 18-month irradiation at the MTR. The desired specimen-surface temperature is 1700 F.

During August, Hastelloy X enclosures with desired thermal, creep, and stress-rupture properties were designed, and their fabrication was initiated. Design of the capsule components was nearly completed, and the construction of some components began.

The UO_2 -BeO pellets, which are to be furnished by General Atomic, are scheduled to be delivered to Battelle during September. The pellet-canning and capsule-assembly operations will commence immediately after the pellets are on hand.

Fission-Gas Release From Refractory Fuel Materials

R. J. Burian, J. D. Bray, J. E. Gates, and R. F. Dickerson

The experimental portion of a study to determine the amount of fission gas released from a fuel of 20 volume per cent UO_2 dispersed in Al_2O_3 has been completed. Six fuel pellets, 1/4 in. in diameter by 1/4 in. long, were irradiated in a NaK-filled capsule in the BRR for 1520 hr at an estimated neutron flux of about 10^{13} nv at near 1500 F. The six irradiated pellets were heat treated for 20 hr, and the released fission gases were collected and analyzed. Three of the pellets were heat treated at 1800 F, two at 2300 F, and one at 2600 F.

The heat treatments have been completed, and the data are now being evaluated. Preliminary analyses of the data, based upon presently available information concerning fuel burnup, indicate that about 0.1 per cent of the fission gas formed was released

during the 1800 F test, about 10 per cent was released during the 2300 F test, and about 30 per cent was released during the 2600 F test. A visual inspection of the specimens after the heat treatments did not reveal any change in the appearance of the specimens during the test. However, the weight of the specimen heat treated at 2600 F decreased about 7 per cent. This may have been due to loss of material from the surface of the specimen even though it was not apparent during the visual examination. The weights of the other specimens were essentially unchanged during the heat treatment.

MGCR MODERATOR STUDIES

Compressive Creep of BeO

J. Koretzky, M. C. Brockway, A. K. Smalley, and W. H. Duckworth

A trial run was made during August in the compressive-creep stand described previously. Specimens were solid BeO cylinders 2 in. in diameter and 2 in. long. They were prepared from Brush unadulterated UOX-grade BeO powder by cold pressing and sintering and had bulk densities of about 95 per cent of theoretical.

The specimens were loaded axially at 1000 psi and heated at 1980 F for 137 hr in flowing helium. Measurements (to ± 0.001 in.) of specimen lengths and diameters indicated no measurable changes in dimensions from the tests.

This trial indicated that the equipment performs satisfactorily under the most severe anticipated operating conditions.

This work has now been recessed.

COATINGS OF URANIUM AND THORIUM CARBIDE POWDERS FOR THE HTGR

J. H. Oxley, A. C. Secrest, and J. M. Blocher, Jr.

Work on this project was resumed at the end of August.

DEVELOPMENTAL STUDIES FOR THE SM-1 AND SM-2 (ALCO)

S. J. Paprocki

Studies are being conducted in assistance to Alco Products that are concerned with the development of techniques for the fabrication of an instrumented blocked-channel fuel plate for irradiation in the SM-1 and the development of fuel, absorber, and suppressor materials for the SM-2.

Techniques have been developed for the fabrication of subsize instrumented fuel plates consisting of a Zircaloy filler sandwiched between two SM-1 fuel plates of one-half thickness. The fabrication of full-size instrumented plates to be incorporated into standard stationary and control-rod elements has been initiated.

Fabrication specifications have been established for the SM-2 fuel plate. Reference and alternate SM-2 fuel test specimens have been irradiated in three capsules in the MTR and one capsule in the ETR to burnups ranging from 20 to 45 per cent of the contained uranium-235. Additional specimens contained in capsules are being irradiated in the ETR.

SM-1 PROGRAMProcess Development for the Preparation of Instrumented
Fuel Plates of the SM-1 Type

S. J. Paprocki, E. S. Hodge, G. W. Cunningham,
C. B. Boyer, and E. G. Smith, Jr.

Techniques are being developed for preparing instrumented fuel plates for use in SM-1 type fuel elements for in-pile test evaluation. Two plates will be used in each element. The blocked-channel method of instrumentation will be used where two instrumented plates and one water channel will occupy the same space as three standard fuel plates and two water channels in the standard SM-1 fuel element.

Niobium has been found to be a satisfactory barrier material between the low-cross-section Zircaloy-2 filler containing the thermocouples and the stainless steel cladding to prevent a eutectic reaction at the pressure-bonding temperatures necessary to obtain good stainless self-bonds. A 3-mil-thick barrier of niobium has been found sufficient to prevent reaction between the Zircaloy and the cladding cover plates. Side and end barriers of 20-mil niobium are being used to prevent the filler material from coming into contact with the stainless steel frame during bonding at 2100 F for 3 hr at 10,000 psi.

Core material has been received from Sylcor, and fuel sheets are presently being fabricated for the full-scale stationary and control-rod instrumented assemblies.

SM-2 PROGRAM

Materials Development

S. J. Paprocki, D. L. Keller, G. W. Cunningham,
A. K. Foulds, and D. E. Lozier

Techniques have been developed and specifications have been written for the SM-2 fuel plate, which contains a 0.030-in. -thick core of 26 w/o UO_2 and 1.2 w/o ZrB_2 dispersed in a Type 347 prealloyed stainless powder matrix and is clad with a 0.005-in. Type 347 cladding. Fuel plates are being fabricated for use in critical-assembly and irradiation tests.

Ten reference SM-2 fuel plates containing fully enriched UO_2 are being fabricated. Picture frames and cover plates are being machined from a special heat of Type 347 stainless which meets specifications of less than 0.025 w/o cobalt and 0.01 w/o tantalum. Fuel-core dispersions and suppressor-section cores have been blended and pressed, and six of the cores have been sintered and recoined.

Radiation-Stability Studies of Reference and Alternate SM-2 Fuels

W. E. Murr, J. H. Stang, J. F. Lagedrost, and J. E. Gates

The irradiation of four capsules with nominal specimen burnups of about 21 to 46 uranium a/o has been completed. The specimens have been examined at the Battelle Hot-Cell Facility. A topical report discussing results of these irradiations is being prepared. Four of the six remaining capsules are being irradiated in reflector positions of the ETR with the remaining two scheduled for insertion for Cycle 32.

Encapsulation and Irradiation

At present, four of the six remaining capsules in this program are in-pile, and the other two are scheduled for immediate insertion. The ETR was inoperative during nearly all of August to accommodate a loop installation.

Evaluation of Irradiated Specimens

Examination of the specimens irradiated in the first four capsules, BMI-32-1, BMI-32-2, BMI-32-3, and BMI-32-4, has been completed, and results of the examination were reported in BMI-1442 and BMI-1448. During August, results of isotopic analyses of 12 representative specimens from the four capsules were obtained from CPP and are listed in Table T-1.

A topical report discussing performance of specimens irradiated in these four capsules is being prepared.

TABLE T-1. ISOTOPIC BURNUP ANALYSES FOR SPECIMENS IRRADIATED IN CAPSULES
BMI-32-1, BMI-32-2, BMI-32-3, AND BMI-32-4

Capsule	Specimen	Reactor and Position	Uranium-235 Burnup ^(a) , a/o
BMI-32-1	2-7V	MTR L-53	39.1 ± 0.3
	3-5	MTR L-53	44.5 ± 0.3
	4-5	MTR L-53	43.9 ± 0.3
	8-5V	MTR L-53	39.9 ± 0.3
BMI-32-2	2-2-2	MTR L-56	45.4 ± 0.3
	5-5V	MTR L-56	44.3 ± 0.3
	10-4	MTR L-56	36.4 ± 0.3
BMI-32-3	14-2-3	MTR L-58	37.4 ± 0.3
	13-3	MTR L-58	35.1 ± 0.3
	10-2	MTR L-58	35.1 ± 0.3
BMI-32-4	4-11	ETR O-7	20.7 ± 0.3
	1-12	ETR O-7	39.5 ± 0.3

(a) Limits are expressed on the 95 per cent confidence level.

Fuel-Element Thermodynamics

J. J. Ward, C. A. Alexander, A. W. Lemmon, Jr.,
and R. B. Filbert, Jr.

Work was continued at a minimum rate during August on the study of the thermodynamics of postulated reactions between fuel-element materials and between fuel-element materials and surrounding atmospheres. The reactions of primary interest are those which would lead to boron losses during fabrication of the current SM-2 elements. Possible reactions of alternate materials will be considered later.

Work is progressing on examination of chemical reactions favorable to losses of boron and on study of diffusion rates of boron in the solid state. Physical properties of boron in the solid gamma iron and steel were found in the literature* and plotted. Smoothed values are given below of solubility and diffusivity.

<u>Temperature, F</u>	<u>Boron Diffusivity, cm² per sec</u>	<u>Boron Solubility, w/o</u>
2050	8.8 x 10 ⁻⁷	0.024
2100	10.2 x 10 ⁻⁷	0.032
2200	13.2 x 10 ⁻⁷	0.048

It was found that increasing the amount of carbon in the steel had no effect on the diffusivity.

*Busby, P. E., Warga, N. E., and Wells, C., Jr. J. Metals, 197, 1463 (1953).

The data show that in going from 2050 to 2200 F, the diffusivity increases by 50 per cent. This would increase the losses of boron by 50 per cent, if mass diffusion is the controlling mechanism. This assumes a constant driving force or no change in activity of the boron in the solid.

As mentioned last month in BMI-1455, the following is a postulated mechanism where elemental boron diffuses from the surface of ZrB_2 imbedded in the stainless steel core:

At 2100 F the activity of boron was calculated to be 3.2×10^{-4} , referred to as mole fraction at standard state. In going from 2050 to 2200 F, the activity or driving force would increase from 2.7×10^{-4} to 4.4×10^{-4} atm which would increase the diffusion rate by 63 per cent. Hence, the over-all net effect of raising the temperature from 2050 to 2200 F is to increase the rate of diffusion by 145 per cent if the above mechanism is postulated.

The solubility of boron in the steel at 2100 F is 0.032 w/o which is a mole fraction of 1.65×10^{-3} . This is considerably greater than the activity of boron of 3.2×10^{-4} which was calculated for the postulated reaction, and saturation is not exceeded.

In the future study, emphasis will be placed on diffusion rates and solution effects.

GAS-COOLED REACTOR PROGRAM (AGN)

D. L. Keller

Studies for Aerojet-General Nucleonics (AGN) and the Army Reactor Branch (ARM) are reported in this section. A large part of these studies are directed to support the ML-1 reactor fuel-element development program in which the reference fuel consists of both solid UO_2 and BeO-UO_2 pellets clad with Hastelloy X tubing.

Examinations of the sinterability of commercial BeO powder and 60 w/o UO_2 have continued. Additional lots of UOX-BeO powder have shown a possible relationship between sinterability and purity. Future studies will be concerned with simplifying the batch processing of BeO-UO_2 mixtures.

Further progress is reported on the encapsulation of both UO_2 and Be-UO_2 specimens clad with Hastelloy X to be irradiated at the BRR and MTR, respectively.

In a program of alloy modification of Hastelloy X for improving its strength, ductility, and corrosion resistance, it was noted that variations in the molybdenum content within the range specified commercially accounted for a 20 per cent difference in strength at 1750 F. Preliminary examinations of the surface of Hastelloy X corrosion specimens indicate that chromium and manganese are increasing while iron and nickel are decreasing in the surface oxide with increasing exposure in nitrogen-1/2 volume per cent oxygen.

Studies with the ML-1 critical assembly during the past month included calibration of two scissors-acting control blades, measurements of the flux and power distortion caused by the scissors blades, and measurements of the temperature coefficient of reactivity of the ML-1 reactor at temperatures between 25 and 73.5 C.

Study of Processing Variables in the
Fabrication of BeO-UO_2 Ceramics

H. D. Sheets, A. K. Smalley, and W. H. Duckworth

This program is aimed at refining the process for fabricating $\text{UO}_2\text{-BeO}$ ceramic fuel pellets by cold pressing and sintering. The suitability of various commercially available high-purity BeO powders for the preparation of pellets by this method currently is being investigated.

Previously, it was found necessary to sinter pellets containing 40 w/o UOX BeO and 60 w/o UO_2 at 3200 F to obtain sintered densities in excess of 95 per cent of theoretical. In addition, the coarse BeO grains in this material resulted in an observable non-uniformity of UO_2 concentration within a pellet. Addition of MgO or ball milling of the BeO powder was found to permit attainment of 95 per cent of theoretical density by sintering at 3000 F. Ball milling had the additional advantage of improving the uniformity of the dispersion.

During this period, two additional lots of Brush UOX BeO were evaluated. The coarse particles in these lots were removed by the vendor, by wet screening the powder

through a 100-mesh (Lot 100-W-24) or a 200-mesh (Lot 200-W-89) sieve. Spectrochemical analyses of the two lots are shown in Table U-1. The results of sintering trials are shown in Table U-2. As with the previous lot, sintering at 3200 F was necessary to obtain pellets having densities in excess of 95 per cent of theoretical.

TABLE U-1. SPECTROCHEMICAL ANALYSES OF BeO POWDERS

Element	Amount Present, ppm	
	Lot 100-W-24	Lot 200-W-89
Silicon	50	20
Manganese	2	<2
Magnesium	35	15
Sodium	25	20
Iron	30	20
Copper	25	15
Lithium	2	<1
Nickel	5	<5
Potassium	10	10
Calcium	10	<10
Aluminum	50	30

TABLE U-2. EFFECT OF PROCESSING CONDITIONS^(a) ON SINTERED BULK DENSITY AND SHRINKAGE OF BeO-UO₂ CERAMICS

Batch	BeO		UO ₂ Amount, w/o	Forming Pressure, psi	As-Formed Density, g per cm ³	Sintering		Sintered Density ^(b)		Diametral Shrinkage ^(c) , per cent
	Lot Designation	Amount, w/o				Temperature, F	Time, hr	G per Cm ³	Per Cent of Theoretical	
74-2	100-W-24	40	60	20,000	2.74	2800	2	4.58	86.6	15.7
				40,000	2.97	2800	2	4.83	91.3	15.0
				20,000	2.74	3000	2	4.94	93.4	17.8
				40,000	2.97	3000	2	5.06	95.7	16.3
				20,000	2.74	3200	1	5.10	96.4	18.8
				40,000	2.97	3200	1	5.16	97.5	16.9
74-3	100-W-24	30	70	20,000	3.12	2800	2	5.23	83.0	15.6
				40,000	3.38	2800	2	5.50	87.3	15.0
				20,000	3.12	3000	2	5.65	89.7	17.9
				40,000	3.38	3000	2	5.81	92.2	16.5
				20,000	3.12	3200	1	5.82	92.4	18.9
				40,000	3.38	3200	1	5.90	93.7	17.1
75-2	200-W-89	40	60	20,000	2.78	2800	2	4.35	82.2	12.8
				40,000	3.01	2800	2	4.58	86.6	12.2
				20,000	2.78	3000	2	4.68	88.5	15.0
				40,000	3.01	3000	2	4.92	93.0	14.2
				20,000	2.78	3200	1	4.97	93.9	16.7
				40,000	3.01	3200	1	5.10	96.4	15.3
75-3	200-W-89	30	70	20,000	3.16	2800	2	4.86	77.1	12.8
				40,000	3.42	2800	2	5.20	83.6	12.2
				20,000	3.16	3000	2	5.56	88.2	15.2
				40,000	3.42	3000	2	5.56	88.2	14.5
				20,000	3.16	3200	1	5.64	89.6	17.1
				40,000	3.42	3200	1	5.81	92.2	15.8

(a) Specimens were pressed in a steel die, and sintered in flowing hydrogen, as indicated.

(b) Data are average for two specimens. Density was measured by xylene adsorption-immersion. Theoretical density of 60 w/o UO₂-40 w/o BeO was taken as 5.29 g per cm³. Theoretical density of 70 w/o UO₂-30 w/o BeO was taken as 6.30 g per cm³.

(c) Average of four specimens. Sintered diameters of the specimens within a group agreed within 0.001 in.

It is interesting to note that pellets containing the finer, and somewhat purer, BeO did not sinter to so high a density as those containing the coarser BeO.

Future work will include the evaluation of additional samples of BeO powder. Effort will be made to simplify batch preparation.

Effects of Irradiation

J. H. Saling, D. G. Freas, J. E. Gates,
and R. F. Dickerson

The study of the radiation stability of various potential ML-1 fuel materials is progressing. Materials presently being evaluated include dispersions of UO_2 in BeO and bulk UO_2 clad in Hastelloy X. The irradiation and postirradiation examination of one capsule, GCRE-BRR-2, containing BeO pellets fueled with 59 w/o fully enriched UO_2 inside Hastelloy X tubes have been completed and the results included in earlier progress reports. A topical report on this irradiation program is presently being prepared. The irradiation and postirradiation examination of a subassembly, No. 1B-2T-2, containing 19 pins of bulk UO_2 contained in Hastelloy X tubes have been completed and the results reported. The only research remaining to be accomplished in this program is completion of fuel-burnup analyses and the preparation of a topical report.

Encapsulation Studies

W. J. Zielenbach, J. F. Lagedrost,
and J. H. Stang

Instrumented irradiation capsules are being designed and fabricated to study the effects of irradiation on Hastelloy X-clad compacts of highly enriched UO_2 and UO_2 in designated matrix materials. Specimen-cladding temperatures are to be maintained at $1725 \text{ F} \pm 25 \text{ F}$ for periods of time relevant to the ML-1 core-development program. The capsules involved are furnished with thermocouples and auxiliary heaters and are based on the conventional double-wall design with NaK-immersed specimens sealed in an inner shell separated from the outer shell by a narrow helium annulus.

A capsule containing 70 w/o and 80 w/o highly enriched UO_2 in a BeO matrix was assembled during this report period and shipped to the MTR. According to the present schedule, this capsule will be inserted during the shutdown starting September 12. While the reported unperturbed thermal flux in the assigned position is about 10 per cent higher than the capsule design flux ($0.8 \times 10^{14} \text{ nv}$), the difference is within the uncertainty limits associated with the experiment. Because of the current backlog of instrumented capsules at the MTR, a delay of several reactor cycles would have been necessary to obtain a space having a quoted flux closer to that initially specified. The target peak burnup for specimens in this capsule is 9.2 total a/o of the uranium-235; this will require an exposure of 9 reactor cycles.

The assembly of a capsule to be irradiated in the Battelle Research Reactor is nearing completion; the fuel for this irradiation is 100 per cent highly enriched UO_2 . This capsule, which will be inserted during September, will operate for 6 cycles to

achieve a total burnup of 2 a/o. It was necessary that the system be provided with an axially finned aluminum jacket shrunk on the outside of the water-contacting outer shell to obtain a heat-transfer surface large enough to preclude boiling.

The parts for two additional capsules have been fabricated and are complete except for final machining to several dimensions which will be decided upon when the specimen compositions and heat-generation rates are selected.

Alloy Modification of Hastelloy X

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

Fuel elements of the ML-1 are to be clad with Hastelloy X. A program is currently in progress to determine if the ductility of the base alloy, which decreases after long-term exposure to a nitrogen-1/2 volume per cent oxygen atmosphere, and the oxidation resistance of the alloy can be increased by modifications of the composition.

For this purpose, modified Hastelloy X alloys containing niobium and yttrium additions, and alloys in which chromium, molybdenum, nickel, and tungsten contents were varied, have been prepared. The effect of these additions and of variations in composition on tensile properties and on oxidation resistance are being studied.

Corrosion tests at 1750 F in nitrogen-1/2 volume per cent oxygen atmospheres are being conducted at AGN. Variations in depth of oxidation penetration as a result of changes in alloy composition have been noted. However, the exposure at 1750 F is accompanied by precipitation of a second phase in both the Hastelloy X and in the modified alloy structures. This phase has been tentatively identified as a complex carbide although from the amounts present it appears that it may be a carbonitride. An understanding of the role of this compound in the loss of ductility and in the mechanism of oxidation is being sought.

Examinations by X-ray fluorescence and X-ray diffraction of Hastelloy X specimens exposed to the nitrogen-1/2 volume per cent oxygen atmosphere for periods of 1000, 2500, and 5000 hr are nearing completion. Preliminary data indicate that the chromium and manganese contents of the surface oxide are increasing with increasing exposure time while the iron and nickel contents of the oxide are decreasing.

Tensile tests of the alloys prepared are nearly completed; data have been shown in BMI-1442 and BMI-1448. Additional data are shown in Tables U-3 and U-4. The data in Table U-4 are for alloys prepared to determine the effects of variations in composition within those specified for Hastelloy X and the effect of variations in molybdenum and tungsten contents. It appears that variations of iron and nickel within the nominal Hastelloy X composition range have little if any effect on tensile properties. However, a definite trend of increased strength with increasing molybdenum content can be observed. Differences of 20 per cent in strength at 1750 F apparently can be obtained by varying the molybdenum content between 8 and 10 w/o. A 3 w/o tungsten addition results in a modest increase in room-temperature tensile strength, while a 10 w/o addition results in a very significant increase in room-temperature strength.

TABLE U-3. TENSILE PROPERTIES OF MODIFIED HASTELLOY X ALLOYS

Alloy	Nominal Selected Alloying Addition ^(a) , w/o	Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation in 1 In., per cent
63	21 Cr-1.6 Y	Room	129,000	146,000	7
		1000	115,000	118,000	1
		1750	13,600	14,800	58
		1950	2,500	4,650	93
67	21 Cr-2 Nb	Room	138,000	145,000	2
		1000	113,000	129,000	4
		1750	18,300	20,100	83
69	21 Cr-1.6 Y-2 Nb	Room	141,000	153,000	2
		1000	126,000	141,000	3
		1750	9,340	11,200	102
		1950	1,590	3,240	158

(a) Alloy contains nominally 10 w/o molybdenum, 0.7 w/o tungsten, 1.0 w/o manganese, 1.0 w/o silicon, 0.001 to 0.005 w/o carbon, 21 w/o chromium, 45 w/o nickel, and the balance iron.

TABLE U-4. EFFECT OF COMPOSITIONAL VARIATIONS ON THE TENSILE PROPERTIES OF MODIFIED HASTELLOY X ALLOYS

Alloy	Nominal Selected Alloying Addition ^(a) , w/o	Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation in 1 In., per cent
80	45 Ni-21 Cr-9 Mo	Room	109,000	125,000	13
		1750	16,900	17,300	32
82	45 Ni-21 Cr-10 Mo	Room	115,000	131,000	14
		1750	18,800	19,500	32
83	45 Ni-21 Cr-8 Mo	Room	119,000	133,000	10
		1750	15,200	15,300	42
84	46 Ni-22 Cr-9 Mo	Room	120,000	135,000	8
		1750	16,400	16,700	42
85	44 Ni-20 Cr-9 Mo	Room	109,000	125,000	13
		1750	16,400	18,200	34
86	45 Ni-21 Cr-10 Mo-3 W	Room	127,000	143,000	7
		1750	18,000	18,900	38
87	45 Ni-21 Cr-10 Mo-10 W	Room	160,000	181,000	6
		1750	(b)	--	--
90	45 Ni-21 Cr-10 Mo-1.0 Si	Room	113,000	131,000	12
		1750	12,500	15,300	50
91	45 Ni-21 Cr-5 Mo	Room	109,000	123,000	10
		1750	11,200	11,500	40

(a) Alloy contains nominally 0.7 w/o tungsten, 1.0 w/o manganese, 0.5 w/o silicon, 0.001 to 0.005 w/o carbon with balance iron and constituents listed above.

(b) Not completed.

ML-1 Critical-Assembly Experiments

J. W. Ray, R. G. Matthews, W. S. Hagan,
D. A. Dingee, and J. W. Chastain

Further experiments relating to the ML-1 reactor were conducted with the ML-1 critical assembly. Experiments during the month of August included the following:

- (1) Calibration of the reference scissors-acting control-blade set constructed of silver-15 w/o indium-5 w/o cadmium alloy
- (2) Calibration of a Type 302 stainless steel scissors-acting control blade
- (3) Measurement of the shadowing effect between adjacent scissors-blade sets
- (4) Measurement of the flux and power distortion caused by the presence of a set of scissors blades in the core
- (5) Measurement of the temperature coefficient of reactivity of the ML-1 reactor at temperatures between 25 and 73.5 C.

The worth of the reference scissors blades was found to be -1.78 per cent $\Delta k/k$ for an angle of separation between the blades of 15 deg. Further calibration indicated that 15 deg was the angle of maximum worth. When opened to the "fully withdrawn" position, approximately 170 deg between blades, the set had a worth of -0.10 per cent $\Delta k/k$.

The worth of the stainless steel blade set was found to be -0.59 per cent $\Delta k/k$ at 15 deg, and zero in the fully withdrawn position.

Shadow studies determined how the worth of a set of scissor blades was affected by the presence of one and two other sets of similar blades. Three identical sets of stainless steel scissor blades, each covered with 20 mils of cadmium, were used for this study. The blades used in these studies were not as wide as either the reference blades or the Type 302 stainless steel blades mentioned above. One set of these blades in the core alone was found to have a reactivity worth of -1.05 per cent $\Delta k/k$. With another set mounted adjacent to it, its worth decreased to -0.97 per cent $\Delta k/k$; with a set mounted on each side, the worth of the initial set decreased to -0.90 per cent $\Delta k/k$.

Data from the flux- and power-distortion measurements are still being processed. Preliminary results show a sizable flux depression immediately adjacent to the blade; at a distance of one fuel element away from the blade this depression almost disappears.

Temperature-coefficient measurements were made at five different temperatures beginning at 25 C and going up to 73.5 C. At 25 C, the coefficient was $+1.60 \times 10^{-4}$ $\Delta k/k$ per C and at 73.5 C, the coefficient was approximately $+0.60 \times 10^{-4}$ $\Delta k/k$ per C. Intermediate data points fell approximately on a straight line between these two values.

Experiments on a flooded core will begin in September. In this core, the tubes containing the fuel elements will be flooded with deionized water. Experiments will be

done to determine the critical loading of such a core, to measure intracell power and flux distributions in two core positions, and to calibrate the reactivity worth of six sets of scissors-acting control blades in this core.

BRR Loop Program

W. G. Rieder, S. J. Basham, and
W. H. Goldthwaite

Operation of the 1B-2T-1 fuel subassembly in the recirculating gas loop located at the Battelle Research Reactor has continued. Irradiation of the specimen at an average-exit-gas temperature of 1300 F was initiated. An inlet-gas temperature of 610 F was maintained at a specimen power generation of 37 kw. Automatic flow control was used to maintain the temperatures at the set levels. Coolant-flow rates of about 660 lb per hr were required to maintain the specimen at equilibrium conditions.

Recent changes in the core configuration were reflected in changes in the operating characteristics of the specimen. Shortly after startup of the most recent reactor cycle the specimen power generation was 45 kw. However, it dropped to 37 kw during the first 24 hr of operation of the reactor. Investigation revealed that the loop specimen power was apparently affected by changes in control-rod position during the initial period of reactor operation. Possible changes in core configuration or control-rod operation are being examined. A specimen power generation of 55 kw would be desirable. This would be similar to the peak operating level of a reference fuel subassembly in the reactor experiment (ML-1) for which the 1B series of fuel elements was developed. However, it appears unlikely that power-generation levels of much over 45 kw can be achieved with any changes in core configuration which will not adversely affect other irradiation experiments. The 45-kw operating level is close to that of a typical prototype fuel subassembly.

The irradiation of the 1B-2T-1 was interrupted for two 12-day reactor cycles as a result of a coolant-gas leak in the pressure vessel of the preheater unit. The loop, which is suspended from a movable bridge, was secured in the back pool of the reactor (away from the core). During the reactor shutdown after the preheater failure, a cask was positioned around the test section. The preheater was removed from the pool, disassembled, and repaired. The unit was reinstalled at the following reactor shutdown.

Although the system has operated in-pile for over 5100 hr and two specimens have ruptured while in the loop, the only requirement which prevented the repair from being described as a routine operation was the placement of the cask around the test section.

CORROSION OF THORIUM AND URANIUM
UNDER STORAGE CONDITIONS (LIVERMORE)

P. D. Miller

A nickel-plating procedure developed at LRL for thorium has shown superior results in preliminary evaluation tests, and specimens coated by this procedure will be incorporated in an exposure program to be started soon.

Metallographic studies are being completed on specimens of bare thorium, uranium, and uranium-10 w/o molybdenum alloy which have been exposed to laboratory environments.

Electrodeposited Coatings for Thorium (SC)

J. G. Beach, E. S. Koglekowski, and C. L. Faust

Several modifications of the procedure for prefinishing and activating thorium for nickel plating were appraised. The screening evaluation was based on the protective value of nickel plated on thorium when exposed to water-saturated air at 200 F.

Samples of nickel-plated (average thickness, 2 mils) thorium prepared by the LRL procedure showed little corrosion after over 400 hr, whereas samples prepared using the BMI procedure generally failed by pit-type corrosion within 48 hr of exposure.

Nickel-plated samples will be prepared using the LRL procedure for exposure at four sites. Although these samples may not represent the ultimate in plated coating, the exposure results should help provide a base line for defining the improvement needed for protecting thorium.

Corrosion Evaluation (UCRL)

E. F. Stephan, P. D. Miller, and F. W. Fink

Metallographic studies are being made of the extent of the corrosion on specimens of thorium, uranium, and uranium-10 w/o molybdenum after exposure in several laboratory environments. Results will be summarized in a later report.

Preparations are being made for a series of atmospheric corrosion tests to be conducted at Daytona Beach, Florida, and Columbus, Ohio.

GAS-PRESSURE BONDING OF BERYLLIUM- CLAD ELEMENTS (ORNL)

S. J. Paprocki, E. S. Hodge, and J. S. Perrin

A beryllium-clad uranium dioxide fuel element is being considered by Oak Ridge National Laboratory for use as an advanced fuel system for gas-cooled reactors. The fabrication of this fuel system by gas-pressure bonding is being studied. The effect of bonding cycles on the self-bonding of beryllium and the properties of the clad element will be studied.

The program concerned with investigating the self-bonding of beryllium and cladding of compartmented uranium dioxide platelets by pressure bonding is divided into three phases of study. The initial phase will be concerned with bonding parameters required for self-bonding of beryllium as a function of surface preparation, bonding cycles, and heat treatments after bonding. The specimen design in this phase will be a composite sandwich consisting of a beryllium plate between two thinner beryllium cover plates. Specimens will be bonded and subjected to metallographic examination and physical-property tests. The results will be used to determine optimum surface-preparation and optimum pressure-bonding conditions.

Results obtained in the initial phase of study will be used for fabricating experimental flat-plate beryllium-clad compartmented uranium dioxide fuel elements. The bonded fuel-element assemblies will be nondestructively evaluated by X-radiography and destructively evaluated by metallographic examination, chisel tests, and gas-pressure burst tests.

Irradiation-test specimens will be prepared in the final phase of this study if the results of the studies demonstrate that the gas-pressure-bonding process is satisfactory and if sufficient lead time is available to schedule the specimens for in-pile testing.

PLAIN BEARING MATERIALS FOR NITRIC ACID-ALUMINA
SLURRY PUMP (IDO)

H. A. Van Dyke, W. A. Glaeser, and C. M. Allen

Bearing and journal materials for a nitric acid-alumina slurry pump are being evaluated in a thrust-bearing system designed to simulate the velocity, load, and minimum lubricant-film thickness anticipated for the slurry-pump guide bearing. The slurry is created during cleanup of off-gas from a fluidized-bed calcination of aluminum nitrate liquid wastes to form granular alumina.

A solid boron carbide thrust runner and solid chromium carbide fixed tapered-land thrust pads were evaluated. Operating time was 100 hr in an aqueous slurry of 40 w/o nitric acid-5 w/o low-temperature alumina at a temperature of 170 F. Wear of the boron carbide runner was negligible, and lapping and grinding scratches were still visible in the wear track. There was some roughening, wear, and microscopic cracking of the chromium carbide pads.

One other material combination is to be evaluated: a stainless steel runner versus graphite pads.

RWD/CRT:all