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REACTOR FUELS AND MATERIALS DEVELOPMENT PROGRAMS
FOR
FUELS AND MATERIALS BRANCH
OF
USAEC DIVISION OF REACTOR DEVELOPMENT AND TECHNOLOGY

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<u>Quarter</u>	<u>Year</u>	<u>Report Number</u>
	<u>1966</u>	
First		BNWL-CC-694
Second		BNWL-CC-761
Third		BNWL-CC-821
Fourth		BNWL-CC-957
	<u>1967</u>	
First		BNWL-435
Second		BNWL-473
Third		BNWL-658
Fourth		BNWL-668
	<u>1968</u>	
First		BNWL-768
Second		BNWL-842
Third		BNWL-919
Fourth		BNWL-971
	<u>1969</u>	
First		BNWL-1058

TABLE OF CONTENTS

SUMMARY.	2.1
NUCLEAR GRAPHITE - R. E. Nightingale	3.1
Inert-Gas-Sensitized Radiolysis Studies -	
G. L. Tingey	3.1
Radiation-Induced Creep in Graphite -	
W. J. Gray	3.2
HTGR GRAPHITE	4.1
The Radiolytic Reaction of Graphite	
with Water Vapor - R. E. Woodley	4.1
NUCLEAR CERAMICS - R. E. Nightingale	5.1
Properties of Molten Fast-Reactor Oxide	
Fuels - R. P. Nelson, J. J. Rasmussen,	
and O. D. Slagle	5.1
Electrical Conductivity of Cerium Oxide -	
J. L. Bates	5.5
Preparation of UO ₂ - 25 wt% PuO ₂ Specimens	
for High-Temperature Creep Studies -	
O. D. Slagle and J. A. Basmajian	5.10
Thermal Diffusivity of (U,Pu)O ₂ -	
R. L. Gibby	5.24
Determination of Oxygen/Metal Ratios	
for Oxide Fuels - R. E. Woodley	5.27
Characterization of Mixed-Oxide Fuel	
Materials - J. L. Daniel and S. J. Mayhan.	5.29
FAST REACTOR NITRIDES RESEARCH	6.1
NONDESTRUCTIVE TESTING	7.1
METALLIC FUELS	8.1
BASIC SWELLING STUDIES - C. R. Hann.	9.1
Postirradiation Examination - C. R. Hann,	
B. Mastel.	9.1
Mixed Nitrides Fuel Irradiation -	
R. K. Marshall	9.9
ATR GAS LOOP OPERATION AND MAINTENANCE.	10.1
FAST REACTOR DOSIMETRY AND DAMAGE ANALYSIS -	
R. E. Nightingale	11.1

EBR-II Dosimetry Test - J. L. Jackson, W. N. McElroy, J. A. Ulseth, L. S. Kellogg, and F. S. Kirn	11.1
Damage Functions and Data Correlation - W. N. McElroy and C. Z. Serpan, Jr.*	11.4
Computer Simulation of Radiation Damage - D. G. Doran	11.14
Computer Simulation of Irradiation Displacement Cascade Formation - G. E. Russcher	11.15
Computer Experimental Study on Carbon Inclusions in Grain Boundaries in FCC Gamma - Iron - R. E. Dahl, Jr., J. R. Beeler, Jr., and R. D. Bourquin	11.17
IRRADIATION DAMAGE TO REACTOR METALS	12.1
Reactor Metals Research	12.1
Irradiation Facilitates Operation - A. L. Ward.	12.1
Microstructural Modifications to Stain- less Steels and Nickel-Base Alloys - I. S. Levy.	12.3
In-Reactor Measurements of Mechanical Properties - E. R. Gilbert, N. E. Harding.	12.5
Formation of Voids in Iron During High-Temperature Neutron Irradiation - G. L. Kulcinski and B. Mastel	12.8
Small-Angle X-Ray Scattering by Voids in Irradiated Metals - H. E. Kissinger.	12.12
Fast Reactor Supporting Studies	12.15
Damage Analysis - J. L. Straalsund and H. R. Brager	12.15
EBR-II Irradiations - P. K. McDaniel	12.22
Acceptance Testing and Clad Characterization - M. M. Paxton	12.23
Uniaxial Creep-Rupture Studies - A. J. Lovell	12.48
Biaxial Stress-Rupture Studies - R. W. Barker	12.53
FTR Vessel Weldment Studies - A. L. Ward	12.67
LMFBR Fuel and Cladding Information Center - F. R. Shober	12.73

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SUMMARY

NUCLEAR GRAPHITE

The 100-eV yield of the radiolytic reaction of CO_2 and H_2 in a helium carrier decreases with CO_2 and H_2 concentration below 3500 ppm. At 350 °C the $G(\text{CO})$ for the mixture is 1.9 molecules/100 eV at concentrations of CO_2 and H_2 above 3500 ppm. At H_2 and CO_2 concentrations of 300 ppm, the $G(\text{CO})$ is 1.5 and 0.65 molecules/100 eV, respectively.

Design of an in-reactor creep experiment for graphites of interest to the Molten Salt Breeder Reactor are complete. The test is designed to measure in-reactor creep for graphite samples with a constant compressive load. This experiment is scheduled to begin in the ETR in October, 1969.

HTGR GRAPHITE

THE RADIOLYTIC REACTION OF GRAPHITE WITH WATER VAPOR

Measurements of the radiolytic reaction of graphite-water vapor are presently being carried out in a single-pass flow system. Product concentrations are very small, a few tenths of 1 vpm. CO_2 concentrations of the same magnitude in the helium carrier gas interfere with radiolytic yield determinations.

NUCLEAR CERAMICS

PROPERTIES OF MOLTEN FAST-REACTOR OXIDE FUELS

Installation has been completed of the high-temperature vacuum resistance furnace within a plutonium glove box located in the Shielded Materials Facility. This arrangement will be used for measurements of liquid density, viscosity, surface tension, and compressibility on both unirradiated and irradiated mixed-oxide fuels to 3000 °C.

Techniques are described for obtaining the surface tension of liquid oxides by analyzing the shapes of pendant drops and capillary menisci which have been radiographed in closed refractory-metal capsules. Values of the surface tension of liquid Al_2O_3 found by using these techniques are in good agreement with values reported in the literature.

ELECTRICAL CONDUCTIVITY OF CERIUM OXIDE

The electrical conductivity of CeO_{2-x} was measured to 2000 °C in argon. The increase in conductivity with a decrease in oxygen content and the anomalous decrease above 1900 °C was confirmed.

PREPARATION OF $\text{UO}_2 - 25 \text{ wt\% PuO}_2$ SPECIMENS FOR HIGH-TEMPERATURE CREEP STUDIES

A study was carried out to determine a method of preparing polycrystalline $\text{UO}_2 - 25 \text{ wt\% PuO}_2$ creep specimens. Several

different mechanically-mixed and coprecipitated powders were compacted and sintered under various conditions, and the resultant microstructures were examined. The study concluded that in order to obtain uniform pellets for creep studies, coprecipitated powders must be used.

THERMAL DIFFUSIVITY OF (U,Pu)O₂

Photomicrographs were obtained for coprecipitated mixed oxide thermal diffusivity specimens which contained 0, 5, 12, 20, 25, 30, and 45 wt% PuO₂. The information obtained from the photomicrographs was correlated with thermal diffusivity data previously reported.

DETERMINATION OF OXYGEN/METAL RATIOS FOR OXIDE FUELS

An experimental system has been constructed to examine the feasibility of measuring oxygen/metal ratios in oxide fuel samples weighing only about 1 mg. It has been found that outgassing of the graphite crucible used in the fuel reduction contributes an appreciable fraction of the CO₂ measured to determine the oxygen content of the fuel sample.

CHARACTERIZATION OF MIXED-OXIDE FUEL MATERIALS

The study of mixed-oxide fuels, using scanning electron microscopy, continued with detailed examination of calcination of plutonium oxalate and nitrate to form PuO₂, and of sintering of PuO₂ and UO₂ - PuO₂. Correlations between calcination temperature, physical treatment of powders, measured particle sizes, sintered densities, microstructure and micro-composition are being developed.

BASIC SWELLING STUDIES

Swelling was low in specimens of uranium, U-5 wt% Pu, and U-14 wt% Pu-12 wt% Zr irradiated at 1000 psi between 375 °C and 625 °C. Swelling in U₃Si specimens was greater at lower temperatures.

When fissionable metals are irradiated, the ordered metallic structure is perturbed in a number of ways. High energy neutrons and fission fragments collide with metal atoms, knocking them out of their structural positions. The resultant defects, vacancies, interstitials, dislocation loops, etc., can adversely affect metallic properties. Similarly, the fission products generated, some of which are noble gases, may be incompatible with the structure and so precipitate or coalesce as second phase inclusions or gas pores. Furthermore, the high, localized temperatures associated with the fission event can cause anisotropic fissionable metals such as uranium to drastically change shape in accordance with the crystallography of the specimens.

These effects and their interactions are being studied with regard to the influence of irradiation variables such as the specimen temperature, burnup, burnup rate, and external pressure, and with regard to the influence of metallurgical variables such as composition, structure, and geometry. The data thus far obtained on this program have established that, contrary to previous beliefs, swelling at low burnup in alpha uranium is due to mechanical processes associated with the irradiation "growth" phenomena and with fission-event induced defects rather than with the agglomeration and growth of fission gas pores. This is manifested as a maximum in the swelling-temperature curve in the 400 to 600 °C range.

A few tenths of a percent alloying addition or an applied external pressure have been found to be effective in overcoming the "mechanical" type of swelling. Swelling continues to decrease above 600 °C up to approximately 700 °C. Above 700 °C, fission gas swelling begins to occur. Efforts are continuing to better define the basic mechanisms involved and the interrelationship between burnup, temperature, pressure and metallurgical state. Fundamental information developed by this program

will provide a basis for engineering exploitation of the many beneficial attributes of metal fuels for use in reactor applications.

FAST-REACTOR DOSIMETRY AND DAMAGE ANALYSIS

Reaction rate ratios can be used to identify changes in neutron spectra since monitors respond differently to neutrons of different energies. Data from the recent EBR-II dosimetry test show a possible change in the spectrum between the low- and high-power tests. Other data from the high-power tests show the spectrum was constant axially in Row 2 up to 4 in. above midplane. Such information on spectra is useful in the analysis of both monitor and irradiation damage data.

A previous report discussed the development of a method for generating energy-dependent damage functions for the correlation and prediction of neutron-induced property change. A damage function was previously derived for a 200 °F ductile-brittle transition temperature (ΔTT) increase generated by neutron irradiation at temperatures less than 450 °F in ASTM A302-B pressure-vessel steel. In the present study, a set of group-averaged values of this damage function was selected and used to compare measured and damage-function calculated fluences for new experimental ΔTT data. The results provide further validation of the applicability of the damage function.

Recent indications of a significant contribution by thermal neutrons to the embrittlement of some ferritic steels have prompted computer experiments on low-energy displacement spikes such as produced by (n,γ) recoils. Preliminary results with individual 400 eV spikes indicate some clustering at low temperatures but not at elevated temperatures.

The CASCADE-type computer code is being reformulated and programmed to simulate damage generation in the FCC lattice

structure. It will permit a more realistic simulation and widen the scope of simulation to include effects in previously damaged lattices and effects of knock-on atoms with energies greater than 20 keV.

In computer simulation studies using the GRAINS code, a 6° tilt boundary in a lattice simulating fcc γ iron was observed to heal upon introduction of a carbon atom in the vicinity of the boundary. A significant reduction in the energy of the system occurred as the carbon atom reached its equilibrium position.

IRRADIATION DAMAGE TO REACTOR METALS

REACTOR METALS RESEARCH

Irradiation Facilities Operation

A total of 162 tensile, creep, and corrosion specimens were discharged from the ETR at the conclusion of Cycle 100.

Core I testing in the ATR was completed on June 6, 1969. Cores II and III and Cycle 1 are scheduled for June 15, 1969; July 6, 1969; and September 20, 1969, respectively.

MICROSTRUCTURAL MODIFICATIONS TO STAINLESS STEELS AND NICKEL-BASE ALLOYS

A comparison was made of the stacking fault hardening rates produced by quenching, straining at room-temperature, straining at 700 °C, and straining at 700 °C followed by a half-hour hold under load at 700 °C. The results showed that hardening-rate increased in the order: straining plus hold > straining > quenching. That straining plus hold treatment produced a faster hardening-rate than did straining alone was rationalized in terms of the added effect of vacancy-enhanced diffusion.

IN-REACTOR MEASUREMENTS OF MECHANICAL PROPERTIES

Discontinuous yielding during neutron irradiation has been measured in annealed 304 SS at 370 °C stressed to 35,000 psi.

This phenomenon, induced by irradiation, resulted in an average creep-rate of $6 \times 10^{-5} \text{ hr}^{-1}$.

The values of the activation parameters and the stress-dependence of the creep-rate in Zr-2.5 Nb are consistent with the "dislocation-climb" model for creep with short-circuited diffusion. Neutron irradiation can be thought of as adding a stress component for faster creep. The stress arises from an osmotic force on climbing dislocations resulting from the super-saturation of point defects created by irradiation.

FORMATION OF VOIDS IN IRON DURING HIGH-TEMPERATURE NEUTRON IRRADIATION

Irradiation of high purity Fe to $3 \times 10^{21} \text{ n/cm}^2$ ($E > 0.1 \text{ Mev}$) at 450°C produces $\sim 1 \times 10^{14}$ voids of average diameter $\sim 280 \text{ \AA}$.

SMALL-ANGLE X-RAY SCATTERING BY VOIDS IN IRRADIATED METALS

Analysis of small-angle scattering from metals irradiated at elevated temperatures yields void diameters consistent with those measured by electron microscopy.

FAST REACTOR SUPPORTING STUDIES

DAMAGE ANALYSIS

An equation describing the effect of fluence and temperature on swelling in solution-treated 304 SS has been refined.

Transmission electron microscopy studies of irradiated SS indicates that the nucleation rate of cavities may increase with accumulated exposure.

An analysis of carbide distributions in 316 SS indicates an increase of carbides precipitation in irradiated material.

EBR-II IRRADIATIONS

At the present time there are seven active experiments (swelling, weldment and structural materials) in EBR-II. Eight

BNWL structural materials experiments have been discharged from EBR-II. The flux monitors from these discharged experiments are in the process of being counted. Four BNWL weldment experiment pins in subassembly x-067 are to have a goal exposure of 1×10^{22} n/cm² (E > 0.1 MeV).

ACCEPTANCE TESTING AND CLADDING CHARACTERIZATION

Elevated-temperature burst tests and 1200 °C stress-rupture tests for times less than 1400 hr have been completed on tubing lots E, F, G, & H. Metallography and check chemical analyses have been completed on tubing lot G. Biaxial creep curves on tubing lots E & F have been completed.

UNIAXIAL CREEP RUPTURE STUDIES

The results of postirradiation creep-rupture tests on 304 and 316 stainless steel are reported for specimens irradiated in the temperature range of 700 °F (371 °C) to 900 °F (482 °C) and to total fluences from 1×10^{21} n/cm² to 1×10^{22} n/cm² (EBR-II calculated). Rupture-life reductions were observed for all irradiated specimens. Both increased and decreased creep-rates were observed depending on temperature and stress-level of test and irradiation fluence.

BIAXIAL STRESS-RUPTURE STUDIES

Biaxial stress-rupture tests at 1200 °F have been initiated on AISI type 304 SS irradiated to 0.8×10^{22} n/cm² (E > 0.1 MeV) in EBR-II. Specimens were stressed sufficiently to cause rupture in 0.1 to 1000 hr. in unirradiated material. Based on these initial tests, the data suggest some increase in rupture strength for short rupture times and loss in rupture strength for long rupture times for irradiated 304 SS. Strain is observed to be sharply reduced from unirradiated values.

FTR VESSEL WELDMENT STUDIES

Characterization of candidate FTR vessel weldment processes is in progress. Tensile test results are reported for

as-fabricated specimens from weldments in 1-inch-thick 304 SS plate. The testing matrix includes four welding processes, three specimen types, and four testing temperatures. This matrix constitutes the baseline mechanical property information to be used on conjunction with the weldment irradiation experiment. Four MK-B-7 type pins containing tensile, creep-rupture, and fatigue specimens have been shipped to the EBR-II site for inclusion in subassembly X067-4F2.

LMFBR FUEL AND CLADDING INFORMATION CENTER

An information storage, retrieval, and data analysis system consisting of six sections (cladding fabrication, fuel fabrication, fuel-pin fabrication, fuel-subassembly fabrication, irradiation history, and postirradiation examination) is being designed to store information related to the irradiation performance of LMFBR fuel pins. The cladding fabrication section is completed and being used.

NUCLEAR GRAPHITE

R. E. Nightingale

INERT-GAS-SENSITIZED RADIOLYSIS STUDIES

G. L. Tingey

Gasification of graphite in helium-cooled, graphite-moderated reactors occurs due to reactions of impurities in the helium coolant. In the high radiation field of a nuclear reactor, radiation-induced reactions as well as thermally-induced processes occur.

In a high-purity system such as the HTGR, most of the radiation energy is absorbed initially in the helium coolant. Thus, the rate of gasification is largely dependent upon the effectiveness of the energy transfer from excited helium to the reactive impurities.

Previous studies⁽¹⁾ have shown that the energy absorbed in helium is just as effective in producing reaction as that energy absorbed directly in the reactive gases when the impurity concentration is greater than about 0.6%. More recently, we have demonstrated⁽²⁾ a significant decrease in the efficiency of energy transfer when the total impurity concentration was 0.06%.

Studies have now been conducted to determine the efficiency of energy transfer at impurity concentrations as low as 54 ppm. For this study, we have measured the $\underline{G}(\text{CO})$ for the reaction of carbon dioxide with hydrogen in a large excess of helium. The measured $\underline{G}(\text{CO})$ at 350 °C at CO_2 and H_2 concentrations above 3500 ppm is 1.9 molecules/100 eV. This value decreases slightly as the CO_2 and H_2 concentration decreases below 500 ppm. At a concentration of H_2 and CO_2 of 300 ppm, the $\underline{G}(\text{CO}) = 1.5$ molecules/100 eV. As the CO_2 and H_2 concentrations decrease further, there is a further decrease in $\underline{G}(\text{CO})$ to a value of 0.65 molecules/100 eV when the CO_2 and H_2 concentrations are each 54 ppm.

Further experiments are planned to extend the measurements down to impurity levels near 1 ppm.

RADIATION-INDUCED CREEP IN GRAPHITE - W. J. Gray

Molten-salt reactors of the types being considered by Oak Ridge National Laboratory require a graphite capable of remaining impermeable both to the molten salt itself and also to fission gases. ORNL has assumed that a graphite that was initially impermeable to the salt and fission gases would remain so, at least until it began to expand rapidly at high fast-neutron fluences. Also, it is known that stresses built up within the graphite by thermal gradients and neutron flux gradients would be relaxed, at least at low fluences, by radiation-induced creep thereby preventing cracking of the graphite. It remains to be demonstrated, however, that the creep constant remains high enough to accommodate the anticipated stresses at high fluences. Consequently, a study has been started to measure creep constants at fluences up to 3×10^{22} n/cm² (E > 50 keV).

Although irradiations for this study have not yet been started, ORNL and PNL have reached general agreement on the design of this study. Specifically:

- Three types of graphite will be irradiated: AXF-80BG, a fine-grained, isotropic graphite manufactured by POCO Graphite, Inc.; H-337, a raw coke, isotropic graphite manufactured by Great Lakes Carbon Corp.; and AGX (CS-112). The latter will be included only as an analogue to AGOT graphite on which ORNL already has some creep data.
- Irradiation temperatures will be 550 and 800 °C with a goal of ±30 °C control.
- Stress levels will be set at 1000, 2000, and 3000 psi, determined by a fixed load but with varied sample cross sections. Stress will be applied by a pneumatic bellows.

- Although tensile and compressive creep should be identical at low fluences, they may very well differ at high fluences. However, only compressive experiments will be done at first, with the idea that tensile experiments will be started after experience is acquired with the compressive tests.
- Samples will be irradiated in the L8 test position of the ETR reactor. Compressive creep capsules will go in two of the quadrants, one capsule for each irradiation temperature. Eventually, a third quadrant will likely be occupied by a tensile creep capsule. Irradiation of the compressive creep capsules will be started in October, 1969.
- A careful determination of the flux profile in the L8 position will be made, taking into account neutrons with energies greater than 50 keV. The desirability of holding the flux profile constant will necessitate the same configuration in the test facility at all times. To achieve this, all four quadrants will be occupied by graphite samples.

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HTGR GRAPHITETHE RADIOLYTIC REACTION OF GRAPHITE WITH WATER VAPOR -

R. E. Woodley

As a consequence of impurity problems in the previous ampoule irradiations, the radiolytic reaction of graphite with water vapor is presently being investigated in a single-pass flow system. In this system, approximately 20,000 volume parts per minute (vpm) of water vapor are added to the helium carrier gas following purification of the helium by passage over hot copper turnings and through a liquid nitrogen-cooled molecular sieve trap. After humidification, the helium is flowed past a TSX graphite sample positioned in the cobalt-60 gamma facility at the point of highest dose rate ($\sim 1.7 \times 10^7$ R/hr). To determine the reaction rate, the influent and effluent gas streams are sampled simultaneously and the gas samples analyzed on a Varian Trace Gas Analyzer. Changes in the concentrations of oxides of carbon can then be related to the rate of reaction. In order to obtain the radiolytic yield, one must know the volume of "activated" gas effective in promoting reaction. For this reason, the graphite sample was machined to fit tightly within its container. Measurements of the apparent density (1.700 g/cm^3) and the helium density (2.188 g/cm^3) allow the calculation of the open pore volume of the graphite sample. It is this volume which is used to calculate radiolytic yields.

Under the experimental conditions presently employed ($\text{CH}_2\text{O} \approx 20,000 \text{ vpm}$, $\bar{T}_{\text{graphite}} = 117^\circ\text{C}$, and helium flow rate $= 30 \text{ cm}^3/\text{min}$) changes in the concentration of CO_2 , the major product analyzed, are very small, amounting to only a few tenths of 1 vpm. The presence in the influent gas stream of CO_2 at concentrations ranging from about 0.2 to 0.6 vpm tends to mask the graphite - water vapor reaction, because CO_2 also undergoes a radiolytic reaction with graphite. One source of this CO_2 appears to be the water used to humidify the helium,

even though the water was initially purified by distillation followed by alternate freezing and pumping. Another source may be CO_2 adsorbed on the walls and in the stopcock grease of the gas sampling bulbs.

The very small changes observed in the CO_2 concentration upon irradiation have been both positive and negative. When the inlet CO_2 concentration is relatively high, 0.5 to 0.6 vpm, a decrease in the concentration has been observed. This decrease is presumably a result of radiolytic reaction with the graphite. The average radiolytic yield for this process is $G(-\text{CO}_2) = 1.7$. At lower initial CO_2 concentrations, <0.5 vpm, an increase in the CO_2 concentration upon passage through the gamma facility has been noted. This increase may arise from the graphite - water vapor reaction and exhibits $G(\text{CO}_2)$ values ranging from 0.8 to 1.6. At the present time, it is difficult to draw any firm conclusions from these data, other than it seems likely that the radiolytic yield for the graphite - water vapor reaction must be small. Obviously, as much CO_2 as possible must be removed from the inlet gas in order to reduce its interference with the vapor reaction. Improved analytical results will be obtained by alternately flowing the inlet and outlet gas streams directly through the sampling system of the gas chromatograph rather than relying on sample bulbs which are always potentially contaminated with CO_2 .

NUCLEAR CERAMICS

R. E. Nightingale

PROPERTIES OF MOLTEN FAST-REACTOR OXIDE FUELS - R. P. Nelson,
J. J. Rasmussen, and O. D. SlagleMeasurements System

Installation of the high-temperature vacuum resistance furnace within a plutonium glove box located in the Shielded Materials Facility has been completed. This arrangement will allow measurements of liquid density, viscosity, surface tension, and compressibility on both unirradiated and irradiated mixed-oxide fuels to 3000 °C. The furnace employs a tungsten-mesh resistance element and a split radiation shield assembly allowing x-radiographic determination of liquid density and surface tension with a minimum of absorbing material in the image-forming X-ray beam. Preliminary measurements of surface tension and density will be made of UO_2 before the system is committed to plutonium.

A second furnace intended specifically for surface tension measurements of mixed oxides by the pendant-drop method is being designed. This furnace, which employs a self-resistance heated capsule, will replace an older furnace which is unsuitable for glove box operation.

Surface Tension

Surface tension is being measured by two liquid surface shape techniques: pendant drop shape and equilibrium meniscus shape. In both cases, a profile of the liquid surface is obtained by x-radiography of the material contained in a closed refractory-metal capsule. The pendant drop shape technique has the advantage that the liquid oxide is only in contact with solid oxide of the same composition. Thus, impurities which

can affect the surface tension value are not picked up from the capsule. However, the surface tension can only be obtained at the melting point temperature. The equilibrium meniscus shape technique can be used at temperatures well above the melting point.

Pendant drops are formed on the end of the sintered oxide rods which are suspended vertically in the refractory-metal capsule in such a way that the free end of the rod coincides with the center of the capsule length. The capsule walls are thinned slightly from each end to the center in order to establish a temperature gradient when the capsule is self-resistance heated. In this way, only the free end of the rod is melted and a stable pendant drop is formed. If the temperature is raised, the drop falls off the rod and a new drop is formed. In this way, several different drops can be radiographed during a single experiment. The equilibrium meniscus shape is obtained by melting a rod of the oxide in a capsule having a uniform temperature. The rod volume is adjusted so that the melt fills the lower portion of the capsule.

When two fluids are separated by an interface, the shape of the interface is governed by the fundamental equation for the definition of surface tension,

$$\frac{1}{R_1} + \frac{1}{R_2} = \frac{P}{\gamma} \quad , \quad (1)$$

where R_1 and R_2 are the principal radii of curvature at a point on the surface, P is the pressure difference across the interface at the point, and γ is the interfacial tension. When the pressure difference is due to gravitation, Equation (1) leads to the differential equation describing the shape of a cylindrically symmetric drop,

$$\begin{aligned}
 & b f''(Z) + \frac{b}{X} \left\{ 1 + [f'(Z)]^2 \right\} f'(Z) \\
 & = \left(2 + \frac{\beta Z}{b} \right) \left\{ 1 + [f'(Z)]^2 \right\}^{3/2}, \quad (2)
 \end{aligned}$$

where

$$\begin{aligned}
 f'(Z) &= \frac{dZ}{dX}, \quad f''(Z) = \frac{d^2Z}{dX^2}, \\
 \beta &= \frac{g b^2 \Delta \rho}{\gamma}, \quad (3)
 \end{aligned}$$

b is the radius of curvature at the base of the drop, g is the gravitational acceleration, $\Delta \rho$ is the difference in fluid densities, γ is the surface tension, and X and Z are the coordinates of points along the line of intersection of the drop surface and a plane containing the radius of curvature b . In most cases, the capsule is sealed in vacuum and $\Delta \rho$ is simply the liquid density. The parameter β describes the shape of the drop, and b is a measure of drop size. All drops with the same value of β are geometrically similar. β is positive for oblate figures of revolution, i.e., for a sessile drop or a meniscus in a capillary and negative for prolate figures of revolution, i.e., for a pendant drop.

A computer program has been written which determines the surface tension by analyzing the shape of a pendant drop or meniscus. Using the pendant drop as an example, the program is set up by measuring X, Z pairs from the drop profile along the arc segment from $\phi = 0^\circ$ to $\phi = 90^\circ$, (Figure 5.1). The largest value of X and the associated Z value are found. These are assumed to be X_{90} and Z_{90} , the drop coordinates at the equator. A table of X_{90}/Z_{90} as a function of β , taken from Bashforth and Adams,⁽¹⁾ is searched to obtain the initial value of β . The function $Z(X, b, \beta)$ is generated from Equation (2) and compared with the experimental X, Z pairs. The parameters b and

FIGURE 5.1. Parameters for Determination of Surface Tension from the Shape of a Pendant Drop

β are then adjusted to obtain the best fit between the experimental values and the calculated values. The surface tension is then calculated from Equation (3) using the adjusted values of b and β .

The techniques have been checked using Al_2O_3 . Preliminary values are in good agreement for the surface tension found by the pendant-drop technique and the equilibrium meniscus-shape technique and with values for the surface tension reported in the literature. A UO_2 pendant-drop capsule is presently being prepared.

ELECTRICAL CONDUCTIVITY OF CERIUM OXIDE - J. L. Bates

Cerium oxide has a fluorite structure and properties similar to that of PuO_2 and is often used as a non-radioactive stand-in for PuO_2 . The purpose of this study was to determine the electrical conductivity (σ) of cerium oxide from room temperature to a temperature as near the melting point as possible in an effort to understand the high temperature physical and structural properties of related oxides of plutonium.

Initial electrical conductivity measurements⁽²⁾ have been reported for three thermal cycles to 1000 °C and for a fourth cycle extending to 1995 °C. This report describes the electrical conductivity results of a fifth thermal cycle to 2000 °C, and the characterization of the sample after the electrical measurements were completed. Figure 5.2 illustrates the results and heating conditions for all five cycles.

The electrical conductivity of the starting stoichiometric CeO_2 near room temperature was below the measurable limits (10^{-6} mhos/cm) of the apparatus. Therefore, the initial measurements in the first four thermal cycles began between 200 and 300 °C. The conductivity increased rapidly with temperature and exhibited two discontinuities in the $\log \sigma$ versus $1/T$ curve (Figure 5.2). These breaks occurred at approximately

FIGURE 5.2. Electrical Conductivity of CeO_{2-x} .

550 and 950 °C with the slope of the curve between these two temperatures less than the slope at higher or lower temperatures. Similarly shaped curves were observed during Cycles 1 through 4 below 1000 °C. Each cycle resulted in significant increases in conductivity and in a reduction of activation energy. In each instance, the slope above and below the break at approximately 200 to 300 °C decreased during subsequent heating cycles.

Above approximately 1000 °C, the electrical conduction increases significantly with increasing temperature and is associated with an activation energy of approximately 5 eV. All of the conductivity curves for which the maximum temperature had not exceeded 1000 °C converge into this straight line, observed in Cycle 4. At approximately 1130 °C, another sudden change occurs in the temperature dependence of the $\log \sigma$ versus $1/T$ curve. In the small temperature range between approximately 1000 and 1130 °C, the electrical conductivity increased approximately 250 times compared to an increase of 2 to 3 in the larger temperature interval from 1130 to 1900 °C. The electrical conductivity decreased from 12 to 3 mhos/cm during an increase in temperature from 1900 to 1995 °C.

Measurements during a fifth cycle were conducted to determine if the unusual changes in the electrical conductivity observed during the cooling portion of Cycle 4 could be reproduced.

The electrical conductivity during both heating and cooling agree very closely with the electrical conductivity values measured during cooling of Cycle 4 (Figures 5.2 and 5.3). The values during heating were slightly less than those during the previous high temperature cycle. Both exhibited a decrease in conductivity above 1900 °C with the change occurring at a slightly higher temperature during the final heating cycle.

FIGURE 5.3. High-Temperature Electrical Conductivity of CeO_{2-x}
 (Values not corrected for dimensional changes
 which occurred near apparent melting point.)

Slight changes in linearity in the $\log \sigma$ versus $1/T$ curve between 1000 and 1300 °C were observed in both high-temperature cycles. A slight hysteresis was observed on heating and cooling in this temperature range in Cycle 5 (Figure 5.3). Therefore, the electrical conductivity values in Cycles 4 and 5 appear to be representative of the CeO_{2-x} removed from the furnace.

The appearance of the sample changed considerably during the electrical conductivity measurements. The color of the original stoichiometric sample was white with a yellowish cast. The sample at the termination of the experiments was a dark blue-black. The sample, although still intact, showed a glassy-like appearance and evidence of material flow. This was interpreted to have resulted from partial melting of the CeO_2 sample. Some changes in sample and electrode probe dimensions were observed (Figure 5.4). The CeO_{2-x} was extremely friable and broke into small pieces when removed from the tungsten holders.

FIGURE 5.4. Bottom View of $\text{CeO}_{1.82}$ Sample in Tungsten Holder Showing Glassy Appearance of Oxide Surface and Areas of Sample Flow. (0.80X)

An analysis of the changes in the sample cross sectional area (A) and the voltage probe spacing (ℓ) showed a decrease of 85% in the A/ ℓ ratio resulting during the electrical conductivity measurements. These changes are small compared to the gross changes in conductivity. The curves corrected for these dimensional changes are shown in Figure 5.5.

Thermogravimetric analysis in air of the CeO_{2-x} showed a strong oxidation reaction between 200 to 250 °C and a smaller reaction near 450 °C. Very little weight change was observed from 450 to 950 °C. The O/Ce ratio for two samples was 1.82.

The microstructure of the $\text{CeO}_{1.82}$ after the conductivity measurements is illustrated in Figures 5.6 and 5.7. The oxide near the outer shiny sample surface was made up of large grains relatively free of porosity. This area, which was near the tungsten holders, contained metallic inclusions of tungsten, a darker second phase, and was grossly cracked.

A sharp demarkation is apparent between the high density area near the surface and the interior of the sample. The interior was free of metallic and darker oxide second phases. The oxide was interlaced with fine cracks, which made the sample very friable and difficult to polish. This structure is very similar to structure observed in CeO_2 which had formed during melting point measurements.

Preliminary X-ray diffraction examination indicated a two-phase system with the major phase being a fluorite cubic structure. Additional X-ray analyses are being conducted.

PREPARATION OF UO_2 - 25 wt% PuO_2 SPECIMENS FOR HIGH-TEMPERATURE CREEP STUDIES - O. D. Slagle and J. A. Basmajian

A program was initiated to determine a method of fabricating UO_2 -25 wt% PuO_2 pellets from which creep test specimens could be prepared. The minimum requirements of such pellets are:

FIGURE 5.5 High-Temperature Electrical Conductivity CeO_{2-x} Corrected for Dimensional Changes Estimated after Temperature Measurements.

Neg 469618A

0.05 cm

Neg 469618B

FIGURE 5.6. Microstructures of $\text{CeO}_{1.82}$ After Electrical Conductivity Measurements Showing Large Grains Near Sample Surface in Areas of Sample Flow. (As-Polished 100X)

*Neg 469618C**0.01 cm* *Sample
Surface**Neg 469618D*

FIGURE 5.7. Microstructure of $\text{CeO}_{1.82}$ Sample after Electrical Conductivity Measurements 500X

- Densities in excess of 90% TD.
- Uniform size and distribution of grains and pores, with no macro- or micro-cracks.
- Large enough to cut creep specimens, 1-1/8 in. long, for four-point bend tests.

The length of the sample requires a fairly large pellet size. This large size complicates sample preparation due to problems of density and nonuniformity produced during pressing and subsequent cracking during heating and cooling. Two different pellet press shapes have been tried:

- A rectangular die 1/2 in. x 1-1/2 in.
- A cylindrical die 1-1/2 in. in diameter.

The pressed pellets are 1/2 in. thick. The rectangular shape is more desirable because there is much less wastage when cutting the bar-shaped creep specimens. However, since rectangular pellets tended to laminate more easily during pressing, all the pellets in this study were pressed in the cylindrical die.

A number of techniques were considered for obtaining crack-free, uniform pellets, and the resulting study is outlined in Table 5.1. The UO_2 used in preparing the mechanical mixtures was ceramic grade powder. All pellets except those containing butyl stearate as a binder were pressed using butyl stearate as a die-wall lubricant. Sintering was carried out in an argon - 8% hydrogen atmosphere. The sintering schedule was 80 °C/hr to 600 °C, 100 °C/hr to 1650 °C, an 8-hour hold at 1650 °C, and a cooling rate of 250 °C/hr.

Table 5.2 lists the densities before and after sintering of samples prepared from Type 1 powder. In general, increasing the pressing pressure increased the green densities but little or no systematic effect on the final densities was observed. In general, addition of carbowax or butyl stearate gives higher green densities together with a reduction in sintered density,

TABLE 5.1. *Outline of Sample Preparation Study for UO_2 -25 wt% PuO_2 .*

Powder	Binder	Pressing Pressures, ksi
1. UO_2 -25 wt% PuO_2 .	Pressed dry.	40
Mechanically mixed by dry milling 40 hr.	2 wt% carbowax.	30
	4 wt% carbowax.	20
PuO_2 prepared from the oxalate.	4 wt% butyl stearate.	
2. UO_2 -25 wt% PuO_2 .	Pressed dry.	40
Mechanically mixed by wet milling for 1 hr.	Butyl stearate.	
a. PuO_2 prepared from the oxalate and wet milled for 6 hr.		
b. PuO_2 prepared from Pu metal.		
3. Coprecipitated UO_2 -20 wt% PuO_2 .	Pressed dry.	40
Sieved through a 70-mesh screen and ball milled in alcohol for 6 hr.	Butyl stearate.	

TABLE 5.2. *Theoretical Density Before and After Sintering for Type 1 Powder Pellets.*

(Percentages of theoretical density before sintering are in parentheses.)

Powder Additive	Pressing Pressure		
	40 ksi	30 ksi	20 ksi
Dry	94.0 (52.0)	94.0 (49.8)	93.8 (46.9)
2 wt% Carbowax	93.9 (53.8)	94.2 (52.0)	94.2 (49.0)
4 wt% Carbowax	91.0 (53.0)	93.2 (51.6)	92.5 (49.4)
4 wt% Butyl Stearate	89.7 (54.3)		89.1 (49.9)

compared to pressing without a binder. For addition of 4 wt% butyl stearate, this amounted to a 4% decrease in sintered density.

Figure 5.8 shows representative microstructures for sintered pellets of Type 1 powder pressed dry at (a) 40, (b) 30, and (c) 20 ksi. The porosity distribution is very similar for samples pressed at 40 and 20 ksi, except that the pellet pressed at 40 ksi has some internal microcracking.

Figure 5.9 shows a similar series of micrographs for pellets pressed with the addition of 2 wt% carbowax. These indicate that the addition of carbowax has resulted in extensive microcracking and an inhomogeneous distribution of porosity. Figure 5.10 shows the micrographs for pellets pressed from powders having 4 wt% carbowax. For these pellets, very little microcracking occurs but the porosity distribution is very inhomogeneous.

Butyl stearate has been found to be a very effective die lubricant for dry pressing UO_2 powder, and addition of butyl stearate to the powder may serve as an efficient powder lubricant during compaction and may result in a higher final density. Actually, the densities in Table 5.2 indicate the opposite effect for the addition of 4 wt% butyl stearate. The microstructure was not uniform and contained large spherical pores (Figure 5.11).

Scanning electron microscope studies of starting powders used in mechanically-mixed fast-reactor fuel studies indicate that PuO_2 particles prepared from the oxalate are spherical in shape and appear hard and more dense than ceramic-grade UO_2 . This suggests that the spherical voids present in these microstructures may result from the characteristics of the PuO_2 powder. The Powder Types 2a and 2b represent attempts to eliminate the spherical pores. In the case of Type 2a, the PuO_2

FIGURE 5.8. Representative Microstructures (Unetched)
of Type 1 Powder Pressed Dry (500X)

Neg 4964B

(a) 40 ksi. Neg 4965B

(b) 30 ksi.

Neg 4966B

(c) 20 ksi.

50 μ

FIGURE 5.9. Representative Microstructures (Unetched) of Type 1 Powder with 2 wt% Carbowax Pressed at the Indicated Pressures. (50CX)

FIGURE 5.10. Representative Microstructures of Type 1 Powder with 4 wt% Carbowax Pressed at the Indicated Pressures (500X)

50 μ

FIGURE 5.11. Representative Microstructures for Mechanically-Mixed Pellets Pressed at the Indicated Pressures with 4 wt% Butyl Stearate (500X)

powder was ball milled to break down the spherical agglomerates, whereas in Type 2b, PuO_2 powder was obtained by burning the metal. Representative microstructures for pellets produced from the Type 2a and 2b powders mixed with 4 wt% butyl stearate and pressed at 40 ksi are shown in Figure 5.12. Ball milling the PuO_2 prepared from the oxalate appears to have eliminated the spherical voids (Figure 5.12a). In the case of the mixed oxide containing PuO_2 prepared from the metal, a number of large irregular-shaped pores are present (Figure 5.12b). The gray areas may be a second phase related to the low O/M of 1.95 of the sintered pellet. During sintering, a hole developed in the alumina muffle and the argon atmosphere around the graphite heating element flowed over the sample. This atmosphere probably had a high CO pressure and was strongly reducing.

The difficulty of obtaining uniform microstructures by mechanically mixing two powders is to be expected when the powders are very dissimilar. This problem can be eliminated by using coprecipitated powders. Sintered pellets for this Type 3 powder had densities of 97.6% theoretical. Representative microstructures before and after etching are shown in Figure 5.13. The unetched micrographs show a marked improvement in microstructure resulting from the use of coprecipitated powder. The random light areas have previously identified as tungsten carbide from the tungsten-carbide ball mills. The etched micrographs indicate that the pellet containing 4 wt% butyl stearate has a grain size twice as large as the one pressed dry. Furthermore, the pellet made from powder containing butyl stearate exhibits a more uniform microstructure.

It was concluded that in order to obtain uniform pellets for creep studies, coprecipitated powders must be used. Further studies will be made to determine methods for varying densities and grain sizes.

Neg 49114B

(a)

Neg 49133C

(b)

50 μ

FIGURE 5.12. Representative Microstructures (Unetched) (b) for Type 2a and 2b Powders Mixed with 4 wt% Butyl Stearate and Pressed at 40 ksi (500X)

FIGURE 5.13. Representative Microstructures Etched [(b), (d)] and Unetched [(a), (c)] of Powder Type 3 Pressed with 4 wt% Butyl Stearate Added [(a), (b)] and Pressed Dry [(c), (d)] (500X)

THERMAL DIFFUSIVITY OF (U,Pu)O₂ - R. L. Gibby

Last quarter, thermal diffusivity data were reported for coprecipitated mixed oxide compositions containing 0, 5, 12, 30, and 45 wt% PuO₂.⁽³⁾ During this quarter, the diffusivity test specimens, plus two other samples from the same coprecipitation series but containing 20 and 25 wt% PuO₂, were examined ceramographically and photomicrographs of typical microstructures were obtained. This information is correlated with the previously reported thermal diffusivity data.

Photomicrographs of the polished and etched samples are shown in Figures 5.14 and 5.15. The specimens containing <30 wt% PuO₂ had densities <95% TD. The photomicrographs of these specimens show that the porosity was uniformly distributed and located both within grains and at grain boundaries. The thermal diffusivities of these specimens were very similar, although the PuO₂ content appeared to have some effect on the temperature dependence for compositions containing <12 wt% PuO₂. Other minor differences in thermal diffusivity appear to be related to the slight differences in the amount of porosity.

The samples containing 30 and 45 wt% PuO₂ have densities near 90% TD. The photomicrographs of these samples (Figure 5.15) show that the lower density is associated with the presence of large void areas as well as uniformly distributed fine porosity. In addition, in the composition containing 45 wt% PuO₂, the microstructure appears to be complicated by the presence of secondary unidentified phases, as shown in both the etched and unetched microstructure. The thermal diffusivity of the composition containing 30 wt% PuO₂ is less than that for the samples with less PuO₂; however, the temperature dependence is the same as the sample with 12 wt% PuO₂. A comparison of the microstructures of these two compositions indicates that the difference in thermal diffusivity is most likely due to the difference in pore volume and pore size distribution.

FIGURE 5.14. Photomicrographs of Coprecipitated $(U,Pu)O_2$ Compositions Sintered at $1650^\circ C$ for 4 hours Showing Microstructure of Etched Specimens at 250X.

(a) $UO_2 - 25 \text{ wt\% } PuO_2$

(b) $UO_2 - 30 \text{ wt\% } PuO_2$

(c) $UO_2 - 45 \text{ wt\% } PuO_2$
(Unetched)

(d) $UO_2 - 45 \text{ wt\% } PuO_2$

FIGURE 5.15. Photomicrographs of Coprecipitated $(U,Pu)O_2$ Compositions Sintered at 1650°C for 4 hours Showing Microstructures of Etched Specimens (with exception of c) at 250X.

The thermal diffusivity of the specimen containing 45 wt% PuO_2 is much less than any of the other specimens, moreover its temperature dependence is also quite different. The presence of additional phases in this composition appear to be responsible for this behavior, since the density of this specimen is not much different from the specimen containing 30 wt% PuO_2 . It is doubtful that these phases are suboxides of the mixed oxides, since the samples were heated in a protective cover gas to prevent oxidation or reduction during the thermal diffusivity measurements. An electron microprobe analysis will be conducted on this specimen to identify the additional phases.

DETERMINATION OF OXYGEN/METAL RATIOS FOR OXIDE FUELS

R. E. Woodley

The ability to determine oxygen/metal ratios on very small samples of oxide fuels would allow the measurement of oxygen distributions across the diameters of fuel elements. An experimental system has therefore been constructed to examine the feasibility of analyzing fuel samples weighing only about 1 mg. The analysis is performed by reducing a weighed sample of the fuel to the corresponding carbide plus CO_2 . For example:

The reduction is carried out in a graphite crucible heated inductively to a temperature of $\sim 2000^\circ\text{C}$. The CO_2 is collected in a liquid-nitrogen-cooled trap which is subsequently attached to the sampling system of a gas chromatograph.

The precise determination of the oxygen/metal ratio depends upon the quantitative collection of the CO_2 as well as the elimination of extraneous sources of this gas. At 1500°K , the equilibrium pressure of CO in reaction (1) is roughly 0.05 torr, so that outgassing of the graphite crucible can be safely performed at temperatures up to 1500°K with negligible loss of

fuel oxygen. Measurements of the outgassing of a TSX graphite crucible in a stream of pure helium at temperatures up to about 2000 °C indicated, however, that about 63% of the carbon oxides were evolved at temperatures in excess of 1100 °C. Literature subsequently revealed that outgassing studies at ORNL⁽⁴⁾ using TSX graphite indicated that about 46% of the CO evolved was released at temperatures between 1400 and 1800 °C.

In an attempt to eliminate the excessive outgassing found with TSX graphite, a crucible was constructed from pyrolytic graphite which is essentially non-porous. Unfortunately, because of its smaller size and its lower electrical resistance, the maximum temperature attainable was only about 1600 °C. Nevertheless, in the temperature range 1300 to 1600 °C, the pyrolytic graphite released 1.57×10^{-6} mole of CO + CO₂ per gram of graphite. On the other hand, TSX graphite in the temperature range 1100 to 2000 °C released only 6.05×10^{-7} mole of CO + CO₂ per gram of graphite. The reason for these outgassing results is not clear, but pyrolytic graphite will not be employed for future crucible construction.

Additional crucibles have been machined from Poco AXF graphite, but outgassing measurements have not as yet been performed. Depending upon the results with this material, it may prove necessary to outgas the graphite crucibles prior to adding the fuel sample. Following outgassing, the crucible would be maintained in an inert atmosphere to preclude reabsorption of oxygen or water vapor, and the fuel sample would then be added remotely.

Preliminary measurements of the CO₂ obtained upon UO₂ reduction have been performed manometrically. However, because the CO₂ resulting from graphite outgassing was an appreciable fraction of the total CO₂ collected, the O/M values calculated were not reasonable.

CHARACTERIZATION OF MIXED-OXIDE FUEL MATERIALS

J. L. Daniel and S. J. Mayhan

The study of mixed-oxide fuels continued, using scanning electron microscopy, in an intensive examination of the formation of PuO_2 feed powders by calcination of plutonium oxalate. Powder specimens were dry-mounted on a cooper support block on which a thin coating of adhesive had been sprayed (Scotch Spray Mounting Adhesive, Catalog No. 6065). The spray adhesive provided excellent retention of specimen particles during mounting and subsequent transfer of the support blocks between glove box and microscope. The spray eliminated the problems which were encountered in earlier experiments from deterioration of cellulose adhesive tape in the electron beam. However, some contamination of the microscope still occurred during examination of the finest powders, and a complete containment method is still being sought.

The plutonium oxalate, as-precipitated and dried at less than 100 °C, occurs in spherical clusters of platelets of random sizes and orientations (Figure 5.16). The clusters are all between 3 and 25 microns in diameter and show no evidence of bonding to each other.

Calcination of the oxalate produces progressively tighter clusters with reorientation of the platelets to produce the slot-shaped porosity reported earlier. Densification of individual particles is readily apparent as calcination temperature increases from 450 °C to 1000 °C (Figures 5.17a, b, and c). Only a few individual platelets occur separated from the clusters, but the open structure results in a relatively large effective surface area. The efficiency of blending with UO_2 for a mixed oxide fuel may be strongly influenced by the pore dimensions and stability of the PuO_2 clusters during the mixing operation. An earlier report showed that blending UO_2

Neg 1601

1000X

Neg 1596

3000X

FIGURE 5.16 *Plutonium Oxalate, As-Precipitated and Air Dried at Less than 100 °C.*

Neg 1369
(a) Calcined at 450 °C

5000X

Neg 1295
(b) Calcined at 700 °C

5000X

Neg 1396
(c) Calcined at 1000 °C

5000X

FIGURE 5.17 Effect of Calcination Temperature on Morphology of PuO_2 Powder Particles Prepared from the Oxalate.

with PuO_2 calcined at 700 °C produces nonporous powder particles, apparently by filling the interstices in the PuO_2 spheres with fine UO_2 powder.

If the oxide is produced by calcination of plutonium nitrate, a finer nonporous powder is obtained (Figure 5.18). Most of the particles are about 1 micron in diameter or smaller, roughly spherical, and show relatively little tendency to agglomerate. Different blending conditions may be required to achieve uniform mixing of this nitrate oxide with fine UO_2 powder, compared to the coarser particles obtained from the oxalate.

The particle-size analyses obtained using a Stokes Law method on the calcined oxalate shows a pronounced decrease in particle size as pre-analysis dispersion time is increased (Figure 5.19). Examination of the dispersed solids revealed that considerable breakup of the original plate clusters has already occurred after 15 min. of ultrasonic agitation. After dispersion for 120 min., nearly all of the original clusters are broken apart and the platelets destroyed, resulting in a large fraction of small particles and fine powder (Figure 5.20). Similar behavior is shown regardless of initial calcination temperature of the oxalate, although the resultant particles from the 1000 °C calcination retain some of the original rosette character. The relatively low bond strength between platelets in the oxide clusters is therefore another factor which must be considered in determining optimum blending conditions in mixed oxides preparation.

As the initial step in detailed evaluation of oxide fuel sintering, PuO_2 powder was pressed and sintered at 1650 °C after calcination at 450, 700, and 1000 °C. The sintered pellets were then fractured and examined directly in the scanning electron microscope. All three specimens achieved

Neg 1606

5000X

FIGURE 5.18 PuO_2 Prepared by Calcination of Plutonium Nitrate
at 1000°C (Compare with Figure 5.17).

VOLUME %
Greater
than
diameter

PuO_2 PARTICLE SIZE ANALYSES

Neg PNL 069 1755-1

FIGURE 5.19 Effect of Pre-Analysis Dispersion Time on Particle-Size Distribution in Calcined-Oxalate PuO_2 .

BNWL-1144

Neg 1616 5000X
(a) 15-Minute Dispersion

Neg 1613 5000X
(b) 120-Minute Dispersion

FIGURE 5.20 Effect of Pre-Analysis Dispersion Time on Particle Morphology of PuO_2 from Oxalate Calcined at 700°C .

only low density (<70%) upon sintering, perhaps reflecting the porous rosette structure of the starting powder. Pellets were sintered for about 4 hr at 1650 °C in argon/8% H₂ atmosphere containing less than 5 ppm H₂O. Density was determined by calculation from measured lattice parameters, and based on an O/M of 2.00. Since sintering was under highly reducing conditions, O/M is probably about 1.8, and true density may be about 5% higher.

All three specimens were similar in fractography, the main difference being the amount of displayed porosity which appeared to correspond roughly to the measured densities. In all cases, however, large intergranular porosity was predominant; virtually no pores occurred within the grains (Figure 5.21). In all specimens a large fraction of the PuO₂ grains had smooth edges with rounded corners, suggesting a significant reduction in surface activity of the grains facing the pores. In addition, the grain boundaries in the lower density specimens were poorly defined, appearing more like melt zones than sintered bonds. Nearly all grains showed a "humped" structure; that is, the centers of the grain surfaces rose abruptly above the peripheral regions where adjacent grains joined. The highest density (700 °C-calcined oxide) pellet displayed considerable surface debris, possibly resulting from its harder, more brittle behavior during fracturing.

The mixed oxides examined to date (25 wt% PuO₂) show a more typical brittle fracture surface. All specimens include particulate deposits or inclusions distributed randomly around the exposed surfaces and imbedded within grain boundaries (Figure 5.22), possibly resulting from the tungsten impurity content. Some specimens contained large regions of relatively small close-packed grains (Figure 5.23). A heavy "sprinkling" of 0.5 micron and smaller spherical particles also occurred in

Neg 1989 100X
(a) Initial Calcination Temperature
450 °C.

Neg 1992 1000X
Pellet Density 68%.

Neg 2033 100X
(b) Initial Calcination Temperature
700 °C.

Neg 2036 1000X
Pellet Density 64%.

FIGURE 5.21 Fractography of Sintered PuO_2 Pellets Prepared from
Calcined Oxalate Shown in Figure 5.17.

Neg 1977 100X Neg 1983
 (c) Initial Calcination Temperature Pellet Density 57%.
 1000 °C.
 FIGURE 5.21 (contd)

Neg 1969 300X Neg 1971
 FIGURE 5.22 Fractography of $UO_2 - 25 \text{ wt\% } PuO_2$
 (Tungsten Impurity Content $\sim 8.5\%$)

Neg 1964

165X Neg 1968

10,000X

FIGURE 5.23 *Fractography of Sintered Pellet of UO_2 - 25 wt% PuO_2 (Tungsten Impurity Content $\sim 2.5\%$)*

those areas. The grains themselves showed faceted surfaces and deep grain boundaries more typical of freely growing regions than of a fractured cross section.

Identification of the inclusions, evaluation of relative Pu and U content in various regions, and determination of general composition of areas shown would be extremely useful in interpreting electron micrographs. Such information can be obtained by use of the X-ray fluorescence attachment recently added to the scanning electron microscope. Study of the mixed oxide pellets by this technique is in progress.

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FAST REACTOR NITRIDES RESEARCH

No Contribution
This Quarter

NONDESTRUCTIVE TESTING

No Contribution
This Quarter

METALLIC FUELS

No Contribution
This Quarter

BASIC SWELLING STUDIES

C. R. Hann

POSTIRRADIATION EXAMINATION - C. R. Hann, B. Mastel

The status of capsules currently under evaluation is indicated in Table 9.1. Four controlled temperature-pressure capsules, P-13, P-21, P-22, and P-23, were opened in the radio-metallurgy facility and a total of 60 specimens recovered for density measurements and metallographic analyses. Table 9.2 gives the nominal chemical compositions of the various fuel materials. Metallurgical histories, irradiation parameters, and measured volume changes for capsules P-21, P-22, and P-23 are summarized in Tables 9.3, 9.4, and 9.5. Data from P-13 are not available at this time. Irradiation temperatures are given as time-average temperatures, and the error limits represent the range of occasional temperature fluctuations caused by flux perturbations and changes in reactor power levels.

The U-0 Pu, U-5 Pu, and U-14 Pu-12 Zr specimens were supplied by ANL as part of a joint program by PNL and ANL. The specimens were prepared by vacuum melting in yttria-coated, stabilized zirconia crucibles and injection casting into yttria-coated Vycor molds. Four pins of 0.130 in. diameter by 12-in. long were obtained from each melt. Irradiation specimens were machined from void free sections. Selected specimens were wrapped in yttria-coated tantalum foil and heat-treated in evacuated Vycor tubes. Holes were drilled in some of the 1/2-in. long rods for identification. Three rods were irradiated at any one position in the capsules; therefore, the first number of the specimen identification code refers to the capsule, the letter refers to the capsule position, and the number following the letter refers to the number of drilled holes.

The U_3Si specimens were supplied by the Chalk River Nuclear Laboratories as part of a joint USAEC-AECL cooperative program.

TABLE 9.1. Capsule Status

Capsule Number	Control Temp., °C	Control Pressure, psi	Goal ^(a) Burnup, at. %	Status
P-10	450	5000	0.2 - 0.8	Samples recovered and initial examinations completed
P-11	550	5000	0.2 - 0.8	Samples recovered and initial examinations completed
P-12	625	5000	0.2 - 0.8	Samples recovered and initial examinations completed
P-13	725	5000	0.2 - 0.8	Samples recovered and initial examinations completed
P-14	700	500	0.2 - 0.8	Samples recovered and initial examinations completed
P-15	700	1000	0.2 - 0.8	Samples recovered and initial examinations completed
P-16	625	1000	0.35 - 0.7	Samples recovered and initial examinations completed
P-17	825	5000	0.2 - 1.5	Samples recovered and initial examinations completed
P-18	825	50	0.1 - 0.7	Samples recovered and initial examinations completed
P-19	725	50	0.2 - 1.5	Samples recovered and initial examinations completed
P-20	725	50	0.2 - 1.5	Samples recovered and initial examinations completed
P-21	450	1000	0.1 - 0.2	Under examination in radiometallurgy
P-22	550	1000	0.1 - 0.2	Under examination in radiometallurgy
P-23	625	1000	0.1 - 0.2	Under examination in radiometallurgy

a. Different burnups are achieved in a single capsule by including specimens of various enrichments.

TABLE 9.2. *Compositions of Specimens in Swelling Capsules*

<u>Fuel Material</u>	<u>Alloy Designation</u>	<u>Composition (Additions in ppm)</u>
High Purity Uranium	HiP	U + 64 Fe-25 Al-6C
High Purity Uranium	E-1	U + 98 Fe-11 Al-34 Si-56 C
High Purity Uranium	E-2	U + 109 Fe-30 Al-36 Si-53 C
High Purity Uranium	U-O Pu	U + 50 Fe-6 Al-172 Si-172 C
Dilute Uranium Alloys	U + Fe - Si	U + 140 Fe-25 Al-95 Si-400 C
Dilute Uranium Alloys	U + Fe - Al - Si	U + 407 Fe-645 Al-95 Si-500 C
Dilute Uranium Alloys	WEB	U + 393 Fe-807 Al-32 Si-402 C
Dilute Uranium Alloys	WS	U + 350 Fe-800 Al-120 Si-600 C
Uranium-5 wt% Plutonium	U-5 Pu	U-4.96 wt% Pu + 30 Fe-33 Al-119 Si-118 C
Uranium-14 wt% Plutonium-12 wt% Zirconium	U-14 Pu - 12 Zr	U-13.89 wt% Pu - 11.86 wt% Zr + 50 Fe-20 Al-315 Si-144 C
Uranium Silicon Alloys	U ₃ Si (471)	U-3.58 wt% Si + 49 Fe-44 Al-62 C-800 Zr
Uranium Silicon Alloys	U ₃ Si (473)	U-3.98 wt% Si + 83 Fe-87 Al-112 C-2050 Zr
Uranium Silicon Alloys	U ₃ Si (501)	U-3.52 wt% Si
Uranium Silicon Alloys	U ₃ Si (5014)	U-3.53 wt% Si
Uranium Silicon Alloys	U ₃ Si (502)	U-3.88 wt% Si
Uranium Silicon Alloys	U ₃ Si (506)	U-3.98 wt% Si
Thorium-Uranium Alloys	Th-1 wt% U	Analysis Incomplete
Thorium-Uranium Alloys	Th-3.85 wt% U	Analysis Incomplete
Thorium-Uranium Alloys	Th-5.4 wt% U	Analysis Incomplete

TABLE 9.3. *Irradiation History and Swelling of Specimens in Capsule P-21
Irradiated at 450 °C and 1000 psi*

Sample	Fuel Material (a)	Met. (b) State	Irradiation Temp, °C		Burnup (c)		As-Irr. Density, g-cm ⁻³	% Swelling, $\left(\frac{P_i - P_f}{P_f}\right) \times 100$	"R" % Swelling at.% BU
			Up	Down	at.%	f-cm ⁻³ x 10 ⁻²⁰			
P21-A0	U-14 Pu-12 Zr	AC	371 ⁺³ ₋₁₁	288 ⁺² ₋₄	0.72	3.05	14.58	5.90	8.2
P21-A1	U-14 Pu-12 Zr	G	371 ⁺³ ₋₁₁	288 ⁺² ₋₄	0.72	3.05	14.69	4.56	6.3
P21-A2	U-0 Pu	G+A	371 ⁺³ ₋₁₁	288 ⁺² ₋₄	0.73	3.52	18.47	3.09	4.2
P21-B0	U-5 Pu	AC	423 ⁺⁹ ₋₅	374 ± 2	0.72	3.47	16.24	17.0	23
P21-B1	U-5 Pu	G	423 ⁺⁹ ₋₅	374 ± 2	0.72	3.47	17.44	8.89	12
P21-B2	U-0 Pu	G+A	423 ⁺⁹ ₋₅	374 ± 2	0.73	3.52	18.45	2.87	3.9
P21-C1	U ₃ Si (5014)		387 ⁺¹³ ₋₂	393 ± 2	0.21	0.87	10.93	42	200
P21-C2	U ₃ Si (506)		387 ⁺¹³ ₋₂	393 ± 2	0.20	0.80	10.29	49	245
P21-D0	U-0 Pu	AC	450 ± 2	410 ⁺² ₋₁₀	0.73	3.52	18.50	2.54	8.5
P21-D1	U-0 Pu	G	450 ± 2	410 ⁺² ₋₁₀	0.73	3.52	18.38	3.21	4.4
P21-D2	U-0 Pu	G+A	450 ± 2	410 ⁺² ₋₁₀	0.73	3.52	18.43	2.98	4.1
P21-E1	U ₃ Si (501)		466 ⁺⁴ ₋₁₁	398 ± 2	0.41	1.70	9.49	65	160
P21-E2	U ₃ Si (502)		466 ⁺⁴ ₋₁₁	398 ± 2	0.40	1.63	9.09	69	175
P21-F0	U-14 Pu-12 Zr	AC	450 ± 5	368 ⁺³ ₋₅	0.72	3.05	14.58	5.69	7.9
P21-F1	U-5 Pu	AC	450 ± 5	368 ⁺³ ₋₅	0.72	3.47	17.41	9.13	13
P21-F2	U-5 Pu	G	450 ± 5	368 ⁺³ ₋₅	0.72	3.47	17.06	11.37	16

a. See Table 9.2 for compositions.

b. AC: As Cast

G: 1000 °C - 24 hr H₂O Quench

A: 640 °C - 24 hr - Furnace Cool

c. Based on Reactor Data

BNWL-1144

TABLE 9.4. *Irradiation History and Swelling of Specimens in Capsule P-22
Irradiated at 550 °C and 1000 psi*

Sample	Fuel Material (a)	Met. (b) State	Irradiation Temp, °C		Burnup (c)		As-Irr. Density, g-cm ⁻³	% Swelling, $\left(\frac{P_i - P_f}{P_f}\right) \times 100$	"R" % Swelling at.% BU
			Up	Down	at.%	f-cm ⁻³ x 10 ⁻²⁰			
P22-A0	U-14 Pu-12 Zr	AC	498 ⁺¹⁵ ₋₁₀	342 ⁺¹⁰ ₋₁₂	0.66	2.80	14.44	7.06	11
P22-A1	U-14 Pu-12 Zr	G	498 ⁺¹⁵ ₋₁₀	342 ⁺¹⁰ ₋₁₂	0.66	2.80	14.47	6.42	9.7
P22-A2	U-0 Pu	G+A	498 ⁺¹⁵ ₋₁₀	342 ⁺¹⁰ ₋₁₂	0.67	3.22	16.83	9.98	15
P22-B0	U-5 Pu	AC	585 ⁺¹⁵ ₋₁₀	455 ± 15	0.65	3.13	18.07	5.31	8.2
P22-B1	U-5 Pu	G	585 ⁺¹⁵ ₋₁₀	455 ± 15	0.65	3.13	17.98	5.78	8.9
P22-B2	U-0 Pu	G+A	585 ⁺¹⁵ ₋₁₀	455 ± 15	0.67	3.22	17.62	5.11	7.6
P22-C1	U ₃ Si (5014)	E	515 ⁺¹¹ ₋₁₂	480 ± 17	0.19	0.78	9.47	66	350
P22-C2	U ₃ Si (506)	E	515 ⁺¹¹ ₋₁₂	480 ± 17	0.18		9.14	68	380
P22-D0	U-0 Pu	AC	590 ⁺³⁰ ₋₃	480 ± 17	0.67	3.22	18.13	4.41	6.6
P22-D1	U-0 Pu	G	590 ⁺³⁰ ₋₃	480 ± 17	0.67	3.22	17.65	7.59	11
P22-D2	U-0 Pu	G+A	590 ⁺³⁰ ₋₃	480 ± 17	0.67	3.22	17.56	8.14	12
P22-E1	U ₃ Si (501)	E	594 ⁺²² ₋₆	452 ± 15	0.37	1.53	14.53	7.6	21
P22-E2	U ₃ Si (502)	E	594 ⁺²² ₋₆	452 ± 15	0.36	1.46	13.27	15.8	44
P22-F0	U-14 Pu-12 Zr	AC	560 ⁺²³ ₋₁₃	385 ± 14	0.66	2.80	14.41	7.43	11
P22-F1	U-5 Pu	AC	560 ⁺²³ ₋₁₃	385 ± 14	0.65	3.13	17.69	7.63	12
P22-F2	U-5 Pu	G	560 ⁺²³ ₋₁₃	385 ± 14	0.65	3.13	17.73	7.28	11

a. See table 9.2 for compositions.

b. AC: As Cast

A: 640 °C - 24 hr - Furnace Cool

E: 800 °C for several days - Furnace Cool

G: 1000 °C - 24 hr - H₂O Quench

c. Based on Reactor Data

BNWL-1144

TABLE 9.5. *Irradiation History and Swelling of Specimens in Capsule P-23
Irradiated at 625 °C and 1000 psi*

Sample	Fuel Material (a)	Met. (b) State	Irradiation Temp, °C		Burnup (c)		As-Irr. Density, g-cm ⁻³	% Swelling, $\left(\frac{P_i - P_f}{P_f}\right) \times 100$	"R" % Swelling at.% BU
			Up	Down	at.%	f-cm ⁻³ x 10 ⁻²⁰			
P23-A0	U-14 Pu-12 Zr	AC	571 ⁺ ₋₁₄ 9	422 ± 4	0.66	2.80	14.26	8.20	12
P23-A1	U-14 Pu-12 Zr	G	571 ⁺ ₋₁₄ 9	422 ± 4	0.66	2.80	14.36	7.31	11
P23-A2	U-0 Pu	G+A	571 ⁺ ₋₁₄ 9	422 ± 4	0.67	3.22	14.88	28	41
P23-B0	U-5 Pu	AC	625 ± 7	534 ⁺ ₋₁₀ 3	0.65	3.13	19.01	5.55	8.5
P23-B1	U-5 Pu	G	625 ± 7	534 ⁺ ₋₁₀ 3	0.65	3.13	17.95	6.02	9.3
P23-B2	U-0 Pu	G+A	625 ± 7	534 ⁺ ₋₁₀ 3	0.67	3.22	18.18	4.51	6.7
P23-C1	U ₃ Si (5014)	E	560 ⁺ ₋₅ 7	542 ⁺ ₋₁₁ 3	0.19	0.78	13.61	15	79
P23-C2	U ₃ Si (506)	E	560 ⁺ ₋₅ 7	542 ⁺ ₋₁₁ 3	0.18	0.72	12.61	21	120
P23-D0	U-0 Pu	AC	621 ⁺ ₋₂ 3	540 ⁺ ₋₁₆ 3	0.67	3.22	18.33	3.71	5.5
P23-D1	U-0 Pu	G	621 ⁺ ₋₂ 3	540 ⁺ ₋₁₆ 3	0.67	3.22	18.28	3.56	5.3
P23-D2	U-0 Pu	G+A	621 ⁺ ₋₂ 3	540 ⁺ ₋₁₆ 3	0.67	3.22	18.04	5.21	7.8
P23-E1	U ₃ Si (501)	E	620 ⁺ ₋₄ 6	517 ⁺ ₋₁₁ 5	0.37	1.53	15.11	3.57	9.6
P23-E2	U ₃ Si (502)	E	620 ⁺ ₋₄ 6	517 ⁺ ₋₁₁ 5	0.36	1.46	14.82	2.77	7.7
P23-F0	U-14 Pu-12 Zr	G	570 ⁺ ₋₆ 12	437 ⁺ ₋₈ 4	0.66	2.80	14.42	6.66	10
P23-F1	U-5 Pu	AC	570 ⁺ ₋₆ 12	437 ⁺ ₋₈ 4	0.65	3.13	18.05	5.10	7.8
P23-F2	U-5 Pu	G	570 ⁺ ₋₆ 12	437 ⁺ ₋₈ 4	0.65	3.13	17.99	5.73	8.8

a. See Table 9.2 for compositions.

b. AC: As Cast

A: 640 °C - 24 hr - Furnace Cool

E: 800 °C - for several days - Furnace Cool

G: 1000 °C - 24 hr - H₂O Quench

c. Based on Reactor Data

BNWL-1144

The split tubular specimens were machined from rods that had been cast and heat treated to transform the U_3Si_2 and U to U_3Si .

Burnups were calculated from reactor calorimetric data or from calculated flux values and the length of irradiation; these burnups will be verified via ^{137}Cs and flux dosimetry analyses.

U-0 Pu, U-5 Pu, and U-14 Pu-12 Zr Specimens

The specimens of the above materials recovered from capsules P-22 and P-23 appeared to be in excellent condition. A few of the U-0 Pu and the U-5 Pu specimens showed evidence of swelling around the identification holes, but no gross swelling was noted. Specimen P-22-F1 (U-5 Pu) showed some marked distortion at one end that could have been caused by irradiation growth.

The U-14 Pu-12 Zr specimens irradiated in capsule P-21 appeared to be in good shape, but the U-0 Pu and U-5 Pu specimens were generally quite distorted by irradiation growth. "R" values were quite low for all the specimens, indicating that a 1000 psi pressure can be quite effective in restraining swelling in these fuel materials.

U_3Si Specimens

Visual examination of specimens recovered from capsules P-21, P-22, and P-23 indicated little swelling had occurred, but density measurements indicate large volume increased. Two specimens, P-23-C1 and P-23-C2, were swollen at only one end. This observation indicates that swelling in U_3Si is quite temperature sensitive. The density data indicate swelling is greater at lower temperatures. A plot (Figure 9.1) of "R" ($\% AU/V \div at\% BU$) as a function of irradiation temperature shows a broad maximum between 400 °C and 600 °C. Metallographic examinations have confirmed the low volume increases ($R = 5-8$) of specimens irradiated at 50 psi between 675 °C and

FIGURE 9.1. Irradiation Swelling of U_3Si

and 710 °C. These preliminary data indicate swelling in U_3Si shows the same temperature dependence as uranium metal; additional metallography will be required to verify the magnitude and to determine the nature of the swelling.

MIXED NITRIDES FUEL IRRADIATION - R. K. Marshall

A test has been designed to study the effect of irradiation on the effective thermal conductivity and structural changes which occur during the short-term irradiation of ($U_{0.8} Pu_{0.2}$) pellet fuel. To determine the effect of irradiation on the effective thermal conductivity, the changes in the core temperature of a pellet are monitored during irradiation. The test is so designed that any change in temperature results from a change in the effective thermal conductivity of the fuel. The test capsule was irradiated for 12 days in the specialty irradiation facility at KW Reactor. The core temperature of the pellet was initially 700 °C and then during the first several hours gradually dropped to and remained around 625 °C for the rest of the test. The predicted temperature for the pellet core was 720 °C. Postirradiation examination revealed severe fragmentation of the pellets. The results of metallographic examination were inconclusive concerning the short-term irradiation effects on the microstructure of the fuel. Burnup analysis is being made from which the thermal conductivity of the fuel will be determined.

ATR GAS LOOP OPERATION AND MAINTENANCE

No Contribution
This Quarter

FAST REACTOR DOSIMETRY AND DAMAGE ANALYSIS

R. E. Nightingale

EBR-II DOSIMETRY TEST - J. L. Jackson, W. N. McElroy,
J. A. Ulseth, L. S. Kellogg, and F. S. Kirn

Reaction-rate ratios can be used to identify changes in neutron spectra, since monitors differ in their response to different neutron energies. For example, at core center the 90% response ranges of the reactions is from 0.038 to 3.0 MeV for $^{235}\text{U}(\text{n},\text{f})$, from 2.1 to 7.9 MeV for $^{54}\text{Fe}(\text{n},\text{p})$, from 1.4 to 6.4 MeV for $^{238}\text{U}(\text{n},\text{f})$, and from 0.003 to 1.2 MeV for $^{58}\text{Fe}(\text{n},\gamma)$. A change in the ratio of reaction rates indicates a change in the spectrum which must be taken into account in the analysis of both monitor and irradiation damage data.

Additional data from the recent PNL-ANL dosimetry test⁽¹⁾ are provided in Table 11.1. Listed are either the fission rates or saturated activities of the different monitors irradiated at core center midplane at two power levels. Since the experimental conditions were otherwise identical, the ratios of the reaction rates at low power to those at high power should be the same for all reactions. This is not the case. For example, for the $^{54}\text{Fe}(\text{n},\text{p})$ reaction the ratio is 1.67×10^{-3} , while for the $^{235}\text{U}(\text{n},\text{f})$ reaction the ratio is 1.82×10^{-3} . The difference between the two of approximately 8% is three times larger than the standard error for the reactions. Such a significant difference implies a change in spectrum between the high- and low-power tests.

Changes in the ratio of reaction rates for different monitors also indicate spectral shifts at different reactor positions. Data were previously reported⁽²⁾ for the radial positions of EBR-II midplane. Figure 11.1 provides ratios for the axial direction above midplane in Row 2. The reaction rate data used to calculate the ratios in Figure 11.1 was obtained from pin 18,

TABLE 11.1. Comparison of High and Low Power Interaction Rates

Reaction	Row 2 - Midplane Saturated Activities, ^(a) dps/nucleus				Ratio Low/ High ($\times 10^3$)
	High Power, 50 MW	1 σ % Std Error, $\pm$	Low Power	1 σ % Std Error, $\pm$	
$^{235}\text{U}(\text{n},\text{f})$	2.90×10^{-9}	1.65	5.28×10^{-12}	1.82	1.82
$^{238}\text{U}(\text{n},\text{f})$	1.81×10^{-10}	5.12	3.24×10^{-13}	0.63	1.79
$^{237}\text{Np}(\text{n},\text{f})$	1.26×10^{-9}	1.31	2.34×10^{-12}	4.16	1.85
$^{232}\text{Th}(\text{n},\text{f})$	4.74×10^{-11}	--	8.18×10^{-14}	2.00	1.72
$^{54}\text{Fe}(\text{n},\text{p})$	3.74×10^{-11}	0.89	6.26×10^{-14}	0.74	1.67
$^{58}\text{Ni}(\text{n},\text{p})$	5.21×10^{-11}	2.16	8.69×10^{-14}	1.43	1.67
$^{46}\text{Ti}(\text{n},\text{p})$	4.89×10^{-12}	1.84	8.63×10^{-15}	(b)	1.76
$^{27}\text{Al}(\text{n},\alpha)$	2.75×10^{-13}	(b)	4.85×10^{-16}	(b)	1.76
$^{238}\text{U}(\text{n},\gamma)$	3.08×10^{-10}	(b)	6.20×10^{-13}	(b)	2.02
$^{58}\text{Fe}(\text{n},\gamma)$	8.67×10^{-12}	1.81	1.66×10^{-14}	2.31	1.91
$^{45}\text{Sc}(\text{n},\gamma)$	3.08×10^{-11}	(b)	6.72×10^{-14}	(b)	2.18
$^{63}\text{Cu}(\text{n},\gamma)$	4.48×10^{-11}	5.78	7.76×10^{-14}	4.38	1.73
$^{197}\text{Au}(\text{n},\gamma)$	4.97×10^{-10}	(b)	1.35×10^{-12}	(b)	2.72
$^{59}\text{Co}(\text{n},\gamma)$	3.14×10^{-11}	(b)	8.06×10^{-14}	(b)	2.57
$^{23}\text{Na}(\text{n},\gamma)$	1.08×10^{-12}	(b)	1.94×10^{-15}	(b)	1.80

(a) Fissions/sec/nucleus for fission foils.

(b) Only one or two monitors were available and no standard error could be calculated. Based on the precision obtained for the other reactions and the consistency in the data as compared with other axial and radial positions, we estimate the 1 σ std error to be less than $\pm 7\%$.

FIGURE 11.1. Ratios for the Axial Direction Above Midplane in Row 2

subassembly X046, position 2B1, irradiated during Run 31F. The $^{235}\text{U}(n,f)$ to $^{54}\text{Fe}(n,p)$ ratio shows no spectral shift axially up to ~10 cm above midplane but decided changes beyond. The same effect is shown by the other ratios. Thus, it is inferred that for this reactor run the spectrum was constant in Row 2 for approximately 10 cm (4 in.) above midplane.

DAMAGE FUNCTIONS AND DATA CORRELATION - W. N. McElroy and C. Z. Serpan, Jr.*

Introduction

A previous report⁽³⁾ discussed the development and study of a method for generating energy-dependent damage functions for the correlation and prediction of neutron induced property changes.

The method was demonstrated by using data on ductile-brittle transition temperature increase (ΔTT) as a function of total neutron fluence for irradiations at <450 °F (232 °C) of ASTM A212-B and A302-B steels in widely differing neutron environments.^(3,4) These data with their respective neutron spectra were used to derive a damage function. An advantage of this semi-empirical method is that damage caused by mechanisms other than displacement-related processes are included in the damage function.

Reference 5 presents the results of a new set of experimental data conforming to the irradiation conditions of the derived damage function (material, temperature, and fluence). The results of this experiment help to establish the validity of the damage function.

Experimental Data

The new data on transition-temperature increase were measured at the 30 ft-lb level, for a 6-in. thick plate of

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A302-B manganese-molybdenum pressure-vessel steel. The irradiations were conducted in a reactor facility designated GMWC (Figure 11.2). Sealed capsules were contained within special shields that permitted $<240^{\circ}\text{F}$ (116°C) water to pass over the capsules, but which altered the reactor spectrum incident upon the capsules to yield neutron-fluence ratios of 9, 5, and 1.8 to 1 thermal-to-fast ($>0.5\text{ MeV}$). Three distinct fluence levels were attained for each set of capsules representing all three neutron energy ratios. The three reactor spectra were assigned spectrum reference numbers 39, 40, and 41, respectively, for comparison with the previously reported spectra (1 through 21 in Figure 11.2) used to derive the damage function.

The data are plotted versus total neutron fluence in Figure 11.2.* Considering experimental uncertainties, the three data points for each of the three thermal-to-fast ratios in the GMWC facility conform well with the arbitrarily placed trend lines superimposed on the figure.** The trend pattern is defined as a fourfold increase in total fluence for a 100°F (56°C) ΔTT in A302-B steel for irradiations $<450^{\circ}\text{F}$ (232°C). This trend was suggested by Rossin⁽⁶⁾ and was subsequently used as a reasonable basis for extrapolation of data to other fluence and embrittlement levels.⁽³⁻⁵⁾ Its use is validated by the results of the GMWC data. Use of the straight-line trend behavior for data $\leq\Delta\text{TT } 400^{\circ}\text{F}$ (222°C) is further justified because full radiation embrittlement saturation for this steel irradiated at $<450^{\circ}\text{F}$ (232°C) has not been observed.

* Defined as the sum of the thermal, intermediate, and fast components of the neutron spectrum.

** The Charpy V specimens for the GMWC test exhibited greater than average scatter, thus decreasing the overall accuracy of the final ΔTT values, see Reference 5.

FIGURE 11.2. Charpy-V 30 ft-lb Transition Temperature Increases Versus Total Neutron Fluence for Irradiations <450 °F (232 °C) of A302-B Steel

Damage Function-Fluence Relationship

It can be seen from Figure 11.2 that the embrittlement in A302-B steel has a neutron-energy dependence. From such evidence, it is becoming evident that data obtained from diverse spectral environments can never be completely correlated in a simple plot of ΔTT versus neutron fluence for neutrons greater than any energy. However, for those systems in which most of the damaging neutrons are above a few tenths MeV, good correlation can be expected on a plot of ΔTT versus neutron fluence for neutrons greater than a specific energy such as 0.5 or 0.1 MeV.⁽⁷⁾ To achieve better correlations, it is necessary to plot the ΔTT data versus other parameters which reflects those mechanisms other than displacements.^{(8-11)*}

Correlation can be improved by a damage model that incorporates displacement effects as a starting point and includes provisions for other damage or "annealing" mechanisms.

The data from Figure 11.2 were used to derive an empirical damage function. That absolute damage function for the calculation of the total fluence required to cause a 200 °F (111 °C) ΔTT in A302-B steel for irradiations at temperatures <450 °F (232 °C) is presented graphically in Figure 11.3. A modified version of the SAND-II Code⁽¹²⁾ was used to obtain the damage function solution $G(E)$ for a set of integral equations of the form:

$$S_j = \iint G(E) \cdot \phi_j(E,t) dE dt \quad , \quad (1)$$

where S_j is a measured integral property change such as ΔTT for a specified material, irradiated for a time t in the j^{th} neutron environment (at constant temperature T), and $\phi_j(E,t)$ is the corresponding j^{th} neutron differential spectrum.

* Displacements are generally thought to be associated with high-energy neutron effects.

FIGURE 11.3. Absolute $G(E)$ Damage Function Solution for a 200 °F (111 °C) Transition Temperature Increase in A302-B Steel for Irradiations <450 °F (232 °C)

The derived damage function can be used with multigroup reactor physics calculations to obtain the fluence (and appropriate energy limits of damaging neutrons) required to cause the property change in any spectrum. Equation (1) is rewritten:

$$S = \phi t \sum_{i=1}^m G_i \cdot \phi_i \quad (2)$$

where

$$\sum_{i=1}^m \phi_i = 1 \quad , \quad (3)$$

and ϕ_i is the normalized integral flux within the i^{th} group, ϕt is the fluence required to cause the property change specified in the derivation of $G(E)$, and G_i is the corresponding group averaged $G(E)$ value.

To use the $G(E)$ function would then require knowledge of the neutron spectrum at the reactor location of interest. Group-averaged values of this spectrum would be multiplied by group-averaged values of the damage function, and Equation (2) would be solved for ϕt according to:

$$\phi t = S \left[\sum_{i=1}^m G_i \cdot \phi_i \right]^{-1} \quad (4)$$

The fluence obtained from Equation (4) would be that required to cause a change of 200 °F (111 °C) ΔT in A302-B steel in the spectrum of interest. It was found (Table 11.2) that a single set of group-averaged values of the damage function could be used instead of the exact form, Figure 11.3, without introducing more than about a 5 to 10% variation in the calculated values of fluences for most reactor spectra.

In the development of the damage function of Figure 11.3, it was recognized that the reactor environments were not sufficiently different to provide a reliable solution for the

TABLE 11.2. Group Averaged Values for Absolute $G(E)$ for a 200 °F (111 °C) ΔTT in A302-B Steel Irradiated at <450 °F (232 °C) and Application to Different Spectra

Lower Energy Limit for Group i MeV	Group Averaged Values G_i (°F/n-cm ⁻²)	CVTR Spectrum 1, (154:1 Thermal:>0.5 MeV)			GMWC Spectrum 39, (9:1 Thermal:>0.5 MeV)		
		ϕ_i	$G_i \cdot \phi_i$	% Contribution by Energy Groups	ϕ_i	$G_i \cdot \phi_i$	% Contribution by Energy Groups
7.79	1.81×10^{-17}	4.35×10^{-5}	7.87×10^{-22}		1.29×10^{-4}	2.33×10^{-21}	
6.07	1.89	1.24×10^{-4}	2.34×10^{-21}		4.76	9.00	
4.72	1.97	2.18	4.29		1.60×10^{-3}	3.15×10^{-20}	
3.68	2.07	3.36	6.96		1.68	3.48	
2.87	2.10	5.60	1.18×10^{-20}		2.31	4.35	
2.23	1.97	7.59	1.50		5.10	1.00×10^{-19}	
1.74	1.87	7.52	1.41		6.54	1.22	
1.35	1.66	6.40	1.06		8.00	1.33	
1.05	1.33	5.85	7.78×10^{-21}	42%	8.34	1.11	85%
8.21×10^{-1}	1.01	5.29	5.34		8.95	9.04×10^{-20}	
6.39	9.57×10^{-18}	7.01	6.71×10^{-21}		9.22	8.88	
4.98	7.95	6.28	4.99		9.21	7.73	
3.88	8.08	4.23	3.42		7.48	6.04	
3.02	6.27	5.91	3.71		3.11	5.09	
2.35	4.47	5.85	2.62		7.68	3.43	
1.83	3.47	7.03	2.44		6.74	2.34	
4.14×10^{-7}	3.00×10^{-19}	8.03×10^{-2}	0.24×10^{-19}	10%	3.48×10^{-1}	10.4×10^{-20}	9%
1.00×10^{-10}	1.32×10^{-19}	9.11×10^{-1}	1.20×10^{-19}	48%	5.60×10^{-1}	7.39×10^{-20}	6%
		$\sum_{i=1}^{18} G_i \cdot \phi_i = 2.47 \times 10^{-19}$			$\sum_{i=1}^{18} G_i \cdot \phi_i = 119 \times 10^{-20}$		
		$\phi \cdot t = \frac{200 \text{ °F}}{2.47 \times 10^{-19}} = 8.10 \times 10^{20} \text{ n/cm}^2$			$\phi \cdot t = \frac{200 \text{ °F}}{119 \times 10^{-20}} = 1.68 \times 10^{20} \text{ n/cm}^2$		

damage model in the intermediate energy region between $\sim 10^{-7}$ and $\sim 2 \times 10^{-2}$ MeV. In this region, therefore, the function is essentially the assumed input model. For this reason, no attempt has been made in these studies to generate or use detailed structure in this region. Furthermore, for the thermal-energy region, the magnitude of the derived damage function for A302-B steel may be too low.

The approximating procedure used in the GMWC experiment (and in References 3 to 5) for defining the magnitudes of thermal, intermediate, and fast components of the neutron spectrum has been compared with a more exact procedure.⁽¹²⁾ This comparison revealed that the approximating procedure may have yielded high thermal fluence results. This implies that the derived damage function may be too low in the thermal energy region. Future re-evaluations of these data may reveal, therefore, that the damage from thermal neutrons is greater than the present results for A302 steel.

Damage Function Applications

Examples of the application of Equation (4) and group-averaged values, G_i , of the damage function are considered in Table 11.2, using the group integral flux values for two of the very different reactor irradiation locations (Figure 11.2). The left column under both spectral listings gives the group fluxes as normalized to one neutron; the middle column is the product of the group flux and the group averaged damage function. The summation of these products for both spectra are then divided into 200 °F to yield the total fluence for a 200 °F ΔT in A302-B steel for irradiations < 450 °F. The right column under each listing shows the thermal ($< 4.14 \times 10^{-7}$ MeV), intermediate (4.14×10^{-7} MeV $< E < 0.183$ MeV), and fast (> 0.183 MeV) energy region percent contribution to the total damage.

The approximate energy limits of damaging neutrons can be inferred from the contributions of the groups to the total property change.

The total fluences in Table 11.2 were used to plot the abscissas of the respective data points appearing for these spectra in Figure 11.4. (A similar procedure was used for the other GMWC data points.) It was found that the previously derived damage function was applicable to the GMWC data. From Figure 11.2 the "measured" total fluences to cause a 200 °F ΔT in A302-B steel for all three GMWC spectra were found and plotted versus total fluence values determined by the damage-function-calculated process shown in Table 11.2. The measured and damage-function-calculated total fluences are compared in Figure 11.4. Perfect correlation is achieved when all points fall on the solid line; that is, using the damage function to calculate total fluences that agree exactly with measured fluences for any reactor environment. The ratios of measured to calculated fluences (Figure 11.4) have a 15.2% standard deviation about a line representing perfect correlation. The GMWC data points (spectrums 39, 40, and 41) compare favorably with this deviation. This indicates that the damage function, considering the limitations previously indicated, is applicable to the GMWC spectra shapes.

Conclusions

This study has demonstrated the applicability of group-averaged values of a previously derived damage function to the neutron embrittlement and related total fluence for three different neutron spectra. The three GMWC spectra used to test the damage function were within the extremes of ratios of thermal to fast (>0.5 MeV) neutrons used for the function development but are, nevertheless, significant because they cover the most reasonable range of neutrons in many test and power reactors. The good agreement between damage-function-calculated

FIGURE 11.4. Verification of the $G(E)$ Damage Function by the Graphite Moderated, Water Cooled Reactor (GMWC) Irradiation Data

fluences and the measured fluences from the new experimental data has helped to establish the damage function technique as being valid for its intended purpose.

A final conclusion of this study is that detailed and accurate neutron dosimetry measurements are necessary for good correlations of irradiation effects data for diverse reactor environments. Good detail in spectral calculations and foil measurements is required for both low and high energy neutrons.

COMPUTER SIMULATION OF RADIATION DAMAGE - D. G. Doran

Recent empirical studies at Battelle-Northwest of the neutron energy dependence of radiation damage in some ferritic steels suggest that thermal neutrons are contributing more than expected to the damage state. The particular effect studied was the increase of the ductile-brittle transition temperature.

The most likely mechanisms by which thermal neutrons can affect mechanical properties are impurity effects caused by transmutations, and displaced atoms caused by recoils from (n,γ) reactions. The latter, being of low energy, are generally discounted relative to the displacements produced by scattering of faster neutrons.

By means of computer simulation, a study is being made of the production of low-energy displacement spikes and their annealing characteristics. The average (n,γ) recoil energy in iron is about 400 eV, although the spectrum of recoils extends to 1000 eV.

The 0 °K configurations for nine isolated 400 eV spikes have been produced with the CASCADE code. Two irradiation temperatures are presently being simulated with the ANNEAL code. As in previously reported^(2,11) computer experiments with more energetic spikes, the low-temperature case involves only interstitial migration, whereas the high-temperature case includes vacancy migration as well as an expanded recombination space for interstitial-vacancy pairs.

The CASCADE runs produced an average of 4.5 displacements per spike, corresponding to an effective Kinchin and Pease displacement energy of 44 eV, in agreement with Beeler's results⁽¹³⁾ for higher energies. No interstitial clusters were produced, but vacancy clusters averaged one per spike including 4 divacancies, 3 trivacancies, and 2 tetravacancies.

A low-temperature anneal has been performed for 100 time steps (an average of 10 jumps per single interstitial). The average number of displacements per spike has decreased to 2.4, the total vacancy cluster population has been reduced to 2 divacancies and 2 trivacancies, and 1 di-interstitial and 1 tri-interstitial have formed. The annealing simulation will be continued.

The elevated temperature simulation was also begun with 100 interstitial time steps, the vacancy motion to follow. However, the increased recombination space has already reduced the average displacements per spike to 1.2, and the only clusters among the 9 spikes are 2 divacancies. Clearly, further annealing of the individual spikes will not result in formation of clusters.

COMPUTER SIMULATION OF IRRADIATION DISPLACEMENT CASCADE FORMATION - G. E. Russcher

A third generation cascade-type computer code is being programmed. SLIDES (Strained Lattice Irradiation Damage Experiment Simulation) is a program designed to simulate, by computer experiment, the generation of irradiation damage in an imperfect or strained fcc lattice structure. It is being designed to study basic physical problems in an environment that more nearly simulates austenitic stainless steel in a fast-reactor irradiation.

Two main objectives for the development of this program are the capabilities of calculating damage generation for:

(1) the highest energy neutrons of a fast irradiation spectrum and their primary knock-on atoms (PKA) and (2) high irradiation flux and fluences of greater importance to structural materials problems. About half of the PKAs produced in a fast reactor irradiation are above 20 keV, hence they contribute an important part to the generation of irradiation damage. However, the existing CASCADE program is limited to computations for PKA energies only up to 20 keV. At very high irradiation fluences and fluxes, the generation of irradiation damage is influenced by neighboring lattice displacements, the interaction between these displacements and impurity inclusions, displacement loops, and other extended defect clusters. Thus, the simulated generation of displacements in a previously irradiation-damaged lattice structure is important to a study of a high fluence fast reactor environment.

The SLIDES program incorporates several computational advances. The following three are perhaps the most important:

- Binary atomic collisions are calculated in a chronological sequence.
- Refined interatomic potentials are used to compute atomic interactions.
- Energy lost to electron excitation in inelastic atomic collisions is accounted for.

During the formation of a lattice displacement cascade, different branches of the cascade interfere with one another. This effect on the damaged lattice structure illustrates the importance of the chronological sequence to the formation of a displacement cascade. Interatomic potentials are also important to damage calculations for they determine: (1) the energy necessary to create a displacement, (2) separation distances between a pair of stable defects, and (3) the energy transferred during close interactions where atoms cannot be represented by

hard spheres. These physical considerations are being incorporated by adding interatomic potentials developed by R. A. Johnson and O. B. Firsov to the screened coulomb and Born-Mayer interatomic potentials presently used by CASCADE. Another consideration important to all displacement cascades is the incidence of close atomic interactions resulting in energy lost due to electron shell overlap. This electronic energy loss can influence both the number and distribution of displacements, and hence the damage state produced. These refinements in the computational basis of SLIDES will make the high energy lattice damage calculations more applicable to the simulation of irradiation of fast reactor materials.

Some of the specific goals of this computer program development are:

- Calculate the displacement cascades resulting from PKAs with energy up to 700 keV.
- Simulate the lattice displacement damage of austenitic stainless steel for an irradiation fluence of 10^{23} n/cm².
- Allow lattice defects to recombine on the basis of annealing statistics for the irradiation temperature.
- Reduce computations time by an order of magnitude below that presently required.

The preliminary design of the SLIDES program is complete. It has also been portrayed as a computer flow chart, and is now being programmed.

COMPUTER EXPERIMENTAL STUDY ON CARBON INCLUSIONS IN GRAIN
BOUNDARIES IN FCC GAMMA - IRON - R. E. Dahl, Jr.,
J. R. Beeler, Jr., and R. D. Bourquin

Small concentrations of interstitial impurity atoms markedly affect the physical and mechanical properties of polycrystalline metal specimens. The mechanisms for these effects are difficult to determine since the macroscopic behavior is

caused by atomic-scale interactions. Computer experimentation is well-suited to this study since many parameters can be easily varied to achieve a thorough analysis not possible in a laboratory and the scale is at or beyond the limit of experimental observation. The results are credible if there is agreement between computer experiment results and measurements for quantities such as grain-boundary energies, vacancy migration and formation energies, and elastic constants.⁽²⁾

The computer code GRAINS used in this study uses the pseudo-dynamic method of experimentation.⁽¹⁴⁾ Atoms are moved to their equilibrium positions according to classical equations of motion.⁽¹⁵⁾ The experiments in this study were conducted in a computational cell containing a 6° tilt boundary. Carbon atoms were inserted into the misfit region of this boundary and the ensuing structural and energy changes observed. Interaction between carbon and iron atoms were calculated by the potential function developed by Johnson.⁽¹⁶⁾ Carbon interactions were computed with a Born-Mayer repulsive potential which went to zero with zero slope at the carbon-carbon distance given by Slater.⁽¹⁷⁾

Structural changes caused by inserting a single carbon atom into the misfit region of a 6° tilt grain boundary can be seen by comparing Figures 11.5 and 11.6. These figures which are direct computer output, illustrate the structure before and after the addition of a carbon atom. The symbols in these figures denote the position and type of atoms in the computational cell. The exact location of atoms is at the end of the fiducial marks which extend into the figures. Hexagons denote movable host (i.e., iron) atoms; squares denote the immovable host atoms which comprise the cell boundary; and triangles denote carbon atoms.

The plane which is observed in the figures is a (100) plane for fcc γ -iron. The lattice constant $3.646 \text{ \AA}$ is the

THIS PLOT IS OF AN FCC LATTICE VIEWED IN THE (100) PLANE
 GRAIN BOUNDARY AT $x = 11$. IS FOR A ROTATION OF 6.00 DEGREES
 THIS PLANE IS: $z = 4$

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FIGURE 11.5. 6° Boundary Before Carbon Atom Added (Plane $z = 4$)

THIS PLOT IS OF AN FCC LATTICE VIEWED IN THE (100) PLANE
 GRAIN BOUNDARY AT $X = 11$. IS FOR A ROTATION OF 6.00 DEGREES
 THERE ARE 1 INTERSTITIALS IN THIS LATTICE
 THIS PLANE IS: $Z = 4$

Neg PNL0692780-2

FIGURE 11.6. 6° Boundary with Carbon Atom Added to Misfit Region (Plane $z = 4$)

observed value at 910 °C, which is the α to γ transition temperature. The grain boundary lies in the (010) plane and is simulated to extend infinitely in the $\langle 100 \rangle$ and $\langle 001 \rangle$ directions through periodic boundaries constructed on the (100) surfaces of the computational cell. Seven (100) plane "layers" comprised the cell used to study point defects, so that the resulting strain fields could be accommodated within the cell and the boundaries would not affect the results.

The effect of the presence of an impurity atom is striking. The hole or misfit in the boundary (see Figure 11.6) is essentially healed because of the bonding of the carbon atom with the neighboring iron atoms. The introduction of the carbon lowered the energy of the computational cell appreciably, thus causing a more ordered structure.

Several more carbon atoms were introduced into the grain boundary to determine how many could be accommodated. Five atoms were finally introduced into the boundary in a region which was symmetric about the center plane of the cell, and extended one interplanar distance in each direction $\langle 100 \rangle$. The energy changes accompanying these additions are seen in Figure 11.7. Note the large reduction in energy (healing) which accompanied insertion of the first carbon atom. The energy changes accompanying subsequent carbon additions were less, but still appreciable. Relaxation occurred each time, indicating that a sizable carbon inclusion can easily be accommodated in the grain boundary and that this is a favored position. The misfit regions apparently then can have an atmosphere analogous to the dislocation atmosphere postulated by Cottrell.⁽¹⁸⁾

Neg PNL0692780-3

FIGURE 11.7. Energy Changes Resulting from Addition of Carbon Atoms into 6° Tilt Boundary

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IRRADIATION DAMAGE TO REACTOR METALS

L. D. Blackburn

REACTOR METALS RESEARCHIrradiation Facilities Operation - A. L. Ward

The purpose of this phase of the program is to provide for the timely accomplishment of irradiation, testing, and subsequent processing of the data obtained from a variety of reactor structural materials. Irradiations will be performed in aqueous, gaseous, and molten metal environments. Elevated temperature irradiations and tests will be emphasized. Special testing techniques, applicable to either irradiated or unirradiated specimens, will be developed.

Facilities Operation

The experiments discharged from the various Engineering Test Reactor (ETR) core positions for Cycle 100 are given in Table 12.1, along with the estimated neutron fluxes and fluences.

The operating history of the ETR and the G-7 hot water loop for Cycles 99 and 100 is given in Table 12.2. The history for the out-of-reactor loop is given in Table 12.3.

The ATR is expected to become available for irradiation experiments in September 1969. ETR G-7 water loop use is expected to be discontinued in favor of the ATR facility at that time. Test Core I was completed on June 6, 1969, and Cores II and III are scheduled for June 15 and July 6, 1969, respectively.

TABLE 12.1. *Summary of Experiments Discharged
at the Conclusion of ETR Cycle 100*

Experiment Designation	No. of Specimens	Type of Specimens	Estimated Flux, n/cm ² /sec (a)	Estimated Fluence n/cm ² (a)
GEH-20-314	36	Zr alloys	5.5×10^{13}	3.1×10^{20}
GEH-20-332	36	Mixed SS	1.04×10^{14}	$\sim 9 \times 10^{20}$
GEH-20-331	36	Mixed SS	1.05×10^{14}	$\sim 9 \times 10^{20}$
GEH-20-299	18	Zr-2	1.06×10^{14}	$\sim 9 \times 10^{20}$
GEH-20-293	36	Zr-2	1.0×10^{14}	$\sim 1.7 \times 10^{22}$

(a) Neutrons with energies greater than 1 MeV.

NOTE: Total specimens discharged, ETR G-7 Loop Cycle 100 = 162.
Total specimens discharged, ETR G-7 Loop to date = 3962.

TABLE 12.2. *Summary of Operating History for ETR and
G-7 Hot Water Loop - ETR Cycles 99 and 100*

Operating History	ETR Cycle Number	
	Cycle 99	Cycle 100
1. Reactor History		
Start up (a)	11-15-68	1-15-69
End of Cycle	1-5-69	2-22-69
Megawatt Days	5690	5781
Effective Days at 175 MW	32.51	33.08
Number of Scrams (b)	5	10
Number of Shutdowns	8	3
2. G-7 Hot Water Loop History		
Maximum Temperature	508 °C	529 °F
Effective Days Above 200 °F	33.9	33.85
Effective Days at Operating Temperature	29.05	31.60
Operating Efficiency, % (c)	95.8	100
Temperature Efficiency, % (d)	85.7	93.3

(a) Includes flux runs at low power.

(b) Power drops below half of full power (175 MW) with immediate recovery.

(c) Effective days of loop operation above 200 °F (95 °C) with respect to effective days of reactor operation at 175 MW.

(d) Effective days of loop operation between 500 and 550 °F (260 and 290 °C) with respect to days of operation above 200 °F (95 °C)

TABLE 12.3. *Summary of Operating History and Discharge Schedule for Out-of-Reactor Loop, ETR Cycles 97 and 98*

1. TIME-TEMPERATURE SUMMARY

<u>Temperature</u>	<u>Time, hr</u>	
	<u>Cycle 97</u>	<u>Cycle 98</u>
200 °C (392 °F)	21.70	34.6
218 °C (424 °F)	9.35	11.0
246 °C (475 °F)	28.45	85.8
282 °C (540 °F)	695.0	623.6

2. SPECIMEN DISCHARGES

<u>Material</u>	<u>Specimen Type</u>	<u>Number</u>
Zry-2	Tensile	28
PDRL-120	Tensile	6
		<u>34</u>

Total specimens discharged to date = 1595

Microstructural Modifications to Stainless Steels and Nickel-Base Alloys - I. S. Levy

The purpose of this program is to optimize the stability of austenitic stainless steels and nickel-base alloys to fast reactor environments through microstructural modifications. These modifications are produced by selected variations in chemical composition and/or by use of thermomechanical treatments.

Last quarter,⁽¹⁾ it was noted that the rate of quench from solution temperature could affect the rapidity of stacking fault hardening at 700 °C in 348 stainless steel; the faster the quench-rate, the sooner hardening occurred. This is presumably due to an increase in the number of potential stacking fault nucleating sites, i.e. dislocations.

This past quarter, an examination was made of another potential technique from increasing the rate of stacking fault formation--tensile straining. The straining treatments involved either strain at room temperature, strain at 700 °C, or strain at 700 °C followed by a half-hour hold at load. After treatment, the specimens were aged at 700 °C, and their rates of increase in strength were monitored by tensile tests. The relative effects on the rate of strength increase between quenching and the various straining treatments can be indicated by the time required at 700 °C to achieve a significant strength increase. Because simple aging will produce a peak increase of approximately 15,000 psi, an increase of 3,000 psi (20% of peak) was considered sufficiently significant and is used as the basis, in Table 12.4 for comparison of these treatments.

TABLE 12.4. *Aging Time at 700 °C to Achieve a 3000-psi Increase in 0.2% Yield Strength for Various As-Treated Conditions*

	Quenching, °C/sec			Prestrain				Prestrain and 1/2 hr Hold	
	2	50	100	3% at 700°C	RT	10% at 700°C	RT	Under Load at 700 °C to 3% Strain	to 10% Strain
Time, hr	108	68	27	11.8	9.7	4.4	3.7	5.5	2

The data show that dislocation generation does increase stacking fault hardening (compare different quench-rates or strain levels). However, this is not the complete story as indicated by the difference between the results of the pre-straining and prestraining plus hold treatments. These latter data indicate that while straining at 700 °C decreases the hardening rate (due to concurrent annealing out of some of the strain-induced dislocations⁽²⁾), straining at 700 °C followed by a half-hour hold under load at 700 °C significantly increases the hardening rate. The recovery effect should presumably still be occurring during the half-hour hold at 700 °C. This should take place because the amount of creep deformation is less than 10% of the total strain produced by the treatment, and recovery

has also been noted during low-strain rate deformation studies at 650 °C.⁽²⁾ Therefore, an additional factor must be playing an important part in this treatment (and, perhaps, as significant but less discernible part in the other strain treatments). Presumably, this factor is a strain-produced increase in vacancy concentration. This would lead to enhanced diffusion of Nb and would account for the more rapid growth of NbC to the critical size required for the nucleation of the stacking faults that have also been observed to a result of prestrain.⁽³⁾

In-Reactor Measurements of Mechanical Properties

E. R. Gilbert, N. E. Harding

The in-reactor measurements program is intended to determine the effects of irradiation on the mechanical properties of reactor structural materials, to establish property data suitable for design purposes, and to consider mechanisms through irradiation effects on macroscopic deformation. The program currently involves conducting in-reactor creep measurements on annealed 304 SS, heat-treated Zr-2.5 Nb, and Zircaloy-2. Capsule development is being pursued in order that program capabilities may be extended.

304 Stainless Steel

Slow, steady, in-reactor creep has been measured in annealed 304 SS at 175 and 300 °C. The rate at 35,000 psi stress is rather insensitive to temperature with an average value of $3 \times 10^{-7} \pm 1 \times 10^{-7} \text{ h}^{-1}$ at both 175 and 300 °C. Upon increasing the temperature to 370 °C, discontinuous yielding was observed, as shown in Figure 12-1. The amplitude of the strain steps is 0.2 to 0.5% strain and occurs at a periodicity of 50 to 100 hr. The initial strain step occurred after a 500 hr incubation period. If a line is drawn through the strain steps, an effective creep rate of $6 \times 10^{-5} \text{ h}^{-1}$ is measured.

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FIGURE 12.1. *In-Reactor Discontinuous Yielding in 304 SS*

The Portevin-LeChatelier effect (serrated stress-strain curve) is observed in 304 stainless steel at temperatures around 400 and 500 °C. In the pneumatically-loaded creep capsule, strain steps are observed instead of the load relaxations that are observed in a screw-driven tensile machine. The discontinuous yielding observed during neutron irradiation in the constant-load creep test may be related to the Portevin-LeChatelier effect. The discontinuous yielding is not observed without the presence of irradiation. The first strain step is fairly easy to rationalize in terms of neutrons displacing solutes from their pinning points along dislocations until the dislocations are free to move. However, successive strain steps require a dislocation nucleation process at constant applied stress with

very little straining. A potential source may be the nucleation of Frank partial dislocations due to the collapse of irradiation-produced point defects in the field of the applied stress. When the Frank partials grow to a critical radius, they can react with Shockley partials to form glide dislocations that are then released to generate a strain step. Thus, it is difficult to account for the succeeding strain steps with the solute pinning interpretation of the Portevin-LeChatelier effect. It is also difficult to visualize the cooperation of about 10^8 dislocations/cm² in simultaneous glide.

Zirconium Alloys

The values of the activation parameters and the stress-dependence of the creep-rate in Zr-2.5 Nb are consistent with the "dislocation climb" model for creep controlled by short-circuited diffusion. The activation energy for creep is similar to that for diffusion of ⁹⁵Zr along dislocations, and the activation area of $3b^2$ is in good agreement with that predicted for the dislocation climb model. The strain rate is proportional to the stress to the 4.8 power which is consistent with the climb model for creep.

The effect of neutron irradiation on increasing the creep-rate can be considered as an irradiation-induced stress assisting the applied stress. In this case, the stress arises from an osmotic force on climbing dislocations resulting from the supersaturation of point defects created by irradiation. Since irradiation creep rates are observed in annealed 304 stainless steel that are of similar magnitudes to those observed in Zr-2.5 Nb at similar stresses and temperatures, the anisotropy of the Zr crystal structure is probably not a prerequisite for irradiation creep.

Measurement of a Zr-2.5 Nb in-reactor creep specimen prior to and following the in-reactor creep test has indicated that the volume of the specimen was increased by approximately

1.4 percent. Previously, it had been assumed that the in-reactor creep was a constant volume process. Approximately 0.6% volume decrease was recovered upon annealing at 500 °C for 4 hours. These results suggest that the resultant jog in the dislocation climb process is the vacancy jog. No cavities were observed in the specimen upon TEM examination.

Formation of Voids in Iron During High-Temperature Neutron Irradiation - G. L. Kulcinski and B. Mastel

It has been recently discovered that high-temperature neutron irradiation of 304 SS and some pure metals results in the formation of voids within the solid. The first voids were reported in 304 SS.^(4,5) Later, voids were also found in pure metals such as Ni,⁽⁶⁻⁸⁾ Cu,⁽⁷⁾ Al,^(7,9) Mo,^(7,10) and V.⁽¹¹⁾ The purpose of this study was to ascertain whether voids can be formed in high purity iron under proper irradiation conditions.

Iron foils of 99.999% purity (metallic elements only), in the form of disks 11 mm in diameter and 75 μ thick, were annealed at 800 °C for 2 hr in a vacuum of 2×10^{-7} torr. They were then irradiated in the EBR-II reactor at 450 °C to a fast neutron fluence of 3×10^{21} n/cm² ($E > 0.1$ MeV). After irradiation, the samples were thinned in a solution of 10 parts glacial acetic acid and one part perchloric acid at >20 °C.

The typical microstructure of the iron after the irradiation is shown in Figure 12.2. The projected shape of the voids is rather non-uniform and the average diameter is 280 Å. The smallest voids observed are approximately 100 Å in diameter and the total density of all the voids is 1×10^{14} /cm³. This latter number was calculated for an assumed average foil thickness of 1000 Å. The calculated volume change is approximately 0.12%.

FIGURE 12.2. Voids in Iron that Has Been Irradiated to 3×10^{21} n/cm² (>0.1 MeV) at 450 °C.

The morphology of the voids is difficult to determine specifically because of the strong (111) texture of the foils and because of the interaction of the Fe foils with the magnetic field of the objective lens. However, it was determined that the edges of the voids are parallel to the $\langle 112 \rangle$ direction for a $\langle 111 \rangle$ foil orientation which is consistent with octahedra bounded by $\{110\}$ planes. The irregular shape of the voids can be accounted for by truncations parallel to the other $\{110\}$ planes.

Calculations were also made to determine if the voids could be filled with neutronically-generated gases. If the gas atoms are under an equilibrium LaPlace pressure of $2\gamma/r$, where γ is surface energy and equal to 1720 ergs/cm²,⁽¹²⁾ one finds that

approximately 200 ppm (atomic) of gas is required to account for the observed cavity size and density. De Pino⁽¹³⁾ has calculated that less than 1 ppm of neutronic gases would be generated during a fast neutron irradiation to 3×10^{21} n/cm². Therefore, it seems rather unlikely that the cavities are gas-filled and it is concluded that they may properly be called voids.

Although the distribution of the voids is rather homogenous within the individual grains, a wide (0.8-1.0 micron) region denuded of voids appears at grain boundaries. Such an effect has been discovered before in Ni and Cu⁽⁷⁾ and has been explained on the basis that the grain boundaries act as sinks for vacancies.

A compilation of the present results and those obtained in other studies is shown in Table 12.5. Iron is the third example of a bcc metal in which voids have been found. The presence of fast neutron-induced voids in both fcc and bcc metals indicates that the phenomenon of void development in metals is quite universal.

An important point to note from Table 12.5 is that in all cases where voids have been discovered, the irradiation temperature has been greater than $0.3 T_m$, where T_m is the melting temperature. The present irradiation of Fe at $0.4 T_m$ is well above this apparent threshold. The physical significance of this threshold is not clear at this time but it is probably connected in some manner with the mobility of vacancies.

Both purity and threshold fluence-level seem to be connected in a rather complex way, as shown by workers at Oak Ridge.⁽⁹⁾ Voids did not form in low purity (99%) Al after 3.5×10^{20} n/cm² at 50 °C, whereas voids did form in high purity Al (99.9999%) irradiated under the same conditions. It was found that irradiation exposure to 1.5×10^{22} n/cm² was required

TABLE 12.5. Summary of Void Formation Data in Pure Metals

Metal	Purity, %	Irradiation Temperature, °C	T/T _m	Fluence × 10 ⁻¹⁹ (E > 1 MeV)	Average Void Diameter, Å	Void Density × 10 ⁻¹⁴ , cm ⁻³	Volume Fraction, %
<u>fcc Metals</u>							
Nickel ⁽⁷⁾	99.997	260	0.31	12	60	200	0.21
	99.997	380	0.38	5.7	83	40	0.11
	99.997	500	0.45	5.7	165	8	0.16
	99.997	575	0.49	6.2	245	2.5	0.17
Nickel ⁽⁸⁾	99.98	370-458	0.38-0.41	2 ^(b)	(a)	(a)	(a)
Copper ⁽⁷⁾	99.999	260	0.39	12	230	3	0.17
Al ⁽⁷⁾	99.999	50	0.35	3.2	270	(a)	(a)
Al ⁽⁹⁾	99	55	0.35	1500	(a)	(a)	(a)
Al ⁽⁹⁾	99.9999	50	0.35	35	(a)	1.3	(a)
<u>bcc Metals</u>							
V ⁽¹¹⁾	Commercial	630	0.41	1700 ^(b)	100	>10	0.05
Mo ⁽⁷⁾	99.99	800	0.37	3	65	100	0.12
Mo ⁽¹⁰⁾	99.98	700	0.34	14	35	200	0.05
Mo ⁽¹⁰⁾	99.98	1000	0.44	13	200	5	0.19
Fe (this study)	99.999	450	0.40	300 ^(b)	280	1	0.12

(a) No information on averages reported

(b) (E > 0.1 MeV)

to form voids in the low purity Al. Similarly, voids were formed in commercial purity vanadium after irradiation at 630 °C to a fluence of 1.7×10^{22} n/cm² irradiation, whereas none were formed in a V-20% Ti alloy irradiated under the same conditions. These observations indicate that the threshold fluence is likely to lower as the purity of the metal is increased and that therefore, voids may form in the present Fe material at fluences even $< 3 \times 10^{21}$ n/cm². Future studies on this relationship may yield important information on the mechanism of void formation.

Small-Angle X-Ray Scattering by Voids in Irradiated Metals

H. E. Kissinger

An X-ray beam transmitted through a material containing an inhomogeneous distribution of electron density is subject to diffraction effects which result in part of the beam being scattered through very small angles. The intensity scattered and the angular distribution of the scattering depends upon the number and size of the inhomogeneous regions and the difference between the electron density of the scattering region and the matrix.

Interpretation of the experimentally determined scattering is most readily done by assuming a matrix of constant electron density, ρ_0 , in which scattering centers of electron density ρ are distributed in a nonregular array. Interparticle interference then does not occur, and the scattering is given approximately by Guinier's formula⁽¹⁾

$$\ln I(\epsilon) = K - \frac{4\pi^2 R_0^2}{3\lambda^2} \epsilon^2, \quad ,$$

where $I(\epsilon)$ = the intensity scattered at angle ϵ , measured in radians,

λ = the X-ray wavelength,

R_0 = a parameter called the "electronic radius of gyration."

If the scattering centers are spheres of radius R , then $R_0^2 = 3/5 R^2$.

The voids that result from elevated temperature irradiation of many metals provide scattering centers that should be ideal subjects for small-angle X-ray studies. As metals are relatively opaque to X-rays, the samples must be in the form of thin foils. The required foils can be prepared by essentially the same techniques used to prepare electron microscopy samples.

To assess the utility and applicability of the small-angle scattering technique to the study of voids, a number of irradiated metals previously examined with the microscope were studied by the X-ray method. The sample types and irradiation conditions are listed in Table 12.6.

TABLE 12.6. Samples for Small-Angle Scattering Studies

<u>Metal</u>	<u>Purity, %</u>	<u>Fluence, (E >1 MeV) n/cm²</u>	<u>Irradiation Temperature, °C</u>
Cu	99.999	1.2×10^{20}	260
Ni	99.997	5.7×10^{19}	500
Fe	99.999	2×10^{21}	450
Zr	99.95	2×10^{21}	450

These samples were thinned to about 3 μ by electrochemical polishing and examined in a Rigaku small-angle goniometer. Crystal monochromated copper K α radiation (1.541 Å) was used. Scattered intensity was measured at 1' intervals from 10' to 30' from the direct beam, the counting interval being 100 sec at each position. Multiple runs were made in each case to eliminate random errors.

The samples were well annealed prior to irradiation and contained large, well-crystallized grains. A sample position could be found by trial for which no Bragg diffraction occurred. The present results are therefore free of double Bragg scattering which is often troublesome in small-angle scattering experiments. All samples, however, contained radiation damage in the form of loops and clusters so that some small part of the

observed scattering may arise from this source. Such scattering effects are small, and are believed negligible except for the zirconium scattering to be discussed later.

The irradiated copper was found to scatter very strongly while iron and nickel showed moderate effects. Zirconium showed extremely weak scattering effects and electron microscopy failed to disclose the presence of voids. The weak scattering observed was therefore presumed to arise from irradiation-induced artifacts other than voids or from hydrides which formed on the surfaces. For the other metals, the scattering was analyzed by the Guinier⁽¹⁴⁾ relation and the equivalent spherical diameter of the scattering centers was determined. These results as compared with those determined by electron microscopy are given in Table 12.7.

TABLE 12.7. *Void Diameters Determined by Small-Angle Scattering and Electron Microscopy*

<u>Metal</u>	<u>R_o, Å</u>	<u>SAS Void Diam, Å</u>	<u>E.M. Void Diam, Å</u>
Cu	105	270	230
Ni	60	150	165
Fe	120	300	280
Zr*	120	~ 300	Not Detected

**Zr scattering probably not due to voids.*

This work demonstrates that small-angle X-ray scattering methods may be used in studies of void formation and growth in irradiated metals. The apparatus is being augmented by an improved data-collection system, and an extensive program of study on the annealing of voids in metals will be carried out by this method.

FAST REACTOR SUPPORTING STUDIESDamage Analysis - J. L. Straalsund and H. R. Brager

The objectives of this effort are to establish the radiation-induced swelling characteristics of FFTF alloys, relate fast-reactor-induced changes in microstructure to corresponding changes in mechanical properties and to provide a basis for extrapolation and interpolation of data using both microscopic modeling and empirical approaches.

Recent experiments concerning the effects of fast reactor irradiation on stainless steels have demonstrated that a considerable volume change occurs during neutron bombardment.⁽¹⁵⁾ Unlike volume changes in fuel materials, swelling in structural alloys and pure metals is due to the condensation of irradiation-produced excess vacancies in the form of three dimensional cavities. United Kingdom investigators have found as much as 6.8% volume change in 316 SS irradiated to 7.8×10^{22} n/cm² total fluence. Volume changes of this magnitude, if not taken into account, are sufficient to cause distortion in reactor components.

Maximum target fluences for FFTF are as high as 10^{24} n/cm². Since such a high fluence cannot be attained in a reasonable time using currently available reactors, design data for high exposures will be based on extrapolation using physically realistic swelling models. The approach taken in this study is to develop swelling data at fluence levels that can be obtained in reasonable times and to use this data to develop the required swelling models.

An equation describing the effect of fluence and temperature on swelling in solution-treated 304 SS has been refined and the results agreed upon by Westinghouse and PNL. The agreed upon equation has the general form:

$$\frac{\Delta V}{V} = A(\phi t)^n [e^{-Q_1/RT} - \alpha e^{-Q_2/RT}] \quad (1)$$

Specifically, the equation is as follows:

$$\frac{\Delta V}{V} = 5.0 \times 10^{-36} (\phi t)^{1.66} [e^{-6800/RT} - (1.87 \times 10^4) e^{-27,000/RT}] \quad (2)$$

where $\frac{\Delta V}{V}$ = percent volume change

ϕt = neutron fluence, $E > 0.1$ MeV

Q = activation energy in cal/mole - °K

Equation (1) was developed by using regression analysis to fit an equation of the form:

$$\frac{\Delta V}{V} = A(\phi t)^n e^{-Q/RT} \quad (3)$$

to 37 data points which were selected from available bulk density measurements on 304 SS components and specimens taken from the EBR-II reactor. This data is given in Table 12.8. In the selection of data, only points corresponding to locations within 10 inches of the midplane were used. The analysis resulted in the following equation

$$\frac{\Delta V}{V} = 5.0 \times 10^{-36} (\phi t)^{1.66} e^{-\frac{6800}{RT}} \quad (4)$$

The second temperature-dependent term in Equation (1), $(\alpha e^{-Q_2/RT})$, was empirically fit to force the swelling to peak at 625 °C and to go to zero at 750 °C.

Figure 12.3 shows the fluence dependency given by equation (2) at 400 °C and 600 °C. Widening of the confidence bands is due to the lack of data beyond about 1×10^{22} n/cm². No confidence limits may be placed on this equation above about 600 °C.

A review of PNL transmission electron microscopy data for 304 SS irradiated in the EBR-II has indicated that the nucleation rate of cavities may increase with accumulated exposure. A summary of the essential features of the void distributions

TABLE 12.8. Swelling Data for 304 Stainless Steel

Component	Position Vert.	Fluence, $^{-22}$ $n/cm^2 \times 10$ ($E > 0.1$ MeV)	Irrad. Temp, °C	$\Delta\rho/\rho$, %	Source
Control Rod	- 4.7	2.92	370	0.51	PNL
Thimble	+ 1.7	5.50	400	1.15	
(5C3)	+ 6.7	2.26	430	0.88	
	+10.0	1.58	440	0.31	
EBR-II Fuel	- 7	2.00	370	0.37	PNL
Subassembly	- 5	2.66	370	0.54	
Thimble (X-G05)	- 2.5	3.16	380	0.80	
(4C2)	0	3.41	390	1.27	
	+ 2.5	3.16	400	1.35	
	+ 5	2.66	420	0.92	
	+ 7	2.00	430	0.47	
EBR-II	- 7.0	4.34	370	0.93	PNL
Safety Rod	- 5	5.34	370	1.55	
Thimble	- 2.5	6.25	380	2.44	
3D1	0	6.75	390	5.09	
	+ 2.5	6.25	400	2.41	
	+ 5	5.33	420	1.55	
	+ 7	4.34	430	0.87	
Experiment	- 5	1.92	400	0.13	
Capsule X-011	- 2	2.25	420	0.27	
2F1	+ 2	2.25	440	0.33	
	+ 6	1.10	450	0.34	
	+ 9	0.66	450	0.13	
EBR-II Control	- 9	2.00	370	0.53	ANL
Rod Thimble	0	3.76	410	1.26	
(5C3)	+ 6	3.42	450	1.67	
	+ 9	2.58	470	1.15	
EBR-II Safety	- 4.0	5.91	385	0.63	ANL
Rod Thimble	- 0.8	6.75	395	2.63	
(3D1)	+ 2.6	6.45	405	3.70	
	+ 5.8	4.89	420	1.85	
Fuel	- 8.4	2.15	370	0.47	ANL
Subassembly	- 5.4	2.96	380	0.44	
Thimble X-G05	0	3.54	430	0.95	
(4C2)	+ 5.6	2.69	460	1.11

Neg 692163-3

FIGURE 12.3. *Swelling in Solution-Treated AISI Type 304 Stainless Steels*

observed to date is given in Table 12.9. The void number density has been plotted as a function of fluence, using logarithmic coordinates (Figure 12.4). A regression analysis of the data indicates that the void density is related to the fluence by a power law in fluence with the exponent being 1.2 ± 0.3 . If the exponent is greater than 1.0, an increase in nucleation-rate with accumulated exposure is indicated. It is possible that a continuously increasing helium concentration, due to transmutation reactions, could contribute to an increasing nucleation-rate by stabilizing cavity nuclei. No consistent trends relating void number density to irradiation temperature were found. A correlation between void volume based upon microscopy and that based upon immersion density is indicated in Figure 12.5. In this figure, microscopy volume changes from

TABLE 12.9. *Transmission Electron Microscopy of Irradiated Type 304 Stainless Steel*

Component	Fast Fluence E > 0.1 MeV, n/cm ² -sec × 10 ⁻²²	Fast Flux E > 0.1 MeV, n/cm ² -sec × 10 ⁻¹⁵	Irrad. Temp. °C	Cavity Density cm ⁻³	Lineal Average Cavity Diameter	Swelling
EBR-II	0.5	0.1	460	0.3	245	0.3
Control	0.9	0.3	370	0.7	125	0.08
Rod Thimble	1.5	0.5	370	1.9	145	0.3
	2.2	0.7	430	1.7	210	0.95
	2.4	0.8	420	2.0	200	1.0
	3.3	1.1	380	6.5	150	1.2
EBR-II X-G05	3.4	1.5	390	2.3	210	1.4
EBR-II Safety Rod Thimble	5.9	1.6	400	8.2	170	2.7

Neg 692070-1

FIGURE 12.4. *Cavity Density as a Function of Fluence for 304 Stainless Steel.*

Neg 692234-1

FIGURE 12.5. Swelling as a Function of Fluence for Various EBR-II Components.

Table 12.9 are superimposed on immersion density values. Except for the point at 5×10^{21} n/cm², all microscopy data are in agreement with the bulk density values.

Transmission microscopy is continuing on irradiated 316 SS. In the previous quarterly report (see reference (1), p. 12.44), it was stated that solution-treated type 316 SS, irradiated to 0.8×10^{22} n/cm² ($E > 0.1$ MeV) at 900 °F, contained small polyhedral and rod-shaped $M_{23}C_6$ precipitates. It was also reported that the carbide morphology was similar to that reported in the literature for unirradiated stainless steels annealed at higher temperatures. In order to determine if a similar carbide distribution could be created in our control material, we annealed unirradiated 316 SS control samples for 3050 hours at 1200 °F and at 1400 °F and then examined them. These specimens contained only a small concentration ($\sim 10^{10-11}$ /cm³) of polyhedral precipitates within the matrix. There were essentially no precipitates

on the grain boundaries. The low concentration of carbides (as compared to the irradiated specimen and to results reported in the literature for non-irradiated specimens after similar anneals) indicates that the unirradiated thermal control sample may have been decarburized during the anneal. Chemical analyses for carbon content in irradiated and unirradiated specimens with various thermal treatments were obtained to explore this possibility. The results of these analyses are given in Table 12.10. These data do not indicate any significant amount of decarburization resulting from the long time anneals. Light microscopy specimens are presently being taken from the annealed specimens in an attempt to resolve the apparent discrepancy between the transmission electron microscopy and carbon analysis results.

Examination of the EBR-II control rod and safety rod thimbles (304 SS) indicates that the carbide morphology varies along the length of the thimble. Furthermore, qualitative examination of the micrographs suggests that there was more carbide from specimens near the core centerline. Accordingly, chemical analyses of carbon content in sections from the two thimbles were obtained to explore the possibility of irradiation induced carbon transfer. The results of these analyses are given in Table 12.11.

The reproducibility of the carbon content (based on eleven National Bureau of Standard samples) was better than 10%. The results were quite different than anticipated. Both samples irradiated at the peak fluence region showed lower carbon concentrations than at either the inlet or outlet regions. While there appears to have been extensive carbon transfer from the cold to the hot end of the control rod thimble, there were no large changes in the safety rod thimble. At the present, we have no explanation for these results.

TABLE 12.10. Carbon Analyses of 316 Austenitic Stainless Steel Subjected to Various Irradiation and Thermal Conditions

<u>Treatment</u>	<u>Carbon Content</u>
1 hr at 1950 °F	550 (ppm)
1 hr at 1950 °F +3050 hr at 900 °F	410 (ppm)
1 hr at 1950 °F +3050 hr at 1400 °F	565 (ppm)
1 hr at 1950 °F +1 × 10 ²¹ n/cm ² (E >0.1 MeV) at 700 °F (3050 hr in Reactor)	490 (ppm)
1 hr at 1950 °F +8 × 10 ²¹ n/cm ² (E >0.1 MeV) at 900 °F (3050 hr in Reactor)	355 (ppm)

TABLE 12.11. Carbon Analyses of EBR-II Components

<u>Component</u>	<u>Location</u>	<u>Fluence,</u> <u>n/cm² (E >0.1 MeV)</u> <u>(× 10⁻²²)</u>	<u>Temperature,</u> <u>°F</u>	<u>Carbon,</u> <u>ppm</u>
Control Rod Thimble	Coolant Inlet	0.9	700	185
	Peak Fluence	3.5	745	140
	Coolant Outlet	0.2	800	445
Safety Rod Thimble	Coolant Inlet	0.6	700	345
	Peak Fluence	5.9	755	280
	Coolant Outlet	1.3	850	305

EBR-II Irradiations - P. K. McDaniel

The objective of this program is to provide coordination and direction for the timely accomplishment of fast reactor irradiations of structural materials. Irradiation experiments on structural materials are in various stages of development. Some are in design; some are under construction; and others are under irradiation in the EBR-II. The status of these experiments are described.

A number of subassemblies, X-009, X-101, X-014, X-018, X-021 (including X-021A), X-022, and X-030, containing BNL structural material experiments have been discharged from EBR-II. The suffix "A" designates pin interchange. A summary of these irradiation experiments is given in Table 12.12. All flux monitors from these experiments, with the exception of X-010 and X-018, have been counted. Mechanical properties tests for specimens irradiated in X-009 and X-014 have been completed. Uniaxial and biaxial tests on specimens irradiated in X-022 are in progress.

Experiments presently in EBR-II include subassemblies X-019, X-020, X-027, X-067, and full subassemblies X-021, (now X-021B), X-041, and X-057. The details of these subassemblies are given in Table 12.13. Subassembly X-067 consists of four pins from Battelle-Northwest, two from Argonne National Laboratory, and one from General Electric.

The four Battelle-Northwest pins contain specimens for an investigation of the effect of fast reactor irradiation on 304 and 316 SS weldments, in support of the FFTF pressure vessel and fuel duct studies. The goal exposure is 1×10^{22} n/cm² (E > 0.1 MeV).

Acceptance Testing and Clad Characterization

M. M. Paxton

The objective of this program is to insure that acceptable cladding material is obtained and used throughout the FFTF fuel development and irradiation testing program. Under this program, lot-to-lot variations in selected mechanical properties of AISI 304 and 316 SS tubing are being determined and evaluated.

TABLE 12.12. Summary of Experiment Discharged From EBR-II

Subassembly Designation	Reactor Position	Date Charged	Date Discharged	Type of Specimen	Material	Accumulated ^(d) Exposure, n/cm ²
X-009 ^(a)	4A2	3-24-66	11-15-66	18 Tensile 10 Uniaxial Creep	304 SS	2.0×10^{22}
X-010 ^(a)	7F3	3-24-66	4-24-69	18 Tensile 10 Uniaxial Creep	304 SS	6.6×10^{22}
X-014 ^(a)	2D1	7-17-66	12-31-66	50 Tensile	304 SS	1.7×10^{22}
X-018	2B1	12-6-66	2-25-69	198 Tensile or Uniaxial Creep	304 SS, 316 SS 348 SS, 321 SS	4.4×10^{22}
X-021 ^(b)	2D1	2-27-67	9-16-68	127 Tensile or Uniaxial Creep	304 SS, 316 SS ^(c) Ni Base Alloys	3.2×10^{22}
X-021A ^(b)	2D1	9-27-68	11-16-68	86 Density 16 Biaxial Creep 14 Tensile 10 Uniaxial Creep	316 SS ^(c) Ni Base Alloys	5.0×10^{21}
X-022	7C4	2-27-67	6-15-68	595 Tensile or Uniaxial Creep 65 Biaxial Creep	304 SS, 316 SS, 348 SS, 321 SS	1.1×10^{22}
X-030 ^(a)	6E1	10-10-67	3-6-68	5 Tensile 3 Uniaxial Creep	304 SS	5.2×10^{21}

(a) Mark A pins irradiated on space available basis, all other pins are B-7 type.

(b) Single pin interchange.

(c) Variety of other alloys also included.

(d) $E > 0.1$ MeV

TABLE 12.13. Summary of Experiment in EBR-II

Subassembly Designation	Reactor Position	Date Charged	Estimated Discharge Date	Type of Specimens	Material Contained	Accumulated ^(a) Exposure, n/cm ²	Goal Exposure, n/cm ²
X-019	6D2	1-13-67	July 69	64 Tensile 24 Uniaxial Creep	304 SS 316 SS	2.5×10^{22}	1.9×10^{22}
X-020	6B5	1-13-67	July 69	28 Tensile 8 Uniaxial Creep	304 SS 316 SS	2.7×10^{22}	1.9×10^{22}
X-021 ^(b)	2D1	2-27-67	Aug. 70	501 Tensile or Uniaxial Creep 65 Biaxial Creep	304 SS, 316 SS 348 SS, 321 SS Ni Base Alloys ^(c)	4.1×10^{22}	8.8×10^{22}
X-027	4B3	11-21-67	Aug. 70	5 Tensile 3 Uniaxial Creep	304 SS	2.7×10^{22}	5×10^{22}
X-041	7C4	7-24-68	May 70	713 Tensile or Uniaxial Creep 74 Biaxial Creep	304 SS, 316 SS 321 SS, 348 SS	1.0×10^{22}	3×10^{22}
X-057	2B1	2-24-69	March 71	577 Tensile or Uniaxial Creep 164 Biaxial Creep 58 Density	304 SS, 316 SS, ^(c) 321 SS, 348 SS, Ni Base Alloys	4.8×10^{21}	6.0×10^{22}
X-067	4FZ	1-5-69	Sept. 69	204 Tensile or Uniaxial Creep 20 Fatigue	304 SS, 316 SS, Weldment	None	1×10^{22}

(a) Accumulated to end of run 33, ($E > 0.1$ MeV)

(b) Original space available with two single pin interchanges.

(c) Variety of other alloys.

A check chemical analysis has been completed on five tube lengths from G-lot tubing, (Table 12.14). Except for phosphorous, the check analysis agrees reasonably well with the analysis supplied by the vendor. The values supplied by the vendor are lower by a factor of two than the values received on the check analysis.

Metallography on five G-lot tube lengths has been completed. Figures 12.6-12.9 are typical transverse and longitudinal photomicrographs from 100X to 1000X. The grain size as determined from the five samples varies between ASTM size 7 to 8.

Burst tests on tubing lots E, F, G, and H have been completed. The results are tabulated in Table 12.15 and shown graphically in Figures 12.10-19 in terms of internal gas pressure required to burst the tube and the reciprocal of absolute temperature. The lines drawn through the data are for a least squares fit to the equation:

$$y = B_0 + B_1(1/x) + B_2(1/x)^2$$

where y = pressure, psi
 x = temperature ($^{\circ}$ K)

B_0 , B_1 , and B_2 are the regression coefficients.

Burst pressure may be calculated at any temperature in the range 1000 to 1800 $^{\circ}$ F using the above equation and the appropriate values for B_0 , B_1 , and B_2 given in Table 12.16.

Burst results depend on the rate of loading, and therefore are not unique unless the rate of loading is specified and controlled. The rate of loading effect is clearly evident in tests performed on E lot reported previously,⁽¹⁶⁾ in which bursts occurred at 1400 psi in 10-14 min. and at 2500 psi in 1-3 min. In order to eliminate loading time as a variable, a series of short term, 0.01- to 1-hr, stress-rupture tests were performed on G and H lot tubing. The results are tabulated in Table 12.17 and shown graphically in Figures 12.14 and 15. If the results

TABLE 12.14. Chemical Analysis "G" Lot Tubing 316 Stainless Steel

Vendor Analysis:

<u>Heat No.</u>	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Ni</u>	<u>Cr</u>	<u>Mo</u>	<u>B</u>	<u>N₂</u>	<u>Cu</u>	<u>Fe</u>
	0.05	1.60	0.010	0.012	0.49	13.32	17.63	2.41	0.0007	0.047	0.11	Bal
	0.05	1.56	0.012	0.016	0.56	13.29	17.70	2.42	0.0013	0.052	0.10	Bal
	0.05	1.56	0.011	0.015	0.53	13.29	17.63	2.41	0.0009	0.054	0.11	Bal
	0.05	1.53	0.010	0.015	0.58	13.34	17.63	2.45	0.0012	0.050	0.15	Bal
	0.06	1.58	0.010	0.012	0.56	13.31	17.33	2.50	0.0010	0.049	0.11	Bal

Check Analysis by Lukens Steel

<u>Identification</u>	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Ni</u>	<u>Cr</u>	<u>Mo</u>	<u>Cu</u>	<u>B</u>	<u>N</u>
G-081	0.049	1.55	0.026	0.015	0.52	13.42	17.62	2.37	0.13	0.0008	0.043
G-163	0.047	1.55	0.025	0.014	0.53	13.42	17.65	2.37	0.13	0.0010	0.044
G-238	0.048	1.55	0.026	0.013	0.52	13.42	17.60	2.37	0.13	0.0008	0.035
G-257	0.048	1.55	0.026	0.014	0.52	13.42	17.60	2.39	0.13	0.0010	0.042
G-320	0.050	1.55	0.024	0.014	0.52	13.42	17.55	2.40	0.13	0.0010	0.040

12.27

BNWL-1144

TABLE 12.15. *Burst Data*

Temp.	Burst-Pressure, psi	Hoop Stress, psi	Load Time	$\Delta P/P, \%$
G Lot Tubing Mill Annealed 316 Stainless Steel				
1000 °F	8,110	55,230	17 min.	16.9
	8,300	56,520	17 min.	16.1
	8,150	55,500	16 min.	16.5
1200 °F	6,960	47,400	6 min.	18.1
	7,220	49,170	6 min.	17.3
	7,210	49,100	6 min.	18.7
	6,950	47,330	5.75 min.	22.5
1400 °F	5,610	38,200	1 min.	20.1
	5,620	38,270	1 min.	19.7
	5,640	38,410	1 min.	22.5
	5,500	37,460	0.75	21.3
1600 °F	3,950	26,900	0.67	22.5
	4,050	27,580	0.67	18.7
	4,160	28,330	0.60	21.7
	4,060	27,650	0.50	21.3
1800 °F	2,780	18,930	0.30	24.1
	2,740	18,660	0.30	27.3
	2,980	20,290	0.60	8.1
	2,580	17,570	0.33	20.9
RT	10,950	74,570	1.0-1.25 min.	21.0
	11,100	75,590	1.0-1.25 min.	21.0
	10,700	72,870	1.0-1.25 min.	21.0
	10,750	74,230	1.0-1.25 min.	21.0
H Lot Tubing Mill Annealed 316 Stainless Steel				
1000 °F	8,380	57,070	10.75	18.9
	8,260	56,250	9.0	16.9
1200 °F	6,940	47,260	1.0	15.3
	7,120	48,490	0.75	22.1
	7,140	48,620	0.80	19.3
1400° F	5,450	37,110	0.67	20.9
	5,440	37,050	0.67	17.7
	5,340	36,980	0.50	22.1
1600 °F	3,880	26,420	0.50	14.5
	3,820	26,010	0.50	23.7
	3,950	26,900	0.70	23.4
1800 °F	2,580	17,570	0.50	20.7
	2,640	17,980	0.50	14.5
	2,630	17,910	0.40	18.9
RT	10,750	73,210	1.0-1.25 min.	20.0
	9,750	66,400	1.0-1.25 min.	16.0
	10,650	72,530	1.0-1.25 min.	19.0
	11,000	74,910	1.0-1.25 min.	20.0
	10,900	74,230	1.0-1.25 min.	19.0

TABLE 12.15. (contd)

<u>Temp.</u>	<u>Burst-Pressure, psi</u>	<u>Hoop Stress, psi</u>	<u>Load Time</u>	<u>$\Delta P/P, \%$</u>
E Lot Tubing Mill Annealed 304 Stainless Steel				
1000 °F	7,390	50,325	10 min.	18.7
	7,430	50,600	8 min.	16.5
	7,410	50,460	12 min.	18.9
1200 °F	6,260	42,630	<6 min.	17.3
	6,280	42,770	<6 min.	18.1
	6,230	42,430	<6 min.	16.9
1400 °F	5,000	34,050	<6 min.	16.9
	5,060	34,460	<6 min.	16.1
	5,020	34,190	<6 min.	15.3
1600 °F	3,530	24,040	<6 min.	17.3
	3,550	24,180	<6 min.	14.5
	3,510	23,900	<6 min.	18.5
1800 °F	2,600	17,710	<6 min.	13.3
	2,460	16,750	<6 min.	21.3
	2,340	15,940	<6 min.	20.7
RT	10,700	72,870	1.0-1.25 min.	20.0
	10,700	72,870	1.0-1.25 min.	22.0
	10,700	72,870	1.0-1.25 min.	19.0
	10,800	73,550	1.0-1.25 min.	22.0
	10,800	73,550	1.0-1.25 min.	22.0
F Lot Tubing Mill Annealed 304 Stainless Steel				
1000 °F	7,075	48,180	8 min.	16.1
	7,070	48,150	6 min.	17.3
	7,020	47,810	7 min.	17.7
1200 °F	5,790	39,430	<6 min.	20.1
	5,830	39,700	<6 min.	18.1
	5,820	29,630	<6 min.	18.1
1400 °F	4,400	29,960	<6 min.	20.1
	4,450	30,300	<6 min.	24.1
	4,460	30,370	<6 min.	20.9
1600 °F	3,090	21,040	<6 min.	20.1
	3,200	21,790	<6 min.	21.7
	3,170	21,590	<6 min.	20.1
1800 °F	2,100	14,300	6 min.	26.7
	2,000	13,620	6 min.	22.9
	2,040	13,890	6 min.	22.1
RT	9,950	67,760	1.0-1.25 min.	21.0
	9,950	67,760	1.0-1.25 min.	21.0
	9,900	67,420	1.0-1.25 min.	20.0
	9,950	67,760	1.0-1.25 min.	21.0
	9,950	67,760	1.0-1.25 min.	24.0

TABLE 12.16. Regression Coefficients for Burst Data

<u>Tubing Lot</u>	<u>B₀</u>	<u>B₁</u>	<u>B₂</u>
E	-1.9714×10^4	3.71978×10^7	-1.26778×10^{10}
F	-1.68999×10^4	3.16296×10^7	-9.88663×10^{10}
G	-2.94949×10^4	5.70268×10^7	-2.14467×10^{10}
H	-2.24968×10^4	4.3455×10^7	-1.50741×10^{10}

of the burst tests are treated as very short-term stress-rupture tests, Figures 12.16 and 17, the two tests give comparable results, except at 1800 °F. In treating a burst test as a stress-rupture test, the stress at rupture is taken as the stress to cause rupture and the time to rupture is the load-time for the burst test.

Stress-rupture tests on tubing lots E, F, G, and H have been completed at 1200 °F for rupture times less than 1400 hours. The tests completed to date are tabulated in Table 12.18 and shown graphically in Figures 12.19-23. The ductilities, shown as $\Delta P/P$ were taken well away from the failed region since most specimens failed in an explosive manner.

The difference between the creep-rupture properties of lots E and F is being investigated. Lot E was found to be considerably stronger than lot F. The main metallurgical differences between these two is a larger grain size in E than in F and a lower interstitial concentration in F than in E. Research literature⁽¹⁷⁻¹⁹⁾ has shown that creep-rates are related to total interstitial concentration. In order to more fully understand how these differences affect rupture-life, a series of biaxial creep tests was conducted. The biaxial creep curves were determined by periodically interrupting the test and measuring diametrical

TABLE 12.17. *Short Term Stress Rupture Data*

<u>Temp.</u>	<u>Gas Pressure, psi</u>	<u>Hoop Stress, psi</u>	<u>Rupture Time, Hours</u>	<u>$\Delta P/P, \%$</u>
G Lot Mill Annealed 316 Stainless Steel				
1800 °F	1,620	11,050	0.026	17.3
	1,320	8,990	0.189	20.1
	1,170	7,970	0.251	18.7
	1,030	7,010	0.449	20.5
	940	6,400	0.756	24.1
1600 °F	3,810	25,950	0.008	20.1
	2,960	20,160	0.045	28.9
	2,640	17,980	0.100	25.3
	2,350	16,000	0.178	22.9
	2,130	14,500	0.394	20.5
	1,910	13,010	0.852	24.1
1400 °F	5,210	35,480	0.018	22.8
	5,000	34,050	0.028	24.9
	4,670	31,800	0.061	26.1
	4,130	28,130	0.175	27.7
	3,860	26,290	0.371	31.7
	3,660	24,920	0.636	27.3
1200 °F	6,800	46,310	0.034	21.6
	6,600	44,950	0.072	20.8
	6,320	43,040	0.450	21.3
	6,170	42,020	0.562	19.3
	6,020	41,000	0.818	23.3
H Lot Mill Annealed 316 Stainless Steel				
1800 °F	1,610	10,960	0.044	20.1
	1,320	8,990	0.117	18.1
	1,170	7,970	0.233	20.9
	1,030	7,010	0.716	25.7
	940	6,400	1.00	22.1
1600 °F	2,930	19,950	0.048	22.1
	2,640	17,980	0.110	23.7
	2,350	16,000	0.195	23.7
	2,130	14,500	0.446	23.3
	1,910	13,010	1.00	18.9
1400 °F	4,700	32,010	0.044	21.3
	4,130	28,130	0.194	22.9
	3,860	26,290	0.346	22.5
	3,660	24,920	0.789	24.1
	3,550	24,180	1.00	23.3
1200 °F	6,800	46,310	0.056	21.7
	6,600	44,950	0.133	22.0
	6,320	43,040	0.340	20.5
	6,170	42,020	0.413	18.1
	6,020	41,000	0.672	17.3

TABLE 12.18. Long Term Stress Rupture Data

<u>Temp.</u>	<u>Gas Pressure, psi</u>	<u>Hoop Stress, psi</u>	<u>Rupture Time, hr</u>	<u>$\Delta P/P, \%$</u>
E Lot Mill Annealed 304 Stainless Steel				
1200 °F	4,810	32,760	9.8	10.1
	4,260	29,010	44.5	12.8
	3,700	25,200	125.0	6.5
	3,310	22,540	268.2	4.4
	3,820	26,010	80.0	---
	3,010	20,500	816.0	6.0
	3,010	20,500	853.0	7.0
F Lot Mill Annealed 304 Stainless Steel				
1200 °F	4,260	29,010	5.8	10.1
	3,790	25,800	17.8	10.9
	2,950	20,090	281.9	13.3
	2,580	17,570	1388.9	7.7
	3,670	24,990	41.8	---
	3,310	22,540	112.5	---
G Lot Mill Annealed 316 Stainless Steel				
1200 °F	5,150	35,070	8.3	15.7
	4,730	32,210	20.9	22.5
	4,400	29,960	42.4	20.9
	4,110	27,990	84.2	20.5
	4,130	28,130	94.9	18.9
	3,840	26,150	172.1	14.5
	3,670	25,000	327.8	11.3
	3,250	22,130	818.4	9.3
H Lot Mill Annealed 316 Stainless Steel				
1200 °F	5,580	38,000	1.5	26.1
	5,150	35,070	3.6	13.0
	4,730	32,210	10.5	13.5
	4,110	27,990	60.5	11.0
	3,670	24,990	222.9	6.9

FIGURE 12.6. *Tubing Lot G Mill Annealed 316 Stainless Transverse Tube Length G-099. Etch 25% Alcohol 75% HCl (elec.) (Neg 46972A; 469-74; 46972B; 46972C)*

FIGURE 12.7. *Tubing Lot G Mill Annealed 316 Stainless Longitudinal Tube Length G-099. Etch 25% Alcohol 75% HCl (elec.) (Neg 46977A; 469-79; 46977B; 46977C)*

FIGURE 12.8. *Tubing Lot G Mill Annealed 316 Stainless Transverse Tube Length G-177. Etch 25% Alcohol 75% HCl (elec.). (Neg 46976A; 469-78; 46976B; 46976C)*

FIGURE 12.9. *Tubing Lot G Mill Annealed 316 Stainless Longitudinal Tube Length G-177. Etch 25% Alcohol 75% HCl (elec.). (Neg 46973A; 469-75; 46973B; 46973C)*

Neg 0692361-16

FIGURE 12.10. E Lot Burst Data 1000-1800 °F Mill Annealed 304 Stainless Steel

Neg 0692361-12

FIGURE 12.11. F Lot Burst Data 1000-1800 °F Mill Annealed 304 Stainless Steel

Neg 0692361-15

FIGURE 12.12. G Lot Burst Data 1000-1800 $^{\circ}\text{F}$ Mill Annealed 316 Stainless Steel

Neg 0692361-13

FIGURE 12.13. H Lot Burst Data 1000-1800 $^{\circ}\text{F}$ Mill Annealed 316 Stainless Steel

Neg 0692361-14

FIGURE 12.14. Short Term Stress Rupture G Lot Tubing -
Mill Annealed 316 Stainless Steel

Neg 0692361-9

FIGURE 12.15. Short Term Stress Rupture H Lot Tubing -
Mill Annealed 316 Stainless Steel

Neg 0692361-6

FIGURE 12.16. Comparison of Burst and Short Term Stress Rupture Data for G Lot Tubing - Mill Annealed 316 Stainless Steel

Neg 0692361-10

FIGURE 12.17. Comparison of Burst and Short Term Stress Rupture Data for H Lot Tubing - Mill Annealed 316 Stainless Steel

Neg 0692361-2

FIGURE 12.18. Stress Rupture 1200 °F E Lot Tubing -
Mill Annealed 304 Stainless Steel

Neg 0692361-11

FIGURE 12.19. Stress Rupture 1200 °F F Lot Tubing -
Mill Annealed 304 Stainless Steel

Neg 0692361-3

FIGURE 12.20. $\Delta P/P$ % Versus Rupture Time for E and F Lot Tubing 1200 °F - Mill Annealed 304 Stainless Steel

Neg 0692361-7

FIGURE 12.21. Stress Rupture 1200 °F G Lot Tubing - Mill Annealed 316 Stainless Steel

Neg 0692361-8

FIGURE 12.22. $\Delta P/P$ % Versus Rupture Time for G Lot
Tubing 1200 °F - Mill Annealed 316
Stainless Steel

Neg 0692361-5

FIGURE 12.23. Biaxial Creep - Specimen E-1 1200 °F σ_{max}
= 26,850 psi, Tubing Lot E, Mill
Annealed 304 Stainless Steel.

strain every 1/4 inch along the specimen. The minimum creep-rates for these tests are given in Table 12.19 and the creep curves are presented in Figures 12.23-26. The effects of grain size and composition cannot be unambiguously accounted for in these tests; however, if one assumes that the relationship between interstitial content and creep-rate given by Stone⁽¹⁷⁾ holds and that rupture life is proportional to minimum creep-rate, then the rupture life of the two lot F tests would increase from 24 to 30 hr and 84 to 105 hr at an interstitial concentration equivalent to E lot.

TABLE 12.19. *Biaxial Creep Rates at 1200 °F
and $\sigma_{max} = 26,850$ psi*

<u>Specimen</u>	<u>Measured hr⁻¹</u>	<u>Corrected hr⁻¹</u>
E-1	2.78×10^{-4}	---
E-2	2.45×10^{-4}	---
F-1	2.40×10^{-3}	6.9×10^{-4}
F-2	2.79×10^{-3}	8.0×10^{-4}

The remaining differences in rupture-life should be attributed to grain size differences in the absence of side effects such as heat treatment or fabrication variables. Garofalo's equation⁽²⁰⁾ can be used to estimate the grain size that gives minimum creep rates for 304 stainless steel. This analysis indicates that a grain size of ASTM 7 is optimum for creep resistance.

The biaxial creep curves on lot F specimens, Figures 12.25 and 26, are shown as two curves. In both tests, the upper curve, dashed line, represents the creep of a localized bulge that appeared near the top of each specimen. Specimen F-1, Figure 12.26, failed in the bulged area, whereas specimen F-2, Figure 12.25, did not fail in the bulge. Close examination of the diametrical measurement throughout the test shows

Neg 0692361-4

FIGURE 12.24. *Biaxial Creep - Specimen E-2 1200 °F σ_{max} = 26,850 psi, Tubing Lot E, Mill Annealed 304 Stainless Steel*

Neg 0692361-17

FIGURE 12.25. *Biaxial Creep - Specimen F-1 1200 °F σ_{max} = 26,850 psi, Tubing Lot F, Mill Annealed 304 Stainless Steel*

Neg 0692361-1

FIGURE 12.26. *Biaxial Creep - Specimen F-2 1200 °F σ_{max} = 26,850 psi, Tubing Lot F, Mill Annealed 304 Stainless Steel*

the failed region of F-2 to be slightly constricted by approximately 0.0005 in. Such a restricted area did not appear on the F-1 specimen. In order to explain this anomalous behavior, the two F specimens are being subjected to optical metallography.

Test E-2 was terminated before failure. Inspection of the specimen after 122 hr revealed a crack (Figure 12.27) which would have resulted in failure had the test been continued.

Neg 469635

FIGURE 12.27. Macrograph of Crack in Biaxial Creep - Specimen E-2 1200 °F σ_{max} = 26,850 for 122 hr

Uniaxial Creep-Rupture Studies - A. J. Lovell

The objectives of this program are to evaluate the effects of fast neutron irradiation and environment on the pre- and post-irradiation uniaxial creep-rupture properties of candidate vessel and core structural materials for LMFBR and to establish the usable materials limits to assure their safe and reliable performance.

Tests are continuing on specimens from the program heat of AISI 304 stainless steel in the solution-treated, unirradiated condition and after irradiation in the static sodium environment of the EBR-II MK-B-X022 subassembly. The 1000 and 1100 °F test results on the solution-treated controls show the same poor creep-rupture properties observed earlier in the tests at 1200 °F. That is, the creep rates are high and the rupture lives are short compared to standard published values for this alloy. This non-typical data points out the necessity, in an irradiation effects study, to compare postirradiation test data with that from controls.

The results of 1000 and 1100 °F creep-rupture tests on this material after irradiation are tabulated in Table 12.20. Stress versus rupture-life plots for control and irradiated specimens tested at 1000 and 1100 °F are shown in Figure 12.28. The reduction in rupture-life due to irradiation is substantial. The comparison of the minimum creep-rates for the unirradiated solution-treated controls and the irradiated specimens are shown in Figure 12.29. There is a reduced creep-rate at 1100 °F when the stress level is low for specimens irradiated at the highest fluence. A comparison of the creep curves of control and irradiated specimens produced from tests at 1100 °F and 30,000 and 35,000 psi are shown in Figures 12.30 and 12.31 respectively. These curves show that irradiation has affected both rupture-life and the first- and second-stage creep rates. The lowered creep-rates in stage one and stage two indicate

Specimen No.	Calc. Total Fluence, EBR-II	Irrad. Temp, °F	Test Temp, °F	Stress, psi	Minimum Creep-Rate, in/in/hr	Rupture Time, Hr		% Elongation
						Irrad.	Control	
13B34-4	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	30,000	0.00000345 (0.00000605)	144.62	On Test	2.4 (On Test)
11F14-1	$\sim 3 \times 10^{21}$ n/cm ²	800	1000	30,000	0.00000814 (0.00000605)	1328.2	On Test	Radiomet (On Test)
13B34-3	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	35,000	0.0000322 (0.0000189)	206.1	1506.1	7.02 (11.55)
12H14-1	$\sim 1 \times 10^{21}$ n/cm ²	800	1000	35,000	0.0000435 (0.0000189)	299.4	1506.1	6.13 (11.55)
10F14-2	$\sim 3 \times 10^{21}$ n/cm ²	800	1000	35,000	0.0000336 (0.0000189)	294.4	1506.1	3.56 (11.55)
15H24-1	$\sim 1 \times 10^{21}$ n/cm ²	800	1000	35,000	0.0000582 (0.0000189)	378.7	1506.1	8.89 (11.55)
12H14-2	$\sim 1 \times 10^{21}$ n/cm ²	800	1000	40,000	0.000102 (0.000113)	297.3	336.7	18.13 (19.17)
10F14-3	$\sim 3 \times 10^{21}$ n/cm ²	800	1000	40,000	0.000147 (0.000113)	67.8	336.7	Radiomet (18.17)
13B34-2	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	45,000	0.00037 (Av. of 4 0.000359)	15.85	Av. of 4 117.8	Radiomet (Av. of 4 19.53)
10F14-1	$\sim 3 \times 10^{21}$ n/cm ²	800	1000	45,000	0.000456 (0.000258)	35.3	117.8	7.11 (12.18)
9H14-1	$\sim 1 \times 10^{21}$ n/cm ²	800	1000	45,000	0.000258 (0.000942)	80.8	117.8	12.18
13B34-1	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	50,000	0.00112 (0.000942)	5.6 to 6.1	29.1	Radiomet (29.01)
15H14-1	$\sim 1 \times 10^{21}$ n/cm ²	800	1000	50,000	0.00074 (0.000942)	30.0	29.1	15.64 (29.01)
13B64-1	$\sim 1 \times 10^{22}$ n/cm ²	900	1100	25,000	-- (0.000117)	532.8	627.0	12.62 (14.93)
10F24-1	$\sim 3 \times 10^{21}$ n/cm ²	800	1100	25,000	0.0000694 (0.000117)	575.6	627.0	Radiomet (14.93)
9K14-1	$\sim 1 \times 10^{21}$ n/cm ²	800	1100	25,000	0.0000855 (0.000117)	364.5	627.0	16.44 (14.93)
13B64-3	$\sim 1 \times 10^{22}$ n/cm ²	900	1100	28,000	0.000089 (Av. of 3 0.000385)	151.5	Av. of 3 269.8	3.46 (Av. of 3 17.57)
13B54-1	$\sim 1 \times 10^{22}$ n/cm ²	900	1100	30,000	0.00019 (Av. of 2 0.000475)	71.9	Av. of 2 201.0	Radiomet (Av. of 2 19.81)
9F24-2	$\sim 3 \times 10^{21}$ n/cm ²	800	1100	30,000	0.000507 (0.000502)	90.2	201.0	11.82
9H24-2	$\sim 1 \times 10^{21}$ n/cm ²	800	1100	30,000	0.000502 (0.00306)	95.2	201.0	Radiomet
13B54-2	$\sim 1 \times 10^{22}$ n/cm ²	900	1100	35,000	0.001245 (0.00306)	17.7	49.0	Radiomet (25.51)
9H24-1	$\sim 1 \times 10^{21}$ n/cm ²	800	1100	35,000	0.00254 (0.00306)	17.5	49.0	14.04 (25.51)
9F24-1	$\sim 3 \times 10^{21}$ n/cm ²	800	1100	35,000	0.00312 (0.00306)	13.7 to 18.3	49.0	12.8 (25.51)
13B64-2	$\sim 1 \times 10^{21}$ n/cm ²	900	1100	40,000	0.0066 (Av. of 2 0.00734)	5.7	Av. of 2 Approx 15.0	Radiomet (Av. of 2 25.73)
11F24-1	$\sim 3 \times 10^{21}$ n/cm ²	800	1100	40,000	0.00507 (0.0121)	8.7	Approx 15.0	9.51
9K14-2	$\sim 1 \times 10^{21}$ n/cm ²	800	1100	40,000	0.0121	4.25	Approx 15.0	19.37

(a) All tests in helium.
 (b) Solution-treated controls.

TABLE 12.20. Uniaxial Creep-Rupture Properties of Irradiated AISI Type 304 Stainless Steel Irradiated in the Solution-Treated Condition(a)

Neg 0691009-2

FIGURE 12.28. Effect of Irradiation on the Postirradiation Creep-Rupture Properties of AISI Type 304 Stainless Steel

Neg 0692296-2

FIGURE 12.29. Effects of Irradiation on the Postirradiation Creep Rate of AISI Type 304 Stainless Steel

Neg 0690705-1

FIGURE 12.30. *Effect of Irradiation on the Postirradiation Creep Properties of AISI Type 304 Stainless Steel*

Neg 0690705-2

FIGURE 12.31. *Effect of Irradiation on the Postirradiation Creep Properties of AISI Type 304 Stainless Steel*

the presence of an irradiation-induced defect that is impeding creep. The final measurements of all failed specimens are not available, but the creep elongations, as indicated by the data shown in Figures 12.30 and 12.31, are rather low.

Creep-rupture data obtained on 316 specimens irradiated in the first EBR-II Mark B-7 subassembly are tabulated in Table 12.21. The stress versus time-to-rupture curves for test temperatures of 1000, 1100, 1200, and 1400 °F are shown in Figures 12.32, 33, 34, and 35. The effect of irradiation on the minimum creep-rates are shown in Figures 12.36 and 12.37. High-fluence irradiation results in increased creep-rates for low-temperature, high stress tests and decreased rates for high-temperature, low-stress tests, particularly at 1200 °F. Creep curves from these two test conditions are shown in Figures 12.38 and 12.39.

Biaxial Stress-Rupture Studies - R. W. Barker

The objective of this program is to determine the combined effects of environment and fast reactor irradiation on the biaxial stress-to-rupture properties of candidate fast reactor cladding alloys.

Biaxial stress-rupture tests have been initiated on AISI Type 304 stainless steel irradiated to $\sim 0.8 \times 10^{22}$ n/cm² (E > 0.1 MeV) in EBR-II. The specimens being tested are composed of segments of irradiated cladding (0.208-in. diameter $\times$ 0.008-in. wall) to which unirradiated fittings have been welded. (21,22)

Four tests on the irradiated cladding have been completed. The test temperature was 1200 °F and the environment and pressurizing gas were argon. Specimens were stressed sufficiently to cause rupture in 0.1 to 1,000 hr in unirradiated material.

Specimen No.	Calc. Total Fluence, EBR-II	Irrad. Temp, °F	Test Temp, °F	Stress, psi	Minimum Creep-Rate, in/in/hr	Rupture Time, hr		% Elongation
						Irrad.	Control	
14E16-14	$\sim 3 \times 10^{21}$ n/cm ²	700	1000	63,000	0.00037 (0.000949)	27.6	60	
14E16-13	$\sim 3 \times 10^{21}$ n/cm ²	700	1000	60,000	0.000507 (0.000329)	104.6	106	
12G26-5	$\sim 2 \times 10^{21}$ n/cm ²	700	1000	60,000	0.000307 (0.000329)	98.1	106	
14E16-11	$\sim 3 \times 10^{21}$ n/cm ²	700	1000	55,000	0.00027 (0.0000915)	120.2	263	
14E16-12	$\sim 3 \times 10^{21}$ n/cm ²	700	1000	50,000	0.0000385 (0.0000293)	410.1	433	11.0 (18)
12G26-6	$\sim 2 \times 10^{21}$ n/cm ²	700	1000	50,000	0.000050 (0.0000293)	247-268	433	18.0 (18)
14E16-15	$\sim 3 \times 10^{21}$ n/cm ²	700	1000	45,000	0.000011 (0.0000115)	657.1	1032	
13E16-6	$\sim 3 \times 10^{21}$ n/cm ²	700	1100	45,000	0.0026 (0.00240)	11.4	23	
13E16-9	$\sim 3 \times 10^{21}$ n/cm ²	700	1100	36,000	0.00040 (0.0002667)	56.4	236	
13E16-10	$\sim 3 \times 10^{21}$ n/cm ²	700	1100	32,000	0.00012	164.1	~ 800	
13E16-7	$\sim 3 \times 10^{21}$ n/cm ²	700	1100	30,000	0.000065 (0.000036)	272.1	1600	6.2 (~ 20)
13E16-8	$\sim 3 \times 10^{21}$ n/cm ²	700	1100	26,000	0.000015 (0.0000052)	700.6	~ 6000	4.1 (~ 18)
12E16-2	$\sim 3 \times 10^{21}$ n/cm ²	700	1200	30,000	0.00125 (0.00239)	15.7	95	5.2 (~ 50)
15G26-10	$\sim 2 \times 10^{21}$ n/cm ²	700	1200	30,000	0.00299 (0.00239)	7.0	95	6.1 (~ 50)
12E16-1	$\sim 3 \times 10^{21}$ n/cm ²	700	1200	28,000	0.00178 (0.001049)	26.3	170	8.8 (51)
12E16-4	$\sim 3 \times 10^{21}$ n/cm ²	700	1200	25,000	0.000425 (0.000631)	55.9	375	3.5 (66)
15G26-9	$\sim 2 \times 10^{21}$ n/cm ²	700	1200	25,000	0.000415 (0.000631)	54.3	375	(66)
12E16-3	$\sim 3 \times 10^{21}$ n/cm ²	700	1200	22,000	0.000136 (0.000169)	346.7	1295	5.5 (35)
12E16-5	$\sim 3 \times 10^{21}$ n/cm ²	700	1200	18,000	-- (0.00002107)	1512.5	~ 7000	
14E26-12	$\sim 3 \times 10^{21}$ n/cm ²	700	1400	15,000	0.0036 (0.00453)	16.4	72	
14E26-9	$\sim 3 \times 10^{21}$ n/cm ²	700	1400	12,000	0.00084 (0.00115)	73.0	270	
14E26-10	$\sim 3 \times 10^{21}$ n/cm ²	700	1400	10,000	0.000255 (0.000293)	146.0	870	4.9 (59)
13E26-5	$\sim 3 \times 10^{21}$ n/cm ²	700	1400	9,000	0.00018 (0.000184)	294.0	~ 2000	
14B26-11	$\sim 3 \times 10^{21}$ n/cm ²	700	1400	8,000	On Test	On Test	~ 4000	

TABLE 12.21. Uniaxial Creep-Rupture Properties of Irradiated AISI Type 316 Stainless Steel Irradiated in the Solution Treated Condition(a)

Specimen No.	Calc. Total Fluence, EBR-II	Irrad. Temp, °F	Test Temp, °F	Stress, psi	Minimum Creep-Rate, in/in/hr	Rupture Time, Hr		% Elongation
						Irrad.	Control	
14B36-3	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	55,000	0.00102 (0.0000915)	26.7	265	17.6 (38.93)
14B36-4	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	50,000	0.000282 (0.0000293)	85.2	433	12.27 (18.2)
14B66-1	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	45,000	0.000230 (0.0000115)	107.8	1032	5.78 (15.4)
14B36-5	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	40,000	0.0000565 (0.0000095)	149.6	2795	(14)
14B46-3	$\sim 1 \times 10^{22}$ n/cm ²	900	1000	36,000	0.0000105	On Test		
14B56-3	$\sim 1 \times 10^{22}$ n/cm ²	900	1100	45,000	0.00496 (0.0024)	11.5	23	14.13 (25.1)
14B46-1	$\sim 1 \times 10^{22}$ n/cm ²	900	1100	40,000	0.00067	45.5		7.9 (~25)
14B56-1	$\sim 1 \times 10^{22}$ n/cm ²	900	1100	36,000	0.000215 (0.000267)	202.8	236	8.0 (20.9)
14B56-5	$\sim 1 \times 10^{22}$ n/cm ²	900	1100	30,000	0.0000507 (0.000036)	On Test	On Test	
14B36-2	$\sim 1 \times 10^{22}$ n/cm ²	900	1200	30,000	0.000845 (0.00239)	56.8	95	(~50)
14B56-2	$\sim 1 \times 10^{22}$ n/cm ²	900	1200	28,000	0.000037 (0.00105)	~157.0	168	(51)
14B46-2	$\sim 1 \times 10^{22}$ n/cm ²	900	1200	25,000	0.0000445 (0.000635)	262.0	375	(66)
14B66-2	$\sim 1 \times 10^{22}$ n/cm ²	900	1200	22,000	0.0000148 (0.000168)	~800.0	1295	(35)
14B46-4	$\sim 1 \times 10^{22}$ n/cm ²	900	1400	17,000	0.00889 (0.01000)	9.3	28	(72)
14B66-3	$\sim 1 \times 10^{22}$ n/cm ²	900	1400	15,000	0.00222 (0.00455)	20.1	72	7.2 (70)
14B66-4	$\sim 1 \times 10^{22}$ n/cm ²	900	1400	12,000	0.0006 (0.00115)	55.1	246	(64)
14B66-5	$\sim 1 \times 10^{22}$ n/cm ²	900	1400	10,000	0.000222 (0.000293)	146.5	1050	5.33 (59)

(a) All tests in helium

(b) solution treated controls

TABLE 12.21. (contd)

Neg 0692157-6

FIGURE 12.32. Effect of Irradiation on the Postirradiation Creep Rupture Properties of the 316 Stainless Tested at 1000 °F

Neg 0692157-5

FIGURE 12.33. Effect of Irradiation on the Postirradiation Creep Rupture Properties of 316 Stainless Tested at 1100 °F

Neg 0692157-3

FIGURE 12.34. Effect of Irradiation on the Postirradiation Creep Rupture Properties of 316 Stainless Tested at 1200 °F

Neg 0692157-7

FIGURE 12.35. *Effect of Irradiation on the Postirradiation Creep Rupture Properties of 316 Stainless Tested at 1400 °F*

Neg 0692157-4

FIGURE 12.36. Effects of Irradiation on the Postirradiation
 Creep Rate of AISI Type 316 Stainless Steel

Neg 0692296-1

FIGURE 12.37. Effects of Irradiation on the Postirradiation Creep Rate of AISI Type 316 Stainless Steel

Neg 0692757-2

FIGURE 12.38. Effects of Irradiation on the Postirradiation Creep Properties of 316 Stainless Steel Tested at 1000 °F at a Stress of 45,000 psi

12.63

Neg 0692459-2

FIGURE 12.39. Effect of Irradiation on the Postirradiation Creep Properties of 316 Stainless Steel Tested at 1200 °F at a Stress of 22,000 psi

BNWL-1144

The test results are summarized in Table 12.22. Hoop stresses were determined using the hoop stress formula for thin-walled tubes:

$$\sigma = \frac{PD}{2t}$$

where P = internal gas pressure

D = inside diameter

t = minimum wall thickness

Irradiated tests 4 and 5 failed when the gas leaked through a pin hole or fissure in the cladding. Test number 7 failed in an explosive manner, tearing the specimen into three separate pieces. The fourth test, number 11, lost pressure slowly at failure, the rupture being a very small fissure or pin hole. The small localized rupture is not characteristic of other tests at the pressures required for short rupture times. Normally, the energy of the compressed gas serves to explode the test piece in a violent manner once the fissure has developed. Rupture strain appears lower than that which might be expected at rapid creep rates. As a result of the peculiar behavior, this test is weighted very low in comparison with the other tests in analyzing the data.

The data from these initial tests, for irradiated AISI Type 304 stainless steel cladding suggests an increase in rupture strength for short rupture times and loss in rupture strength for longer rupture times (Figure 12.40). Thus, the line describing the irradiated data crosses the unirradiated control curve at about 3 to 5 hr. The data further suggests an increasing loss in rupture life with increasing rupture times.

A great deal of scatter is observed in the rupture strain shown in Figure 12.41. However, it can be observed that rupture strain for irradiated cladding will be markedly lower than the unirradiated values of the longer rupture times.

TABLE 12.22. *Biaxial Stress-Rupture Tests on AISI Type 304 Stainless Steel Irradiated to $\sim 0.8 \times 10^{22}$ n/cm² ($E > 0.1$ MeV) in EBR-II. (Test Temperature was 1200 °F. The Irradiation Temperature was 1000 $\pm$ 100 °F.)*

Unirradiated Data ^(a)			Irradiated Test No.	Irradiated Data		
Hoop Stress, psi	Rupture Time, hr	Rupture Strain, %		Hoop Stress, psi	Rupture Time, hr	Rupture Strain, %
34,000	0.1	16.5	11 ^(b)	34,000	0.06	2.4
29,000	1.0	14.5	7	29,000	1.55	14.8
19,000	100.0	11.8	5	19,000	30.4	0.96
15,800	1000.0	10.5	4	15,800	110.0	2.66

(a) Stress and strain values were taken from unirradiated test curves at rupture times shown.

(b) Test appeared to have failed prematurely.

Neg 0691998-1

FIGURE 12.40. *The Effect of Biaxial Stress on the Rupture Life of AISI Type 304 Stainless Steel Irradiated to $\sim 0.8 \times 10^{22}$ n/cm² ($E > 0.1$ MeV) at Approximately 1000 °F.*

Neg 0691998-2

FIGURE 12.41. *The Effect of Biaxial Stress on the Rupture Strain of AISI Type 304 Stainless Steel Irradiated to $\sim 0.8 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at approximately 1000°F .*

Biaxial stress-rupture tests on AISI Type 316 stainless steel irradiated in EBR-II to $\sim 0.8 \times 10^{22} \text{ n/cm}^2$ ($E > 0.18 \text{ MeV}$) have been initiated. At this time, tests for short rupture times at 1200°F have been completed. The data obtained from these tests is given in tabular form in Table 12.23.

TABLE 12.23. *Biaxial Stress-Rupture Tests on AISI Type 316 Stainless Steel Irradiated to $\sim 0.8 \times 10^{22}$ n/cm² ($E > 0.1$ MeV) in EBR-II. (Test Temperature was 1200 °F. The Irradiation Temperature was 815 $\pm$ 500 °F.)*

Unirradiated Data ^(a)			Irradiated Test No.	Hoop Stress, psi	Rupture Time, hr	Rupture Strain, %
Hoop Stress, psi	Rupture Time, hr	Rupture Strain, %				
42,000	0.1	15				
36,000	1.0	10	12	56,000	2.1	1.9
30,000	10.0	7	13	30,000	32.0	2.4
24,500	100.0	5				

(a) Stress and strain values were taken from unirradiated test curves at rupture times shown.

FTR Vessel Weldment Studies - A. L. Ward

The objective of this program is to provide mechanical property and metallurgical stability data on LMFBR vessel weldments in direct support of FFTF in order to define adequate lifetime performance under anticipated FTR service conditions.

A tensile testing matrix involving 60 as-fabricated weldment specimens has been completed. Included in this matrix are four welding processes (TIG, Submerged-Arc, Stick-Electrode, and MIG), three specimen types (weld metal, fusion line, and base metal), and four testing temperatures (600, 700, 800, and 900 °F). In addition, 20 specimens were annealed at 1200 °F and 1850 °F to determine whether residual stresses caused the observed yield strength increases and ductility decreases in weld and fusion line specimens relative to base metal values. Optical metallography is being employed to relate observed mechanical properties to microstructural features. These results will be used in conjunction with an EBR-II irradiation experiment and a thermal aging study to characterize the behavior of FTR vessel weldments under projected conditions.

Based upon the results of the tests described above and in the tensile data listed in Tables 12.24, 25, 26, and 28, the following preliminary conclusions are drawn:

- For each welding process, the weld and fusion line specimens exhibit similar strength and ductility values under

TABLE 12.24. *TIG Process, Tensile Properties*

<u>Specimen</u>	<u>Test Temp, °F</u>	<u>0.2% Yield Stress, psi</u>	<u>Ultimate Stress, psi</u>	<u>Uniform Elong, %</u>	<u>Total Elong, %</u>	<u>RA, %</u>
Weld	600	51,900	63,700	7.5	12.4	64
Weld	600	52,200	64,800	12.3	16.5	66
Weld	600	52,200	64,800	12.3	16.5	66
Weld	700	52,500	64,600	11.5	16.1	66
Weld	800	51,600	62,700	10.3	15.4	59
Weld	900	48,100	59,400	10.9	15.4	69
Fusion Line	600	58,500	70,800	6.6	12.1	74
Fusion Line	700	52,000	63,800	8.7	13.4	66
Fusion Line	800	55,500	67,600	13.3	17.5	59
Fusion Line	900	53,700	62,100	7.8	11.6	67
Weld(1200)*	800	43,200	57,700	11.0	16.1	62
Weld(1850)*	800	16,000	63,000	31.0	36.3	72
F.L.(1200)*	800	41,900	57,600	8.3	12.2	54
F.L.(1850)*	800	12,300	62,800	34.9	38.7	59

* Annealed at the indicated temperature (°F) for 1 hr prior to tensile test.

TABLE 12.25. *Submerged-Arc Process, Tensile Properties*

<u>Specimen</u>	<u>Test Temp, °F</u>	<u>0.2% Yield Stress, psi</u>	<u>Ultimate Stress, psi</u>	<u>Uniform Elong, %</u>	<u>Total Elong, %</u>	<u>RA, %</u>
Weld	600	38,000	66,200	22.0	23.7	40
Weld	600	44,400	67,500	18.7	20.8	45
Weld	700	33,500	66,900	28.6	30.9	46
Weld	700	41,100	58,000	18.2	19.2	50
Weld	800	37,900	61,000	18.5	19.5	35
Weld	900	34,800	63,500	24.0	25.8	39
Weld	900	34,800	56,300	17.1	18.5	35
Fusion Line	600	34,100	65,600	25.6	30.0	66
Fusion Line	600	46,500	65,400	19.5	24.9	76
Fusion Line	700	40,200	69,100	24.1	29.6	72
Fusion Line	700	41,600	63,500	21.1	25.7	73
Fusion Line	800	46,700	62,600	19.0	26.4	75
Fusion Line	800	31,600	62,500	26.2	32.8	66
Fusion Line	800	41,800	65,100	20.4	25.7	63
Fusion Line	900	31,600	59,500	22.9	29.6	70
Fusion Line	900	46,900	57,900	13.3	15.3	36
Weld(1200)*	600	32,600	63,900	17.4	21.1	44
Weld(1850)*	600	26,900	59,600	23.9	25.6	37
Weld(1200)*	700	27,900	60,300	21.9	23.6	36
Weld(1850)*	700	23,800	64,700	32.3	33.1	42
F.L.(1200)*	700	28,700	57,900	22.1	25.2	45
F.L.(1850)*	700	13,600	60,200	30.8	35.3	71
F.L.(1200)*	800	25,600	57,900	22.8	24.9	43
F.L.(1850)*	800	12,500	59,600	33.2	38.3	64

* Annealed at the indicated temperature (°F) for 1 hr prior to tensile test

TABLE 12.26. *Stick-Electrode Process, Tensile Properties*

Specimen	Test Temp, °F	0.2% Yield Stress, psi	Ultimate Stress, psi	Uniform Elong, %	Total Elong, %	RA, %
Weld	600	53,100	71,100	16.6	19.1	44
Weld	600	47,000	69,700	20.3	24.0	52
Weld	700	48,400	67,200	16.3	18.7	48
Weld	700	43,800	67,500	20.7	24.4	50
Weld	700	46,400	66,100	18.6	21.8	55
Weld	800	45,400	66,800	15.6	19.6	47
Weld	900	43,600	62,300	15.8	17.6	43
Weld	900	45,000	65,300	16.7	19.4	42
Fusion Line	600	51,200	65,400	15.0	20.0	70
Fusion Line	600	42,500	68,600	18.2	22.4	63
Fusion Line	700	46,700	68,100	20.9	26.7	
Fusion Line	800	45,500	68,400	16.7	22.0	71
Fusion Line	800	42,500	67,400	18.4	24.6	72
Fusion Line	800	43,100	64,800	20.5	27.5	66
Fusion Line	900	40,200	59,200	14.8	21.6	73
Fusion Line	900	46,800	63,600	18.5	23.1	66
Weld(1200)*	700	42,800	67,000	20.7	25.8	56
Weld(1850)*	700	15,100	62,000	30.7	34.6	74
Weld(1200)*	800	38,800	62,900	17.4	20.8	54
Weld(1850)*	800	14,400	61,000	32.4	38.3	67
Weld(1850)*	800	30,300	68,000	41.2	45.7	56
F.L.(1200)*	700	41,100	64,500	20.5	23.9	48
F.L.(1200)*	800	40,000	65,800	24.4	28.9	71
F.L.(1850)*	800	12,600	62,000	35.5	39.7	65

* Annealed at indicated temperature (°F) for 1 hr prior to tensile test.

TABLE 12.27. MIG Process, Tensile Properties

Specimen	Test Temp, °F	0.2% Yield Stress, psi	Ultimate Stress, psi	Uniform Elong, %	Total Elong, %	RA, %
Weld	600	44,300	63,900	11.4	15.1	56
Weld	600	45,000	67,600	19.4	21.5	61
Weld	600	35,200	63,900	17.3	20.8	59
Weld	600	41,500	61,700	20.2	23.8	50
Weld	700	46,200	66,200	19.5	21.9	37
Weld	700	39,800	67,800	18.6	23.1	71
Weld	700	37,300	61,600	21.8	25.6	50
Weld	700	39,800	66,200	22.0	26.9	61
Weld	800	48,600	64,300	14.7	19.2	57
Weld	800	39,800	61,300	13.2	17.7	64
Weld	800	36,200	60,600	21.6	25.6	59
Weld	800	34,400	60,100	21.9	24.9	55
Weld	900	48,400	59,600	11.1	14.5	51
Weld	900	35,800	56,700	14.8	19.4	67
Weld	900	37,500	56,600	15.9	19.4	57
Fusion Line	600	46,400	67,800	22.3	27.9	71
Fusion Line	700	45,100	62,700	15.2	19.5	57
Fusion Line	800	42,800	64,900	22.4	29.4	55
Fusion Line	900	41,600	59,000	16.8	19.7	50

TABLE 12.28. Base Metal, Tensile Properties

Specimen	Test Temp, °F	0.2% Yield Stress, psi	Ultimate Stress, psi	Uniform Elong, %	Total Elong, %	RA, %
As-Fab.	600	22,200	64,600	38.5	43.2	65
As-Fab.	700	24,700	61,000	33.6	38.9	62
As-Fab.	800	23,400	65,600	40.0	44.6	64
As-Fab.	900	18,300	56,900	27.6	35.4	67
Ann. 1850 °F	800	14,600	63,000	38.1	42.9	71

the same test conditions. Ductility of welds can be significantly lower than the ductility of base metal. Total elongation of some as-fabricated welds was as low as 15%, compared to 35% for the base metal. This value is already close to an end-of-life limit of 10% total elongation, without the additional effects of irradiation.

- Post-annealing property comparisons between weld and base metal specimens are complicated by variations in structure and composition; however, it is apparent that the increased strengths are the result of residual stresses in the weld metal. Minimal recovery of ductility was observed for a short anneal at 1200 °F, while full recovery was achieved at 1850 °F. These results indicate that very high annealing temperatures for a component as large as the vessel may be required if post-weld heat treatments are to be applied for improved ductility.
- Tensile test results and optical metallography suggest that the mechanical properties exhibited by fusion line specimens are those of the composite material--including weld metal, base metal, and heat affected zone--and are not controlled by a single component.

Four MK-B7 type pins were assembled and shipped to the EBR-II site for inclusion in subassembly number X067-4F2. Contained in the four pins are 159 tensile and creep-rupture weldment specimens; 21 solution-treated 304 stainless steel tensile specimens; 24 tensile specimens of 316 stainless steel in annealed and 10%, and 20% cold-worked conditions; and 20 low cycle fatigue specimens of 304 stainless steel. This experiment will be used to determine the effects of neutron irradiation on the mechanical properties of the weldment materials.

LMFBR Fuel and Cladding Information Center - F. R. Shober

An information center was established to document and to record all qualitative and quantitative information relating to the irradiation performance of LMFBR fuel pins. The system includes materials information on melting processes, fabrication techniques, heat treatments, mechanical properties, physical properties, quality inspection results, and performance records of individual lots of material. The system is a computerized information storage, retrieval, and data analysis system consisting of six sections: cladding fabrication, fuel fabrication, fuel pin fabrication, fuel subassembly fabrication, irradiating history, and postirradiation examination. The sectional nature of the system permits analysis and evaluation of data from each section, independent of others or, if preferred, allows a multisection analysis.

Activities of the information center presently include:

- Identification of terms and fields related to the expansion of the system.
- Preparation of a format for storage of data available on fabricated fuel-pins and experimental subassemblies.
- Identification of, and formation of, interfaces with data sources and information needs.
- Storage of data from stainless steel cladding procured by:
 - (a) BNW (lots G and H)
 - (b) Argonne National Laboratory
- Evaluation of data logging systems for reactor operation with respect to pertinent irradiation history section of system.

It appears that the major problems confronting the staff is to identify onsite and offsite sources of data and to interface with these sources.

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