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JANUARY, FEBRUARY, MARCH, 1970
REACTOR FUELS AND MATERIALS
DEVELOPMENT PROGRAMS
FOR FUELS AND MATERIALS BRANCH
OF
USAEC DIVISION
OF REACTOR DEVELOPMENT AND TECHNOLOGY
April 1970

AEC RESEARCH &
DEVELOPMENT REPORT
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1.0 SUMMARY

2.0) FAST-REACTOR CERAMIC FUELS RESEARCH

High Temperature Creep of Unirradiated Fast-Reactor Oxide Fuels

A study was undertaken to determine if the creep rates of UO$_2$ in bending exhibit the same nonreproducible behavior as has been observed in UO$_2$-20 wt% PuO$_2$. Although a similar behavior was observed, no single cause could be determined for the effect.

An analysis has been carried out to determine the effect of a nonlinear stress-dependent strain rate on the evaluation of stresses and strains in four-point bending.

In-Reactor Creep of Mixed Oxide Fuels

The second test capsule has been irradiated in the K-West Reactor for 80 hr. The problems encountered with the first capsule were eliminated by design modifications. One of the two test specimens broke 1 hr after irradiation began, and the other broke at some undetermined time. Breakage appeared to be related to microcracks in the sample.

Thermal Conductivity of (Pu,U)O$_2$

The thermal conductivities of stoichiometric UO$_2$-PuO$_2$ solid solutions containing 0, 5, 12, 20, 25, and 30 wt% PuO$_2$
were calculated from measured thermal diffusivity values. Additions of PuO$_2$ to the UO$_2$ lattice resulted in a slight but measurable decrease in thermal conductivity at all temperatures within the range investigated (100 to 1200 °C).

Properties of Molten Fast-Reactor Oxide Fuels

Measurements Facility

The fluoroscopic observation of the molten UO$_2$-PuO$_2$ fuels has been improved by the installation of an X-ray image intensifier.

Density

A gas pycnometer technique and a gamma attenuation technique are being evaluated as methods to improve the accuracy of measurement of the volume expansion on melting and liquid density of UO$_2$-PuO$_2$ fuels.

Surface Tension

A new furnace has been constructed to measure surface tension of UO$_2$-PuO$_2$ fuels by the pendant drop technique. The precision of surface profile techniques for surface tension measurement is limited to ±10% by the technique used to mechanically measure the profile.

Hot-Pressing of Fast-Reactor Ceramic Oxides

High density (U$_{0.75}$Pu$_{0.25}$)O$_2$ fuels have been fabricated by hot pressing. Specimens are being evaluated for physical property measurements.

Lattice Expansion of UO$_2$-PuO$_2$

A lattice thermal expansion of $9.8 \times 10^{-6}/°C$ has been measured for (U$_{0.75}$Pu$_{0.25}$)O$_2$ to 1600 °C. Temperatures were determined from measurement of the lattice expansion of molybdenum powder additions.
Phase Studies

The present micro-thermobalance apparatus is being modified to extend the temperature range for measurements of O/M ratios of mixed oxides as a function of oxygen potential.

Determination of Oxygen/Metal Ratios for Oxide Fuels

The existence of CO₂ sources other than that arising from the carbon reduction of the oxide fuel sample has continued to plague the development of a method of O/M determination on milligram quantities of oxide fuels. It may prove necessary to eliminate any exposure of the powdered graphite employed as a fuel reductant to gases which are subsequently released as oxides of carbon.

(3.0) IRRADIATION BEHAVIOR OF FAST REACTOR FUEL MATERIALS

In-Reactor Thermal Performance of UO₂-PuO₂ Fuel

Irradiation of eight UO₂-25 wt% PuO₂ fuel pins with variable fuel clad gaps and Xe/He ratios in the fill gas was completed. Initial postirradiation examination showed substantial gap closure for nominal FTR gap and peak heat rating conditions during a 100-hr test. Fuel with a nominal FTR gap operated with significantly higher fuel temperatures than did fuel with a minimum initial gap. Changing Xe/He progressively from 0 to 1 caused a commensurate and consistent increase in fuel temperature for both large- and small-gap pins.

Fission product molybdenum, zirconium, cerium, and ruthenium distributions were measured in UO₂-25 wt% PuO₂ fuels with various time and heat rating histories. Studies of high, fast-flux burnup effects on UO₂-25 wt% PuO₂ fuel melting heat rating were begun.

A survey and analysis of existing fuel-clad gap conductance data clearly showed that present knowledge is not adequate for fuel design purposes.
Irradiation of Hypostoichiometric UO$_2$-PuO$_2$ Fuel

Extensive precipitation of fission-product-metal inclusions and nuggets was observed in UO$_2$-25 wt% PuO$_2$ fuels irradiated to 75,000 to 100,000 MWd/MTM. Migration of plutonium toward the fuel center was also detected. Compositions of a number of metal and oxide fission product inclusions was studied with the shielded microprobe. Radial distributions of molybdenum, ruthenium, neodymium, cerium, zirconium, and xenon were measured. Iron-, nickel-, and, in some cases, chromium-bearing inclusions in the fuel were interpreted in terms of fuel-clad-fission product chemical interactions. The clad was found to contain both fuel and manganese inclusions.

Terminal gamma scans of fuel pins previously scanned at midlife showed gross redistribution of central, fission-product nuggets.

Fuel Swelling and Analytical Modeling

Scanning electron microscope examination of replicas from several EBR-II irradiated fuel pins is in progress. The same instrument is being used to characterize oxide starting materials. Results are being used to quantitatively evaluate fission gas bubble migration models. Model development was focused on characterizing size and distribution of fission gas bubbles trapped on grain boundaries.

Fabrication of fuel and components for the first in-reactor basic oxide swelling experiment was completed. The first assembly, which will contain 27 specimens with various fissile atom densities and geometries, will be irradiated in April to evaluate analytical predictions of specimen temperatures and fission rates.
Migration Behavior in Mixed Oxides

The major thermal-gradient-induced forces responsible for oxygen migration in fuels are being investigated theoretically and experimentally. These are the electronic component, characterized by the electronic thermo-electric power (electronic heat of transport) and the ionic component, characterized by the ionic heat of transport.

(4.0) IRRADIATION DAMAGE TO REACTOR METALS

Irradiation Facilities Operation

Operation of the ETR G-7 pressurized hot water loop has been discontinued. The pressure tube was removed in November 1969. A total of 402 corrosion, tensile, and creep-rupture specimens were discharged from the loop at the conclusion of cycles 103 and 104.

In-Reactor Measurements

In-reactor creep measurements in a Hanford K Reactor are compared with published data. Irradiation-induced creep strain data can be described as the sum of a fluence-dependent transient strain and a steady-state creep term. The irradiation-induced transient strain is larger in 20% cold-worked Type 316 SS than in solution treated Type 304 and 316 SS. Irradiation-induced creep decreases with increasing temperatures over the range of 43 to 370 °C. The greater amount of irradiation-induced creep obtained in the tensile test compared to the torsion test is attributed to the influence of the hydrostatic pressure component of the tensile stress on swelling.

Fracture Analysis

The results of the fractographic examination of Type 316 SS creep-rupture specimens are reported. Determinations are given for the effects of aging temperature, test
temperature, and irradiation on the fracture behavior (fracture mode and mechanism). Correlation between fracture mode and ductility are established and microstructural examination reveals the presence of a platelet form of precipitate at grain boundary microcracks. Increased crack propagation rates in irradiated material (potential flaw sensitivity) are demonstrated fractographically.

The Stability of Voids in Neutron Irradiated Nickel

Some additional information supplementary to that reported previously has been derived from experiments on the characteristics and stability of voids in nickel during postirradiation annealing treatments. The applicability was assessed of several small angle X-ray scattering methods for determining the distribution of void sizes in nickel irradiated to $3 \times 10^{21}$ n/cm$^2$ ($E > 0.1$ MeV) at 450 °C as a function of annealing. The stability of the voids during annealing is attributed in part to the presence of some gas atoms in the voids.

Grain Boundary Stabilization

Grain size stabilization of small grain nickel powder compacts (~10 μ grain size) has been achieved in ex-reactor annealing studies. Stabilization accomplished by a technique using helium-atom pinning of boundaries required a volume concentration of helium of only 0.3 ppm.

The effect of variation of injection parameters on the quantity and binding depth of the trapped gas has been investigated.

Irradiation Effects on the Fracture of Heavy Section Pressure Vessel Steels

Planning, engineering, and procurement has been completed for the installations of electrical heaters in the ETR M-3 loop to provide greater temperature flexibility during specimen
irradiation. Postirradiation annealing of A533-B pressure vessel steel specimens revealed that the reduction of irradiation hardening commenced above 540 °F and was 70% complete by 650 °F. A fluence increase from 2 to $4.4 \times 10^{19} \text{n/cm}^2$ ($E > 1 \text{ MeV}$) produced only a modest increase in strength. The fatigue crack behavior indicated the propensity for crack propagation hindrance.

**EBR-II Irradiations**

Pins BNW B41 and B42 are being consolidated with BNW Subassembly 5. The fourth in a series of five swelling experiments (Pin BNW 25) is being assembled for placement in EBR-II at the beginning of Run 41. A revised summary of current irradiation is presented.

**Damage Analysis**

Density measurements were made to determine the extent of irradiation swelling in a pin which was used to contain tensile specimens in Row 7 in the EBR-II. Both the total amount of swelling and the gradient in swelling along the axial length of the pin were less than that predicted by the most recent empirical swelling equation.

Empirical equations are presently being developed to describe the effects of neutron exposure and irradiation temperature on void size and concentration in Types 304 and 316 SS.

**Mechanical Testing of Fuel Pin Cladding**

Previously reported burst data on PNL-1 series fuel pins revealed a very low strength and ductility region above the reactor midplane. The low strength is thought to be due to grain boundary attack during testing of sensitized cladding.

Additional fueled cladding specimens from above the midplane of PNL 1-16 and 1-18 have revealed intergranular cracks
during room temperature leak testing. The orientation of these cracks suggests that the pins were subjected to a bending stress during storage.

Electron microprobe results, ring test results, and cellulose acetate replication findings are presented, all of which help narrow down the possible causes for the observed behavior.

Acceptance Testing and Clad Characterization

The characterization of PNL tube lots E, F, G, and H is essentially complete except for long-term stress-rupture data, and testing is continuing on PNL tube lots N-1 and N-2. A data handbook for each lot of tubing is now being assembled.

Tensile Studies of Types 304 and 316 Stainless Steels

Tensile test results from AISI Type 316 SS specimens irradiated in Row 2 of the EBR-II have been combined with previously reported data from the EBR-II 5C3 control rod thimble (Type 304), in order to clarify the roles of helium embrittlement and displacement damage in elevated temperature ductility losses. These materials represent helium concentrations in the range from 1 to 16 ppm (atomic). The study has shown that post-irradiation annealing at 2000 °F for 1 hr produces nearly complete restoration of the pre-irradiation tensile ductility at test temperatures below \( T_{m}/2 \), but only partial recovery at higher temperatures.

Uniaxial Creep-Rupture Studies of Types 304 and 316 Stainless Steels

A comparison of Types 304, 316, and 348 SS shows that the creep-rupture properties of the three alloys at 1000 °F are similar after irradiation to \( 1.2 \times 10^{22} \) n/cm\(^2\) (total) at \( \sim 900 \) °F, even though the alloys exhibited quite different properties prior to irradiation.
Weldment Studies

Results are reported for all-weld specimens representing the four weld processes included in the FTR vessel weldment program. These data show that tensile properties are essentially unaffected by irradiation to $1 \times 10^{21} \text{n/cm}^2$ (total) at 700 and 800 °F, while substantial strengthening and ductility loss are observed following irradiation to 3.8 and $4.4 \times 10^{21} \text{n/cm}^2$ (total) at 800 °F, except in the case of the stick-electrode weld where ductility is apparently unaffected by the higher fluence irradiation.

Tensile test results are also reported for weldment and base metal specimens of a modified design. The study was to determine whether a larger diameter (0.250-in. gage diameter) would provide an advantage over the 0.125-in. gage diameter miniature specimen used in the vessel weldment irradiation experiment.

Creep-rupture test results for all-weld specimens from two of the weld processes are reported. These results show that the submerged arc weldment and the TIG weldment respond differently to an irradiation to $8 \times 10^{21} \text{n/cm}^2$ (total) at 1100 °F.

(5.0) FAST REACTOR DOSIMETRY AND DAMAGE ANALYSIS

Damage Functions and Data Correlation

A preliminary form of a damage function has been obtained for a change in yield strength to 60 ksi in Type 304 SS irradiated and tested at $\sim 450 ^\circ \text{C}$. The derived damage function is used to evaluate a parameter in an equation that relates the measured property change to total fluence. This allows the application of available test reactor data to other spectral environments of interest in the development of Liquid Metal Fast Breeder Reactors.
SAND-II Solution Accuracy Study for EBR-II Spectra

A study has been initiated to more precisely define the accuracy of integral fluxes determined by the SAND-II code for the EBR-II run 31F dosimetry test. Accuracy limits due to uncertainties in cross sections and reaction rate measurements will be defined by a computerized error propagation technique. From the initial steps in the study, uncertainties due to lack of solution uniqueness are estimated to be no more than about 4% (1σ) in the core and bordering areas and about 25% (1σ) in the blanket regions.

Fast Reactor Materials Dosimetry Center

The Fast Reactor Materials Dosimetry Center data file has been expanded by the addition of unfolded neutron spectra. Axial flux distribution for any particular radius above or below any specified energy, among other data, can now be obtained in computer plotted or tabulated form by request. Aside from data applicable to past experimentation, the center can also provide data estimates applicable to future EBR-II irradiations.

Computer Simulation of Radiation Damage

The overlap of 20 keV displacement cascades in α-Fe was simulated with the CASCADE-CLUSTER and HAP-4 computer codes. The result was a noticeable increase in both maximum and average cluster sizes. A parameter study with HAP-4 indicates that, for short term annealing simulations, a 112-site isotropic recombination region may be equivalent to the 62-site anisotropic region presently employed.

Computer Tabulation of Dounreay Fast Reactor Flux and Reaction Rate Data

DFR flux and reaction rate data have been incorporated into a computer library and analysis program. Highly detailed
flux and linear heat generation profiles can now be obtained for any pin or subassembly irradiated within the DFR core or blanket.

Computer Simulation Studies of Dislocation Core Structure

Dislocation structures have been modeled using the GRAINS program. This modeling is a necessary step in determining the parameters and mechanisms for creep and swelling in structural materials.

Fluence Determinations Using Time-Varying Flux

A computer program, TIMH, has been written to use reactor power history data to determine flux and fluence by foil activation analysis. This means of flux and fluence calculation avoids excessively large errors by accounting for the effects of reactor cycling.

(6.0) FAST REACTOR ABSORBER MATERIALS

Irradiation in a Thermal Flux

Irradiation experiments have been completed on B₄C in a thermal flux. B₄C powders and 65, 80 and 99% TD pellets were irradiated to a maximum exposure of $15 \times 10^{20}$ captures/cm$^3$ at temperatures ranging from 500 to 1200 °F. Gas release fractions increased with temperature from about 10% at 500 °F to 25% at 1200 °F.

Fast Reactor Irradiation

Postirradiation examination of samples in the tantalum irradiation capsule, TA-5, has been completed. Annealing of the silicon carbide temperature monitor in the capsule indicated a maximum temperature of 2100 °F, considerably higher than the calculated temperature of 1650 °F. Swelling measurements show density changes of ~0.5% for a sample irradiated at 2100 °F to an exposure of $8.6 \times 10^{21}$ nvt ($E > 0.1$ MeV) and a change of 0.8% for a sample irradiated at 1300 °F to an exposure of $3 \times 10^{21}$.
(7.0) **NUCLEAR GRAPHITE**

**Dimensional Changes of Graphites at High Neutron Fluences and High Temperatures**

Irradiation data have been obtained on a variety of conventional and experimental graphites at temperatures up to 1400 °C and fluences up to $1.4 \times 10^{22}$ n/cm$^2$ ($E > 0.18$ MeV). Post-turnaround expansion of at least some graphites is less rapid at temperatures above 1200 °C than at temperatures of 900 to 1200 °C. Graphites manufactured by POCO Graphite Inc. exhibit smaller dimensional changes and less rapid expansion at high fluences than do conventional graphites.

**Computer Calculations of Graphite Oxidation in HTGR**

A steady state of H$_2$/H$_2$O ratio of 28.3 is calculated for normal operation of the 1100 MW$_e$ HTGR based on current estimates of the leak rate and purification factor as obtained from Gulf General Atomic.

**Radiolysis of H$_2$-CO$_2$-He Mixtures**

A study of the radiolysis of a mixture of carbon dioxide and hydrogen in helium shows that the radiolytic rate decreases with a decrease in the relative concentrations of CO$_2$ and H$_2$. At relative concentrations of H$_2$ and CO$_2$ greater than 1000 ppm the 100 eV yield of products is about 1.8 molecules/100 eV which decreases slowly with relative concentrations to a value of less than 1.0 molecule/100 eV at 50 ppm. At concentrations in an HTGR the product yield would have a very low value.

(8.0) **NONDESTRUCTIVE TESTING**

**Eddy Current Methods**

A multiparameter eddy current tester has been used to solve a practical tube testing problem. This technique makes it possible to test for flaws both on the inner and outer
surface of a tube even though the flaws are under simulated tube supports. The advantages of using multiple frequencies for practical testing problems are presented in this study.

Ultrasonic Methods

Phase measurements of reflected ultrasonic waves incident near the Rayleigh critical angle are reported. These measurements substantiate the predictions of our model which differ substantially from previous theories. The critical angle is sensitive to changes in material properties such as elastic modulus and density.

Research into the selection and development of alloys for use in the core assembly of the Fast Flux Test Facility (FFTF), has indicated that for a material to mechanically survive the high fluence environment anticipated, its structural state (e.g., grain size and cold work) is of critical importance. To ensure that the specified requirements of structural state have been met, nondestructive evaluation of the core material, especially in fuel pin tubing and fuel bundle ducting, is mandatory. A testing methods program is described making use of critical angle ultrasonic techniques.

A series of measurements have been made to identify flaw size and shape by the use of wide bandwidth ultrasonic pulses and appropriate spectrum analysis techniques. Differences in the wide bandwidth spectrum are indicated for a series of flat disks, spheres, and ribbons of different sizes. Further study is required before practical implementation of a flaw identification ultrasonic system can be designed and built.

Thermal Methods

A full scale experimental thermal image transducer is described which was designed to test conical specimens. Also, an emittance-independent, noncontacting thermal-transducer
Holographic Methods

Interferometric holography has been applied in a non-destructive manner to a number of sample configurations. These applications include double exposure holograms of pressurized Fast Flux Test Fuel reactor cladding pin weld samples and thermal conductivity gradients in honeycomb construction panels. Weld defects and annealed areas were resolved in the tubing. Bond discontinuities and void regions in the honeycomb panels correlated with results from a previous thermographic test.

(9.0) ATR WATER LOOP OPERATION AND MAINTENANCE

The ATR accumulated 6800 MWd at 185 MW during Cycle 1 ending in February. Cycle 1B ended in March after an accumulation of 3700 MWd. The 1D-N loop, which operated during these periods at design conditions, contained stainless steel tensile specimens and Zircaloy corrosion coupons.
2.0 FAST-REACTOR CERAMIC FUELS RESEARCH

HIGH TEMPERATURE CREEP OF UNIRRADIATED FAST-REACTOR OXIDE FUELS
O. D. Slagle

Considerable difficulty has been experienced in obtaining reproducible strain rates as a function of stress for UO$_2$-20 wt% PuO$_2$ under four-point bending.\(^{(1,2)}\) One possible cause is microstructural alteration produced when large stresses are applied which subsequently change the strain rates obtained for low stresses.

Because of difficulties induced in obtaining suitable samples of UO$_2$-20 wt% PuO$_2$, tests were initiated on stoichiometric UO$_2$ to determine if a similar effect could be observed. The UO$_2$ samples had a density of 96.5% TD and a grain size of 14 µ. A series of measurements was carried out at 1495 °C in which loads were varied between stresses of 2100 psi and 8320 psi. All stresses and strains refer to the calculated values for the area under maximum tensile stress. The initial steady-state strain rate of 2100 psi was $3.4 \times 10^{-4}$ hr$^{-1}$. After applying a load of 8320 psi, little change occurred in the strain rate at 2100 psi. At a total accumulated strain of 1.6%, a series of loads: 8320, 6960, 5270, 3690, and 2100 psi were applied. The strain rate for 8320 psi dropped 25% from the previous measurement; however, a plot of log strain rate versus log strain (Figure 2.1) for these five stresses fell on a line with a slope of 2.08. The strain rate at 2100 psi after this series of tests and an accumulated strain of 2.5% was $1.21 \times 10^{-4}$ hr$^{-1}$ or about half that at 1.6% strain. Further straining to 4% strain resulted in an initial decrease in strain rate to $0.85 \times 10^{-4}$ hr$^{-1}$, then an increase to $1.23 \times 10^{-4}$ hr$^{-1}$, and finally a decrease to $0.89 \times 10^{-4}$ hr$^{-1}$. These tests gave no firm conclusion as to what is responsible for changing creep rates during testing.
FIGURE 2.1. Plot of Maximum Strain Rate Versus Maximum Stress for UO$_2$ at 1495 °C

MAXIMUM STRESS, 10$^3$ psi

MAXIMUM STRAIN RATE, hr$^{-1}$

n = 2.08
A second sample was used to determine the activation energy for creep of stoichiometric UO$_2$ for comparison with existing data. A series of strain rates were measured for a stress of 3420 psi at temperatures of 1495 °C and 1410 °C. The least amount of scatter in the data occurred for strains between 2.3% and 3.1%, and strain rates versus time decreased evenly at both temperatures. The data is listed in Table 2.1. The activation energy was calculated from an average of the strain rates observed, and was found to be 106 kcal/mole, in good agreement with existing values.

TABLE 2.1. Comparison of Maximum Strain Rates at 1495 °C and 1410 °C for a Stress of 3420 psi to Determine Activation Energy. Data Were Taken in the Order Listed. Strain Rates Are in Units of 10^-4 hr^-1.

<table>
<thead>
<tr>
<th>Maximum Strain Rate</th>
<th>Temperature, °C</th>
<th>Maximum Strain Rate, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.87</td>
<td>1495</td>
<td>0.80</td>
</tr>
<tr>
<td></td>
<td>1410</td>
<td></td>
</tr>
<tr>
<td>3.09</td>
<td>1495</td>
<td>0.70</td>
</tr>
<tr>
<td></td>
<td>1410</td>
<td></td>
</tr>
<tr>
<td>2.84</td>
<td>1495</td>
<td>0.52</td>
</tr>
<tr>
<td></td>
<td>1410</td>
<td></td>
</tr>
<tr>
<td>2.55</td>
<td>1495</td>
<td></td>
</tr>
</tbody>
</table>

From the tests on these two samples, it is evident that obtaining reproducible strain rates as a function of stress, strain, or temperature in four-point bending is a problem common both to UO$_2$ and UO$_2$-20 wt% PuO$_2$.

Calculation of maximum stress and strain rate for creep in four-point bending is commonly done by the use of expressions derived for elastic behavior which assume both small deflections and stress proportional to strain. Creep
measurements in mixed oxides have indicated that creep rates are not proportional to the applied stress. Therefore, an analysis has been carried out for the case where strain rate ($\dot{\varepsilon}$) is proportional to stress ($\sigma$) raised to the $n$th power or

$$\dot{\varepsilon} = B \sigma^n$$  

(1)

where $B$ is a proportionality constant.

The previous work of Popov\(^{(3)}\) has been extended to relate push rod deflection rate ($\dot{w}$) and the applied load ($P$). The resulting expression is

$$\dot{w} = B \frac{6c}{bh^2} \frac{c(3L - 4c)}{3h}.$$  

(2)

The different symbols are defined in Figure 2.2. For $n = 1$, this expression reduces to

$$\dot{w} = B \frac{6c}{bh^2} \frac{c(3L - 4c)}{3h}.$$  

(3)

Equation (3) agrees with elastic theory when compared with the expressions for maximum strain rate ($\dot{\varepsilon}_m$) and stress ($\sigma_m$)

$$\dot{\varepsilon}_m = \frac{c(3L - 4c)}{3h} \frac{1}{w}$$  

(4)

and

$$\sigma_m = \frac{6c}{bh^2} P.$$  

(5)

The importance of Equation (2) may be realized by comparing the values of $B$ which are computed using Equation (2) and those which would result from inserting Equations (4) and (5) into Equation (1) or

$$\dot{w} = B \frac{6c}{bh^2} \frac{c(3L - 4c)}{3h}.$$  

(6)
Denoting $B$ calculated for the creep case in Equation (1) as $B_{cr}$ and for the elastic case in Equation (6) as $B_{el}$, then for given measured values of $\dot{\omega}$ and $P$ the ratio of $B_{cr}/B_{el}$ can be calculated as a function of $n$. The results of this calculation are plotted in Figure 2.3. For example, for the UO$_2$ data obtained in this quarter which had an $n$ value of 2.08, the actual value of $B$ would be 60% higher than that calculated using the elasticity relations.
FIGURE 2.3. Ratio of the Proportionality Constant Calculated Using Equation (2) to That Calculated Using Equation (6) Plotted Versus Stress Exponent
The second in-reactor creep test of UO$_2$-20 wt% PuO$_2$ fuel was completed during this quarter. Capsule modifications were incorporated in this test which eliminated problem areas found in the initial test. An internal view of the modified creep test capsule is shown in Figure 2.4.

In order to eliminate the spilling problem caused by rotation of the capsule during insertion, a tilt mechanism was developed and used. This device signaled the technicians when the capsule was rotated approximately 5° in either direction from the vertical. The second improvement was the use of a gas bellows to apply the load to the specimens and thus prevent the bouncing of the previously-used dead weights. This technique allowed the capsule to be loaded into the reactor with essentially no load on the samples. The third change involved moving the heater out of the tin to prevent mass transfer and also resulted in less electric power being needed to maintain the temperature at 900 °C.

The experiment ran in the K-West Reactor for 4 days. The temperatures recorded by the two thermocouples were between 875 and 900 °C during the entire run. The load on the bellows was kept at 200 psi gage which resulted in a load of 450 g on one sample and 800 g on the other. The maximum stresses on the two samples were 413 psi and 735 psi, respectively.

The sample breakage signal was observed on the sample with the 413 psi load approximately 1 hr after insertion. The second signal was received approximately 2 hr later, but was found to have resulted from a short in the system. When the capsule was examined in the hot laboratory, both samples were found to be broken.
Recent out-of-reactor creep tests have resulted in some mixed oxide samples breaking after very short times under light loads. Investigation of the cause of this problem is in progress. Microstructural examination of untested samples has indicated the presence of crack networks which are likely to be extended by stress and result in failure during testing.

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

The purpose of this study was to determine the effect of PuO₂ content on the thermal conductivity of stoichiometric (Pu,U)O₂ solid solutions. These data are required to estimate what effect plutonium redistribution during irradiation might have on the thermal conductivity of the fuel element. The thermal diffusivities of samples of UO₂, UO₂-5 wt% PuO₂, UO₂-12 wt% PuO₂, UO₂-30 wt% PuO₂, and UO₂-45 wt% PuO₂, all prepared by coprecipitation from a nitrate solution, have been previously reported.(4) The thermal diffusivities of UO₂-20 wt% PuO₂, UO₂-25 wt% PuO₂, and PuO₂ have now been measured to complete the solid solution range of interest. During the last quarter, the thermal conductivities of each of the above oxides have been calculated in the temperature range 100 to 1200 °C.

Thermal conductivity was calculated using the relationship

\[ \lambda (W/cm\cdot{}°C) = 4.186 \alpha \rho C_p \]  

(1)

where

\[ \alpha = \text{thermal diffusivity, cm}^2/\text{sec} \]
\[ \rho = \text{density, g/cm}^3 \]
\[ C_p = \text{heat capacity, cal/g\cdot{}°C}. \]

Since thermal diffusivity was the only parameter measured as a function of temperature, it was necessary to assign values for
density and heat capacity. The density at room temperature was measured by immersion in FC-75 (an organic liquid). The samples were first coated, however, with a thin layer of collodion to assure that bulk density was determined. The variation of density with temperature was calculated from thermal expansion data. Because of thermal expansion, it was also necessary to recalculate previously reported thermal diffusivity data since it was found that neglecting changes in sample thickness due to thermal expansion would result in as much as 6% error in thermal diffusivity at 1200 °C. Heat capacity data for the mixed oxides were calculated using heat capacities reported for UO$_2$\(^{6}\) and PuO$_2$,\(^{7}\) assuming a Kopp's law relationship.

Calculated thermal conductivities are plotted as a function of temperature in Figures 2.5 through 2.11. The data for each of the different samples obey the relationship

$$
\lambda = (A + BT)^{-1}
$$

which is as expected for dielectric solids. Values of A and B, determined from a least squares analysis, are shown in Table 2.2.

The results of this study for UO$_2$ are compared in Figure 2.5 with the UO$_2$ thermal conductivity data of Godfrey et al.\(^{8}\) which was measured directly using a radial heat flow steady-state technique. The data are in reasonable agreement considering the possible differences in samples such as impurity content, microstructure, and stoichiometry.
$I = 0.0836 \text{ cm}$


data corrected to 96% TD

least squares fit to

$\lambda = (A + BT)^{-1}$

**FIGURE 2.5.** Thermal Conductivity of UO$_2$
FIGURE 2.7.
Thermal Conductivity of U$_{0.88}$Pu$_{0.12}$O$_2$

THERMAL CONDUCTIVITY $\times 10^2$, W/cm$^\circ$C

LEAST SQUARES FIT TO MEASUREMENTS DURING COOLING

$\lambda = \frac{1}{(A + BT)}$

UO$_2$ THIS STUDY (TRU) 96% TD
(L = 0.0655 cm)
U$_{0.88}$Pu$_{0.12}$O$_2$ (TRU) 97% TD

$\lambda = (A + BT)^{-1}$

- Measurements during cooling

BNWL-1349-1
FIGURE 2.8. Thermal Conductivity of $U_{0.80}Pu_{0.20}O_2$
FIGURE 2.11. Thermal Conductivity of PuO₂
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>% TD</th>
<th>% PuO₂</th>
<th>A cm⁻²°C/W</th>
<th>B cm/W</th>
<th>Std. Dev. for Single Mea.</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>TR-0</td>
<td>96</td>
<td>0</td>
<td>3.08 ± 0.04</td>
<td>0.02294 ± 0.00004</td>
<td>±0.9006</td>
<td></td>
</tr>
<tr>
<td>TR-5</td>
<td>97</td>
<td>5</td>
<td>3.04 ± 0.09</td>
<td>0.0239 ± 0.0001</td>
<td>±0.8043</td>
<td></td>
</tr>
<tr>
<td>TR-12</td>
<td>97</td>
<td>12</td>
<td>2.20 ± 0.03</td>
<td>0.02714 ± 0.00003</td>
<td>±0.7665</td>
<td></td>
</tr>
<tr>
<td>TR-20</td>
<td>98</td>
<td>20</td>
<td>3.09 ± 0.13</td>
<td>0.0261 ± 0.0001</td>
<td>±1.2279</td>
<td></td>
</tr>
<tr>
<td>TR-25</td>
<td>97</td>
<td>25</td>
<td>3.13 ± 0.10</td>
<td>0.0260 ± 0.0001</td>
<td>±1.1557</td>
<td></td>
</tr>
<tr>
<td>TR-30</td>
<td>97</td>
<td>30</td>
<td>5.30 ± 0.09</td>
<td>0.0252 ± 0.0001</td>
<td>±0.9222</td>
<td>Corrected to 97% TD from 92% TD</td>
</tr>
<tr>
<td>GCW-10</td>
<td>97</td>
<td>100</td>
<td>0.46 ± 0.18</td>
<td>0.0283 ± 0.0002</td>
<td>±1.670</td>
<td>Corrected to 97% Td from 93.3% TD</td>
</tr>
</tbody>
</table>
The data for \((U,Pu)O_2\) solid solutions and PuO\(_2\) are compared with the data for UO\(_2\) in Figures 2.6 through 2.11. These comparisons make it readily apparent that PuO\(_2\) substitution into the UO\(_2\) lattice has a small but measurable effect on thermal conductivity. The decrease in thermal conductivity resulting from addition of PuO\(_2\) to the UO\(_2\) lattice is small in comparison, however, to decreases resulting from deviations from stoichiometry in mixed oxide compositions.\(^{(9)}\) It has been shown\(^{(9)}\) that increases in thermal resistance arising from deviations from stoichiometry are associated primarily with the term A in Equation (2). This term can be associated with thermal resistance arising from the presence of lattice defects which scatter phonons. As shown in Table 2.2, the slight increases in thermal resistance resulting from addition of plutonium to the lattice in this study appear, on the other hand, to be associated with the term B in Equation (2), which is related to the thermal resistance contribution arising from phonon-phonon interactions called Umklapp processes.

It appears at this time that the changes observed in the Umklapp resistance with plutonium content can be explained theoretically and, in fact, are related to changes in melting temperature which also occur on addition of plutonium.

**PROPERTIES OF MOLTEN FAST-REACTOR OXIDE FUELS**

R. P. Nelson, J. J. Rasmussen and O. D. Slagle

**Measurements Facility**

Improvements have been made in the facility (Figure 2.12) which will be used to measure volume expansion on melting and liquid density, surface tension, viscosity, and compressibility of UO\(_2\)-PuO\(_2\) fuels.

A high resolution, high sensitivity (brightness) X-ray image intensifier tube has been substituted for the fluorescent screen previously used in fluoroscopy of molten samples. This
FIGURE 2.12. Facility for Measurement of Properties of Molten UO₂-PuO₂ Fuels
change will facilitate the observation of melting and the condition of the liquid in density, surface tension, and viscosity measurements.

Provisions have been made in the glove box to install a new furnace (Figure 2.13) for measurement of surface tension by the pendant drop technique. This furnace will be operated alternately with the tungsten-mesh resistance furnace. Both furnaces will use a common power supply and radiographic system.

A rapid advance roll-film cassette has been designed and will replace the single sheet cassettes used previously. The cassette has been designed to be used in conjunction with the image intensifier, permitting simultaneous fluoroscopy and film radiography. This arrangement will be particularly useful in rapidly obtaining radiographs of \( \text{UO}_2-\text{PuO}_2 \) pendant drops which are inherently unstable due to the high density of the molten oxides.

Density

Two techniques are being investigated which may lead to an improvement in the accuracy of measurements of the volume expansion on melting and liquid density of \( \text{UO}_2-\text{PuO}_2 \) fuels. Liquid density of refractory oxides is currently determined by using radiography to measure the height of a liquid column of known mass and diameter.\(^{10}\) The accuracy of the radiographic technique is limited to about \( \pm 30\% \) by the accuracy of determining the average column height.

One technique under consideration will employ a high-temperature gas pycnometer.\(^{11}\) A tube extending from a refractory metal capsule containing the oxide sample will connect to a limited-volume gas system outside the furnace. Melting will result in a change in pressure of an inert cover gas as measured on a sensitive capacitance-type pressure gage,
FIGURE 2.13. Furnace for Surface Tension Measurements by Pendant Drop Technique
which may be related to the volume expansion. Since it is anticipated that rapid measurements may be made, this technique is potentially useful for measuring compositions such as PuO$_2$ which react to some extent with the refractory metal crucible. The tungsten-mesh furnace in the measurements facility can be adapted for this measurement.

The second technique for determining precise density will involve measuring the attenuation of a gamma ray beam in the sample above and below the melting point. Measurements of molten metal densities using this method have been reported.\(^{(12,13)}\) The tungsten-mesh furnace can easily be adapted for this measurement. A $^{60}$Co source and a simple electronic counting system consisting of a scintillation detector, single-channel analyzer, and scaler/timer will be employed.

Both techniques will be tested for measurement precision using Al$_2$O$_3$ and UO$_2$. Equipment has been assembled and construction of the systems has been initiated.

**Surface Tension**

A new furnace has been built to measure the surface tension of UO$_2$-PuO$_2$ (Figure 2.13) by the pendant drop technique (Figure 2.14).\(^{(14)}\) This furnace is a double-shelled aluminum furnace which is completely water cooled. The unit has a D-shaped cross section which allows the radiographic film to be placed within 2-1/2 in. of the capsule center. A smaller capsule-film distance will reduce the geometric uncertainty which has been observed previously.

The surface profile computer program error was analyzed by rounding off sessile drop data reported by Butler and Bloom\(^{(15)}\) which had been measured to 0.00001 in. As long as the data was accurate to 0.001 in. or better, the error was less than 1%. If the data was only accurate to 0.01 in.,
FIGURE 2.14. Capsule Design for Pendant Drop Measurement of Surface Tension
the error jumped to 17%. In current studies, scribe marks used to outline the meniscus or the pendant drop profiles on enlarged prints made from radiographic negatives can be read to ±0.001 in. Since the standard deviation of the calculated UO₂ surface tension is about 10%, the greatest source of error comes from not being able to accurately place the scribe marks on the photographic enlargements. Preliminary tests indicate that a commercially available microdensitometer can be used to obtain the profile data to 0.001 in. optically.

HOT-PRESSING OF FAST-REACTOR CERAMIC OXIDES
P. E. Hart

Mixed oxide fuel specimens, containing 25 wt% PuO₂ and having densities in excess of 95% TD, have been fabricated via hot-pressing for use in in-reactor and out-of-reactor creep tests.

Specimens are being fabricated from coprecipitated plutonium hydroxide and ammonium diuranate powders calcined for 8 hr at 850 °C under flowing Ar-8% H₂. Hot pressing was previously performed at temperatures up to 1300 °C. However, specimens did laminate on reheating to 1600 °C. Consequently, pressing temperatures have been increased to 1600 °C. The present calcining technique yields powders with O/M's that fall between 2.01 and 2.02. Higher initial O/M's yield pellets with lower densities. The dependence of densities on O/M probably results from O₂ release during reheating. Due to the relatively short time at elevated temperatures during hot-pressing, not enough strength is developed to prevent laminating when O₂ is released during reheating. By increasing the pressing temperature, the specimen strength has been increased and the excess O₂ concentration reduced.

Density versus temperature curves for hot pressing of 25 wt% PuO₂ specimens are shown in Figure 2.15. Autoradiographs
FIGURE 2.15. Influence of Pressing Temperature and Powder Preparation on Density of Hot-Pressed UO$_2$-25 wt\% PuO$_2$; Pressure = 5000 psi
of the unmilled powder specimens indicate that some areas contain high Pu concentrations. Calcined powders ball milled in ethyl alcohol yield reduced hot-pressed densities; PNL HP-86 density equals 10.60 g/cm$^3$. The influence of milling on plutonium distribution has not been determined yet. The deactivation may be due to changes in surface adsorbed ions. Microstructures of this hot-pressed pellet are being examined.

As previously reported, (2) microstructures of hot-pressed UO$_2$ powders, initially having high O/M (above 2.2) exhibited two phases. Pressed pellet O/M ratios were approximately 2.04. This O/M corresponds to about 2 mole % U$_4$O$_9$. The pellets exhibited microstructures typical of U$_4$O$_9$ in a UO$_2$ matrix as reported by Schaner. (16)

Density of UO$_2$ specimens hot-pressed at low temperatures strongly depends on powder initial O/M. As shown in Figure 2.16 for specimens pressed at 900 °C, small changes in O/M cause significant changes in final density. These results reflect like influence of O/M on creep behavior of UO$_2$.

A typical microstructure of hot-pressed UO$_2$ specimens is shown in Figure 2.17. Specimen density is 10.90 g/cm$^3$, O/M = 2.01 and grain size is 14 μ. The specimen bulk is free of cracks. However, pressing defects appear on pellet surfaces.

LATTICE EXPANSION OF UO$_2$-PuO$_2$

P. G. Pallmer

A lattice thermal expansion of $9.8 \times 10^{-6}/°C$ has been measured for (U$_{0.75}$Pu$_{0.25})_2$ to 1600 °C. The temperatures were obtained from the lattice expansion of added molybdenum powder. The measured values were corrected for misalignment by extrapolation to 180°-2θ versus the function $\cos \theta \cot \theta$. The extent of misalignment resulting from expansion of the ribbon appeared to vary uniformly with temperature. The same
FIGURE 2.16. Influence of Powder O/M on Hot-Pressing Behavior of UO₂ at 900 °C.

Pressure = 4000 psi

PNL HP-79, O/M = 2.16
PNL HP-78, O/M = 2.12
FIGURE 2.17. Microstructure of Hot-Pressed UO$_2$; Density = 10.90 g/cm$^3$ (PNL HP-55)
correction was used for the single molybdenum reflection used as a temperature reference. The temperatures were read from curves fitted to published parameter values.\(^{(17,18)}\) The values are listed in Table 2.3, and plotted in Figure 2.18.

**TABLE 2.3.** Lattice Expansion of \((\text{U}_{0.75}\text{Pu}_{0.25})\text{O}_{2.00}\)

<table>
<thead>
<tr>
<th>Lattice Parameter</th>
<th>Molybdenum, Å</th>
<th>Oxide, Å</th>
<th>Temperature, (^{(a)}) °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.1466</td>
<td>5.4530</td>
<td></td>
<td>25</td>
</tr>
<tr>
<td>3.1548</td>
<td>5.476</td>
<td></td>
<td>505</td>
</tr>
<tr>
<td>3.1612</td>
<td>5.492</td>
<td></td>
<td>825</td>
</tr>
<tr>
<td>3.1650</td>
<td>5.498</td>
<td></td>
<td>995</td>
</tr>
<tr>
<td>3.1700</td>
<td>5.513</td>
<td></td>
<td>1215</td>
</tr>
<tr>
<td>3.1740</td>
<td>5.523</td>
<td></td>
<td>1390</td>
</tr>
<tr>
<td>3.1744</td>
<td>5.524</td>
<td></td>
<td>1410</td>
</tr>
<tr>
<td>3.1800</td>
<td>5.539</td>
<td></td>
<td>1640</td>
</tr>
</tbody>
</table>

\(^{(a)}\) Temperatures derived from the expansion of molybdenum from 25 °C.

Powdered samples of the coprecipitated stoichiometric dioxide were affixed to an aligned tungsten ribbon heater. After the vertical tungsten ribbon is aligned in the diffractometer, as shown in Figure 2.19, the stage is moved to a horizontal position for application of the powders as a slurry. Alignment is recovered with the aid of auxiliary clamps and shims, as shown in Figure 2.20. A temperature gradient in the oxide powder layer influences the temperature of the admixed molybdenum powder and its normally well-resolved lines were also broadened. To minimize thermal gradients, an additional U-shaped ribbon was positioned in front of the sample ribbon to function as a heat shield. The diffracted beam was occulted from about 130 to 140\(^{\circ}\) 20 and though resolution was improved, line broadening was still present above 1600 °C. The
FIGURE 2.18. Lattice Expansion of $(\text{U}_{0.75}\text{Pu}_{0.25})_2\text{O}_{2.00}$
Neg 700669-4

**FIGURE 2.20.** Vacuum Chamber for High Temperature Lattice Parameter Measurements on the X-Ray Diffractometer
thickness of the applied powder layer now appears to have the greatest effect on line broadening at high temperature. The broadening results from a temperature gradient arising because of the low absorptivity of the oxide.

Further improvement in the accuracy of the thermal expansion values may benefit from more direct temperature measurements. Nominally corrected optical pyrometer readings as well as temperatures indicated by tungsten lattice thermal expansion are somewhat lower than the molybdenum temperatures. A helium atmosphere did not lead to differing expansion values. The helium cools the ribbon but apparently does not efficiently transfer heat to the sample, although some reduction in line broadening was noted. Tungsten-rhenium alloy thermocouple wires have been spot welded to the back of the tungsten ribbon to provide a more direct measurement of the temperature.

PHASE STUDIES
C. E. McNeilly

Modification of the micro-thermobalance apparatus for use at higher temperatures is in progress in order to extend the measurement of O/M ratio of mixed oxides as a function of oxygen potential to a temperature of at least 1500 °C from the present limit of about 1100 °C. The present upper temperature limit is determined by the maximum temperature to which the alumina reaction tubes can be heated in hydrogen atmospheres containing low oxygen potentials (<-110 kcal/mole O₂). We have been attempting to replace the alumina with thoria tubes, but this has proved extremely difficult since thoria tends to fail in the present experimental arrangement after only a few heating-cooling cycles.

This problem will be circumvented by protecting the alumina from the reducing atmosphere by means of a close-fitting liner made from 0.001-in. thick molybdenum. The liner
has been fabricated and will be installed and tested and, if satisfactory, experiments should be started soon.

In order to cover the stoichiometry range of interest (1.90 < O/M < 2.00) in the temperature interval of 1100 to 1500 °C, the oxygen potential must be controlled over the range of about -100 to -140 kcal/mole O₂. This region of oxygen potentials, coupled with the temperature dependency in the equilibrium constant for the H₂/H₂O equilibrium, requires wide variations in the controlled parameters for the sample atmosphere. The two parameters most amenable to control are the partial pressures of hydrogen and water vapor. Hydrogen content is controlled manometrically whereas the water vapor partial pressure is varied by controlling the temperature of a water (ice) trap through which the hydrogen gas circulates. The most severe case [\(\Delta\overline{\theta}(O₂) = -140\) kcal/mole O₂ at 1200 °C] calls for an ice bath temperature of -51.4 °C. This temperature is attained by means of a dry-ice-boosted, mechanically refrigerated cryostat. By using a thermistor temperature controller with the thermistor sensing element cemented to the cooling coils, the cryostat temperature can be controlled to better than ±0.1 °C.

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, on the order of 1 mg, would allow the measurement of oxygen distributions across the diameters of fuel elements. The reduction of a fuel sample of this magnitude by carbon yields an amount of CO₂ which may be readily analyzed chromatographically. However, the precision of the determination depends upon the elimination of extraneous sources of CO₂. In the present study, the graphite crucible initially employed was replaced by a tungsten crucible because
of outgassing difficulties with the graphite crucible. Even the small amount of powdered graphite currently employed is not readily relieved of its oxides of carbon prior to reduction of the fuel sample. Furthermore, any minute air leakage into the experimental system, upon reaction with the heated graphite, yields significant quantities of CO₂.

Several measurements made during the past quarter to determine the extent of this extraneous CO₂ were performed using the following procedure or slight variation thereof:

About 10 mg of finely powdered, pure graphite, which is more than sufficient to reduce 1 mg of UO₂, was inserted in the tungsten crucible which, in turn, was installed in the experimental system. Following room temperature evacuation, the crucible was heated inductively to about 2000 °C for at least 1 hr at which time the pressure was <10⁻⁵ torr. The crucible was then returned to room temperature, the system brought to atmospheric pressure with pure helium, and the crucible plus contents were exposed to air for up to 10 min to simulate the loading of the UO₂ sample. After reassembling the system and evacuating, the crucible plus graphite were heated to about 1200 °C for 1 to 2 hr under either vacuum or a stream of pure helium. At 1200 °C, outgassing of the graphite occurs without reduction of the fuel sample. Following this outgassing period, the crucible temperature was increased to about 1700 °C and maintained at this temperature for a period sufficient to reduce 1 mg of UO₂. During the simulated fuel reduction, released gases were swept through a heated tube of CuO, where CO is oxidized to CO₂, and into a trap immersed in liquid nitrogen where the evolved CO₂ was collected for subsequent analysis.

The smallest amount of CO₂ collected, 0.18 micromole, is equivalent to Δ(O/M) = 0.056 for 1 mg of UO₂. This amount of CO₂ is considered excessive, and future work on this method of
O/M determination will concentrate on its elimination or reduction to an acceptable value. Although it would not be as convenient, it may prove necessary to eliminate any exposure of the powdered graphite to air following the initial high temperature outgassing by loading the fuel sample into the crucible in an inert atmosphere.

To improve the accuracy of the CO$_2$ analysis, the gas chromatograph employed in this investigation has been calibrated for CO$_2$ concentrations in the range from 0 to 1%. Four separate analyses at each of nine different CO$_2$ concentrations within this concentration range were averaged to construct the desired calibration curve.

REFERENCES


3.0 IRRADIATION BEHAVIOR OF FAST REACTOR FUEL MATERIALS

J. A. Christensen

IN-REACTOR THERMAL PERFORMANCE OF UO₂-PuO₂ FUEL

J. A. Christensen

Fuel-Clad Gap Conductance Measurements

L. A. Lawrence, J. A. Christensen and G. R. Horn

The high heat fluxes and large fuel-clad gaps characteristic of FTR fuel pins result in large calculated temperature drops across the fuel-clad interface. The associated decrease in allowable heat rating may be 30 to 40% of the rod powers permissible in rods with closely mating fuel and clad. Closure of the fuel-clad gap has been observed and is anticipated early in life for FTR. Object of the present study is to measure the rate of gap closure and associated change in $\Delta T$ as a basis for specifying reactor startup schedules.

Capsule FP-37, designed to study fuel-clad gap closure kinetics, was successfully irradiated for 100 hr in a Hanford K Reactor. Fuel characteristics and test conditions are listed in Table 3.1. The test variables were fuel-clad gap and fill gas. The fill gas contained various amounts of xenon to simulate effects of fission gas contamination of the initial helium bond on gap conductance. The two fuel-clad diametral gaps were 0.002 in. (the practical fabrication lower limit) and 0.011 in. The latter is chosen to maintain the gap-to-fuel diameter ratio expected in nominal FTR fuel pins.

The capsule was equipped with upstream and downstream thermocouples to measure individual rod powers. The assembled capsule was flow tested prior to irradiation to determine the coolant flow in the four channels. The flow data along with the temperature differential measurements provided a continuous heat rating record for each pin during irradiation.
<table>
<thead>
<tr>
<th>TABLE 3.1. Fuel Characteristics and Test Conditions for FP-37</th>
</tr>
</thead>
<tbody>
<tr>
<td>A. Fuel</td>
</tr>
<tr>
<td>Form</td>
</tr>
<tr>
<td>Composition</td>
</tr>
<tr>
<td>Oxygen/Metal</td>
</tr>
<tr>
<td>Density</td>
</tr>
<tr>
<td>$^{234}\text{Pu}/\text{Pu}$</td>
</tr>
<tr>
<td>$^{235}\text{U}/\text{U}$</td>
</tr>
<tr>
<td>Diameter</td>
</tr>
<tr>
<td>Cladding</td>
</tr>
<tr>
<td>Cladding OD</td>
</tr>
<tr>
<td>Impurity Content</td>
</tr>
<tr>
<td>Cold pressed and sintered pellets</td>
</tr>
<tr>
<td>UO$_2$-25 wt% PuO$_2$</td>
</tr>
<tr>
<td>1.96 ± 0.01</td>
</tr>
<tr>
<td>95% TD</td>
</tr>
<tr>
<td>87%</td>
</tr>
<tr>
<td>Normal</td>
</tr>
<tr>
<td>0.320 in.</td>
</tr>
<tr>
<td>Type 304L SS</td>
</tr>
<tr>
<td>0.458 in.</td>
</tr>
<tr>
<td>&lt;400 ppm cation impurity</td>
</tr>
<tr>
<td>B. Test Conditions</td>
</tr>
<tr>
<td>Rate of Power Increase</td>
</tr>
<tr>
<td>During Charging</td>
</tr>
<tr>
<td>60% per hr</td>
</tr>
<tr>
<td>Rate of Power Decrease</td>
</tr>
<tr>
<td>During Discharge</td>
</tr>
<tr>
<td>3% per sec</td>
</tr>
<tr>
<td>Time at Steady-State</td>
</tr>
<tr>
<td>Power</td>
</tr>
<tr>
<td>100 hr</td>
</tr>
<tr>
<td>C. Capsule</td>
</tr>
<tr>
<td>Fuel Pin Number</td>
</tr>
<tr>
<td>Fuel-Clad Gap, in.</td>
</tr>
<tr>
<td>Fill Gas(^{(a)})</td>
</tr>
<tr>
<td>FP-37-1A</td>
</tr>
<tr>
<td>0.002</td>
</tr>
<tr>
<td>He</td>
</tr>
<tr>
<td>1B</td>
</tr>
<tr>
<td>0.011</td>
</tr>
<tr>
<td>He</td>
</tr>
<tr>
<td>2A</td>
</tr>
<tr>
<td>0.002</td>
</tr>
<tr>
<td>He-41.2 Xe</td>
</tr>
<tr>
<td>2B</td>
</tr>
<tr>
<td>0.011</td>
</tr>
<tr>
<td>He-41.2 Xe</td>
</tr>
<tr>
<td>3A</td>
</tr>
<tr>
<td>0.002</td>
</tr>
<tr>
<td>He-75.6 Xe</td>
</tr>
<tr>
<td>3B</td>
</tr>
<tr>
<td>0.011</td>
</tr>
<tr>
<td>He-75.6 Xe</td>
</tr>
<tr>
<td>4A</td>
</tr>
<tr>
<td>0.002</td>
</tr>
<tr>
<td>Xe</td>
</tr>
<tr>
<td>4B</td>
</tr>
<tr>
<td>0.011</td>
</tr>
<tr>
<td>Xe</td>
</tr>
</tbody>
</table>

\(^{(a)}\) Preirradiation analysis
Following irradiation, the four fuel pins were removed from the capsule assembly and sampled for fission gas. Each of the eight fueled segments was sectioned at the fuel mid-plane and two adjacent samples removed—one for ceramography and the other for radiochemical burnup analyses based on $^{95}$Zr, $^{141}$Ce, and $^{140}$Ba-La. Fuel Pin 3A was radially sampled for the fission products $^{95}$Zr, $^{99}$Mo, and $^{141}$Ce by selectively removing small amounts of fuel (approximately 0.5 mg) with a 0.025 in. diameter high speed twist drill and vacuum sniffer. Ceramography samples were impregnated with epoxy resin and polished through a 600 grit. Macrographs and alpha and beta-gamma autoradiographs were taken of these surfaces (Figures 3.1 through 3.4).

Postirradiation structures are consistent with the increase in fuel-clad gap and xenon content of the fill gas. Pins 1B, 2B and 3B show cracking of the unrestructured fuel some of which probably occurred during the postirradiation cutting operation. All of the "B" fuel pins have an approximately cylindrical central void which increases in size with xenon content of the fill gas. Pins 3B and 4B show fuel melting to 10% and 35% of the fuel radius, respectively. Only these pins show any plutonium relocation on the alpha-autoradiographs, with a depleted band corresponding roughly to the pore free zone on the macrographs. Preliminary examination of the fuel-clad gaps indicates only a decrease in the initial 0.011 in. fuel-clad diametral gap after 100 hr or irradiation to 0.004 to 0.006 in. Precise measurements of the postirradiation fuel-clad gaps is in progress.

Calculations to determine relative values for the fuel-clad gap conductance for the eight pins by matching observed fuel structures with temperature profiles are in progress. These computations are performed using the SINTER program operational on the Battelle-Northwest Hybrid Computer.
FIGURE 3.3. Postirradiation Structures of Fuel Pins 3A and 3B
Molybdenum Distribution in Irradiated Oxide Fuels
L. A. Lawrence and J. A. Christensen

The distribution of fission products affects fuel lifetime and safety. Fission product molybdenum is of particular interest in that the oxygen balance in irradiated oxide fuels hinges upon the oxidation state of the molybdenum. The relative location of the molybdenum is thus important in considerations of the radial O/M profile in irradiated oxide fuels. In addition, estimations of the contributions of solid fission products to the swelling of oxide fuels requires a knowledge of their redistribution during irradiation. As part of the postirradiation examination of several UO$_2$-25 wt% PuO$_2$ fuel pins, radial distributions of $^{99}$Mo were measured.

Because of the relative short half-life of the only gamma active molybdenum isotope present ($^{99}$Mo, $\tau_{1/2} = 67 \text{ hr}$) the elapsed time from sample discharge to postirradiation examination must be less than 1 week. The close proximity of the special irradiation facilities at the Hanford reactors and the Radiometallurgy Laboratory permits this short elapsed time. The fuel characteristics and test conditions of the two fuel pins of interest are listed in Table 3.2.

The radial distribution of molybdenum was measured by removing from selected positions across the fuel radius small amounts of fuel using a high speed, 0.025 in. diam twist drill and a vacuum sniffer. The samples (approximately 0.5 mg) were radiochemically analyzed for $^{99}$Mo and plutonium. To normalize the molybdenum activity to a constant sample size it was assumed that no plutonium relocation occurred during irradiation. This should be a valid approximation as the alpha-autoradiographs (Figure 3.5) show no indication of any plutonium movement. The similarity of the postirradiation structures of the two pins (Figure 3.5) indicate that they operated at approximately the same heat rating, the only
TABLE 3.2. Fuel and Test Characteristics and Results

A. Preirradiation

<table>
<thead>
<tr>
<th>Form</th>
<th>Cold pressed and sintered pellets</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>UC₂-25 wt% PuO₂</td>
</tr>
<tr>
<td>Oxygen/Metal</td>
<td>1.96 ± 0.01</td>
</tr>
<tr>
<td>Density</td>
<td>95% TD</td>
</tr>
<tr>
<td>239Pu/Pu</td>
<td>87%</td>
</tr>
<tr>
<td>240Pu/Pu</td>
<td>11%</td>
</tr>
<tr>
<td>235U/U</td>
<td>Normal</td>
</tr>
<tr>
<td>Average Fuel-Clad Gap</td>
<td>0.003 in.</td>
</tr>
<tr>
<td>Cladding</td>
<td>Type 304L SS</td>
</tr>
</tbody>
</table>

B. Irradiation

<table>
<thead>
<tr>
<th>Capsule/Pin</th>
<th>Irradiation Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>FP-35 35-3A</td>
<td>10 hr</td>
</tr>
<tr>
<td>FP-37 37-3A</td>
<td>100 hr</td>
</tr>
</tbody>
</table>

| Rate of Power Increase | 60% per hr |
| Rate of Power Decrease | 3% per sec |

C. Postirradiation

<table>
<thead>
<tr>
<th>Pin/Sample</th>
<th>R/R₀₂(a)</th>
<th>Activity(b)</th>
</tr>
</thead>
<tbody>
<tr>
<td>35-3A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>20</td>
<td>0.180</td>
</tr>
<tr>
<td>2</td>
<td>26</td>
<td>0.198</td>
</tr>
<tr>
<td>3</td>
<td>50</td>
<td>0.325</td>
</tr>
<tr>
<td>4</td>
<td>67</td>
<td>0.415</td>
</tr>
<tr>
<td>5</td>
<td>87</td>
<td>0.778</td>
</tr>
<tr>
<td>37-3A</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>9</td>
<td>0.174</td>
</tr>
<tr>
<td>2</td>
<td>21</td>
<td>0.196</td>
</tr>
<tr>
<td>3</td>
<td>42</td>
<td>0.216</td>
</tr>
<tr>
<td>4</td>
<td>68</td>
<td>0.554</td>
</tr>
<tr>
<td>5</td>
<td>92</td>
<td>0.892</td>
</tr>
</tbody>
</table>

---

a. Sample width 18% of fuel radius.
b. Normalized to 1.00 at the fuel surface.
difference being the length of irradiation. Radiochemical burnup determination based on $^{95}$Zr, $^{141}$Ce, and $^{140}$Ba-La, showed that the 10-hr and 100-hr samples operated at linear power densities within 10% of each other.

The molybdenum activity of the microdrilled samples was normalized to a value of 1.00 at the fuel surface. Results (Figure 3.6) show no difference between the 10-hr and 100-hr test suggesting no significant relocation of the molybdenum in the first 100-hr of irradiation at heat ratings below incipient melting.

**Fission Product Migration in Oxide Fuels**

L. A. Lawrence and J. A. Christensen

Five identical UO$_2$-25 wt% PuO$_2$ fueled pins, irradiated for a short period of time at carefully controlled conditions, were radially sampled for the fission products $^{95}$Zr, $^{103}$Ru, and $^{141}$Ce. Fuel characteristics and test conditions are listed in Table 3.3.

Radial fission product distributions were obtained by selectively drilling polished fuel cross-sections at several preselected positions using a high speed, 0.025-in. diam twist drill and collecting the drillings with a vacuum sniffer. The microdrilled samples were radiochemically analyzed for $^{95}$Zr, $^{103}$Ru, $^{141}$Ce, and plutonium. Plutonium content was obtained from an alpha spectrum analysis. From the specific activity of the fuel and plutonium activity of the microdrilled samples, sample size was calculated.

Fission product activity was normalized to a constant sample size (Figures 3.7 through 3.11).

The resulting fission product concentration profiles show no major relocation of $^{95}$Zr and $^{141}$Ce except for a slight minimum associated with partial fuel melting in Pins 3, 4, and 5.
FIGURE 3.6. $^{99}$Mo Distribution

RELATIVE FUEL RADIUS, $r/r_0$

RELATIVE ACTIVITY, (ARBITRARY UNITS)
### TABLE 3.3. Fuel and Test Characteristics and Results

**A. Preirradiation**

<table>
<thead>
<tr>
<th>Composition</th>
<th>UO₂-25 wt% PuO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen/Metal</td>
<td>1.96 ± 0.01</td>
</tr>
<tr>
<td>Density</td>
<td>95% TD</td>
</tr>
<tr>
<td>239Pu/Pu</td>
<td>87%</td>
</tr>
<tr>
<td>235U/U</td>
<td>Normal</td>
</tr>
<tr>
<td>Diameter</td>
<td>0.320 in.</td>
</tr>
<tr>
<td>Average Fuel-Clad Gap</td>
<td>0.003 in.</td>
</tr>
<tr>
<td>Cladding</td>
<td>Type 304L SS</td>
</tr>
</tbody>
</table>

**B. Irradiation**

| Rate of Power Increase During Charging | 60% per hr |
| Rate of Power Decrease During Discharge | 3% per sec |
| Time at Steady-State Power             | 10 hr       |
| Calculated Fuel Surface Temperature at Steady-State | 950 °C |

**In-Reactor Calorimetry Data**

| Heat Rating, kW/ft
<table>
<thead>
<tr>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Pin No.</td>
</tr>
<tr>
<td>---------</td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>2</td>
</tr>
<tr>
<td>3</td>
</tr>
<tr>
<td>4</td>
</tr>
<tr>
<td>5</td>
</tr>
</tbody>
</table>

*Thermocouple failed during irradiation.*
C. Postirradiation

<table>
<thead>
<tr>
<th>Pin No.</th>
<th>Maximum Extent of Fuel Melting*</th>
<th>Minimum 103Ru Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$R_{\text{melt}}/R_0$ %</td>
<td>$R/R_0$ %</td>
</tr>
<tr>
<td>1</td>
<td>0</td>
<td>---</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>25</td>
<td>30</td>
</tr>
<tr>
<td>4</td>
<td>43</td>
<td>50</td>
</tr>
<tr>
<td>5</td>
<td>47</td>
<td>45</td>
</tr>
</tbody>
</table>

*Identified as the outer boundary, moving outward of the pore free zone.

Migration of plutonium can be seen on the alpha-autoradiographs from these sections. The autoradiographs are not quantitative, thus some estimate of plutonium relocation is necessary. Plutonium redistribution has been observed in mixed-oxide fuels operating at comparable heat ratings. The observed redistribution was on the order of 5% which is consistent with the minimum observed in the $^{95}\text{Zr}$ and $^{141}\text{Ce}$ data.

The $^{103}\text{Ru}$ concentration profiles show gross relocation. The $^{103}\text{Ru}$ concentration minimum corresponds approximately to the observed maximum extent of fuel melting. This correlation suggests that a zone refining process is responsible for the ruthenium redistribution.

### Effects of Burnup on Melting Heat Rating

C. R. Hann

The accumulation of fission products in mixed-oxide fuel decreases the thermal conductivity and melting point, thereby reducing the allowable heat rating of high burnup fuel rods.
Neg 701168-3

FIGURE 3.7. Fission Product Distribution and Postirradiation Structures of Pin 1
FIGURE 3.8. Fission Product Distribution and Postirradiation Structures of Pin 2
FIGURE 3.9. Fission Product Distribution and Postirradiation Structures of Pin 3
FIGURE 3.10. Fission Product Distribution and Postirradiation Structures of Pin 4
FIGURE 3.11. Fission Product Distribution and Postirradiation Structures of Pin 5
Comparative irradiations of fast-flux-irradiated, high-burnup fuels and their unirradiated counterparts will be used to determine the magnitude of the heat rating reduction.

High burnup (to 47,000 MWd/MTM), EBR-II-irradiated specimens will be available by the first of April. Estimated activity and gamma radiation levels of the test specimens are about 700 curies and about 600 R/hr at 3 ft, respectively. These high levels require an irradiation assembly that is compatible with hot-cell encapsulation requirements and handling techniques available at the irradiation testing facilities. Suitable closure techniques have been designed for the capsule that will contain the irradiated fuel; tests will be conducted on mockup capsules prior to encapsulation. Hazard analyses for reactor charging operations are in progress. Preliminary work indicates shielding problems will be difficult but not impossible. Efforts are continuing on methods to accommodate instrumentation leads in the shielding facilities.

The capsules will be irradiated for short times at heat ratings that result in small amounts of center melting. The effects of burnup on melting heat rating will be determined by measuring the extent of melting and compensating for changes in fissile atom and fission product inventories.

Evaluation of Alternate Irradiation Facilities
C. R. Hann and L. A. Lawrence

The thermal irradiations are scheduled for the remaining Hanford K Reactor. Alternate facilities at the National Reactor Testing Station in Idaho were evaluated in the event the existing facilities at Hanford were phased out. No serious obstacles were foreseen in transferring tests to the Engineering Test Reactor, and some test positions in the Advanced Test Reactor probably could be used with appropriate shielding. However, neither the ETR nor the ATR provides the
precise experimental conditions available in the K reactors, nor are they as convenient and economical to use.

Fast Reactor Experiments
C. R. Hann

Thermal irradiation facilities have been used in this program thus far because they permit more precise study of certain selected variables. Not only are the facilities more accessible than the existing fast test space, but thermal irradiations are simpler, less time consuming, and less expensive. One of the main goals of this program has been to identify, through the use of thermal test facilities, critical experiments for EBR-II. Such tests now being planned include:

- Fuel swelling measurements.
- Measurement of fission gas release rate during irradiation.
- A study of the effects of power cycles on fuel-cladding mechanical interactions.

These experiments are being scoped for preliminary discussions with the ANL EBR-II Project staff prior to submitting the Approval-in-Principle documentation.

FUEL-TO-CLADDING GAP CONDUCTANCE ANALYSES
G. R. Horn

Fast reactor fuel pins operate with twice the surface heat flux characteristic of thermal reactor fuel rods. A commensurately more precise knowledge of the heat transfer coefficient at the fuel-to-clad interface is required for fuel temperature calculations. Several investigators have attempted to measure fuel-clad gap conductance but, as will be shown, the results are not sufficiently precise.

Gap conductance data published by Bain, Duncan, Baily, et al., and Craig, et al. were analyzed for this
study and are summarized in Figure 3.12. Conditions represented in each experiment are summarized in Table 3.4. Figure 3.12 shows that a correlation between conductance and initial diametral gap does exist. A least squares fit of the data yields the functional relationship:

\[ h_g = 1873e^{-67.6G} \] (1)

where

- \( h_g \) = gap conductivity, Btu/hr-ft\(^2\)-°F
- \( G \) = cold diametral gap, in.

The imprecision of this relationship is so great (as indicated by the 95% confidence band on Figure 3.12) as to render it virtually useless. For example, for a fuel pin having a cold diametral gap of 0.006 in., Equation (1) predicts (with 95% confidence) that the gap conductivity will be between 540 and 2850 Btu/hr-ft\(^2\)-°F. As will be shown, this range is too broad for design purposes.

Attempts to develop more sophisticated correlations involving other parameters such as heat rating, fuel diameter, fill gas thermal conductivity, etc., have so far provided no better correlation than the one shown in Figure 3.12. Results of some individual experimenters have shown, however, that improved correlations should exist. For example, Bain(2) showed that, in fuel pins with constant cold fuel-to-clad gaps, gap conductance increased with increasing heat rating. Bain also showed that gap conductance in helium-filled pins was higher than that for similar, argon-filled pins. The latter conclusions was confirmed by Duncan, but Craig et al. concluded that fill gas had no significant effect on the heat transfer coefficient.

The large uncertainties that exist in correlating fuel-to-clad heat transfer coefficients with fuel pin fabrication
FIGURE 3.12. Gap Conductivity as a Function of Cold Diametral Gap (From Data of Bain\textsuperscript{2}, Duncan\textsuperscript{3}, Baily, et al.\textsuperscript{4} and Craig et al.\textsuperscript{5})
TABLE 3.4. Summary of Conditions Embodied in Measurements of Gap Conductivity

<table>
<thead>
<tr>
<th>Experimenter</th>
<th>Cold Diametral Gap, in.</th>
<th>Fuel Diameter, in.</th>
<th>$\int^T_{t_s} k dT$, W/cm</th>
<th>Fill Gas</th>
<th>Measured Gap Cond.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bain(2)</td>
<td>0.003 to 0.0331</td>
<td>0.660 to 0.740</td>
<td>38 to 89</td>
<td>Argon Mostly, Some W/He</td>
<td>116 to 2460</td>
</tr>
<tr>
<td>Duncan(3)</td>
<td>0.002 to 0.012</td>
<td>0.334 to 0.432</td>
<td>40 to 54</td>
<td>Helium</td>
<td>635 to 5100</td>
</tr>
<tr>
<td>Baily et al.(4)</td>
<td>0.002 to 0.010</td>
<td>0.218</td>
<td>40.5</td>
<td>Argon</td>
<td>600 to 2000</td>
</tr>
<tr>
<td>Craig et al.(5)</td>
<td>0.0008 to 0.0101</td>
<td>0.218</td>
<td>32 to 48</td>
<td>Argon or Helium</td>
<td>520 to 3000</td>
</tr>
</tbody>
</table>

or operating parameters result primarily from uncertainties associated with individual gap conductance measurements. Although none of the investigators have reported error limits for their data, it must be realized that the experimental error in all of the measurements is very large. Craig et al.(5) for example, reported a gap conductance of about 1000 Btu/hr-ft$^2$-°F for a fuel pin having an initial cold diametral gap of 0.0041 in. If one assumes that power generation was known to ±5%; coolant temperature was known to ±5 °F; material properties and conditions of heat transfer between coolant and cladding were known to ±5%; fuel thermal conductivity was known exactly; the temperature at which columnar grains form was known to ±50 °C; and dimensions of structural features observed during postirradiation examination were known to ±0.001 in.; the calculated gap conductance varies from 408 to 2935 Btu/hr-ft$^2$-°F. This magnitude of uncertainty is typical for all of the reported gap conductivity values.

The importance of knowing precise values for gap conductivity is illustrated by Figure 3.13. This figure was constructed for a hypothetical fuel pin having the following characteristics:
It was assumed that the pin was cooled with 750 °F sodium and that thermal conductivity of fuel was that published by Baily et al. It was further specified that fuel melting defined the maximum allowable heat rating. With these assumptions, Figure 3.13 shows how allowable heat rating (i.e., the heat rating which would just cause melting) varies with changing gap conductance. Obviously, if gap conductances in fast reactor fuels are high (above ~3000 Btu/hr-ft²-°F), the precise knowledge is not important to fuel pin design (increasing gap conductivity from 3000 to 4000 increases allowable heat rating by less than 2%). Alternatively, if gap conductances are characteristically low (below ~1600 Btu/hr-ft²-°F), precise knowledge of conductances is important. As an illustration, suppose a conductance of 600 is used for design when the value actually is 1600. The pin would be limited to a heat rating of ~11.5 kW/ft instead of the 14.7 kW/ft actually allowable. In this case, the heat rating could be increased 30% if gap conductance was more precisely known.

These results illustrate the importance of continuing to collect gap conductance data, particularly those data obtained by more accurate experimental methods than ordinarily employed. Search for a data correlation which reduces the error bands in Figure 3.12 is continuing.
IRRADIATION OF HYPOSTOICHIOMETRIC UO$_2$-PuO$_2$ FUEL

J. E. Hanson

High Burnup Thermal Flux Irradiations


This task includes irradiation testing of mixed-oxide fuel specimens of different stoichiometrics to high burnups in a thermal flux. Substoichiometric mixed-oxides are expected to exhibit better fuel cladding-sodium compatibility at high burnups than the nearly stoichiometric oxides currently specified for the FTR. System compatibility, swelling, and gas release characteristics of UO$_2$-PuO$_2$ fuel as a function of stoichiometry and fuel form must be determined at high burnup before a maximum performance fuel can be designed.

Status of Thermal Flux Irradiation Tests

The general status of the fuel pins being examined is summarized in Table 3.5. The reported burnup values were derived from $^{148}$Nd analyses of sections removed from the center of the fuel pins. The burnup values for those pins not yet examined were estimated from the reported fluence, assuming a linear relationship between fluence and burnup. BNW 1-10 and 1-12 were discharged on November 9 at the end of ETR Cycle 104. These two capsules have been gamma scanned at the ETR. When the MTR reaches approximately 12 MW, expected early in February, they will be neutron radiographed. A decision will be made then whether to return the capsules to the ETR for further irradiation or terminate the irradiation and start examinations.
### TABLE 3.5. Summary Status of Irradiation Experiments

<table>
<thead>
<tr>
<th>Test Designation</th>
<th>Description (a)</th>
<th>Fuel Form</th>
<th>O/M</th>
<th>Facility</th>
<th>Goal Exposure, MWd/MTM</th>
<th>Actual Exposure, MWd/MTM</th>
<th>Discharge Date</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>BNW 1-1</td>
<td>Solid</td>
<td>Solid</td>
<td>1.93</td>
<td>MTR</td>
<td>100,000</td>
<td>(Est)120,000</td>
<td>2-69</td>
<td>Being Exam. at BNW</td>
</tr>
<tr>
<td>BNW 1-2</td>
<td>Solid</td>
<td>Solid</td>
<td>1.93</td>
<td>ETR</td>
<td>50,000</td>
<td>(Est) 70,000</td>
<td>2-69</td>
<td>Being Exam. at BNW</td>
</tr>
<tr>
<td>BNW 1-3</td>
<td>Solid</td>
<td>Solid</td>
<td>1.96</td>
<td>MTR</td>
<td>100,000</td>
<td>(Est)150,000</td>
<td>2-69</td>
<td>Being Exam. at BNW</td>
</tr>
<tr>
<td>BNW 1-4</td>
<td>Solid</td>
<td>Solid</td>
<td>1.96</td>
<td>MTR</td>
<td>50,000</td>
<td>71,000</td>
<td>6-68</td>
<td>Exam. Completed at BNW</td>
</tr>
<tr>
<td>BNW 1-5</td>
<td>Solid</td>
<td>Solid</td>
<td>2.00</td>
<td>MTR</td>
<td>100,000</td>
<td>(Est)160,000</td>
<td>2-69</td>
<td>Being Exam. at BNW</td>
</tr>
<tr>
<td>BNW 1-6</td>
<td>Solid</td>
<td>Annular</td>
<td>2.00</td>
<td>MTR</td>
<td>50,000</td>
<td>75,200</td>
<td>10-68</td>
<td>Exam. Completed at BNW</td>
</tr>
<tr>
<td>BNW 1-7</td>
<td>Annular</td>
<td>Annular</td>
<td>1.93</td>
<td>MTR</td>
<td>50,000</td>
<td>&lt;1,000</td>
<td></td>
<td>Lead Tube Failed. Exam at BNW</td>
</tr>
<tr>
<td>BNW 1-8</td>
<td>Annular</td>
<td>Solid</td>
<td>1.93</td>
<td>MTR</td>
<td>100,000</td>
<td>29,000</td>
<td>1-68</td>
<td>Failed. Exam. at BNW</td>
</tr>
<tr>
<td>BNW 1-9</td>
<td>Annular</td>
<td>Solid</td>
<td>1.96</td>
<td>MTR</td>
<td>50,000</td>
<td>81,800</td>
<td>6-68</td>
<td>Exam. Completed at LASL</td>
</tr>
<tr>
<td>BNW 1-10</td>
<td>Annular</td>
<td>Solid</td>
<td>1.96</td>
<td>ETR</td>
<td>100,000</td>
<td>(Est)92,200</td>
<td>(Est)2-70</td>
<td>(b)</td>
</tr>
<tr>
<td>BNW 1-11</td>
<td>Annular</td>
<td>Annular</td>
<td>2.00</td>
<td>MTR</td>
<td>50,000</td>
<td>87,400</td>
<td>8-68</td>
<td>Exam. Completed at LASL</td>
</tr>
<tr>
<td>BNW 1-12</td>
<td>Annular</td>
<td>Annular</td>
<td>2.00</td>
<td>ETR</td>
<td>100,000</td>
<td>(Est)96,500</td>
<td>(Est)2-70</td>
<td>(b)</td>
</tr>
</tbody>
</table>

#### a. Fuel
- 25% PuO₂ - 75% UO₂
- Pellets 0.212 in. OD (0.052 in. ID for annular pellets)
- Smeared density - 87.5% TD
- Fuel column length 13.5 in.

Postirradiation Examination of BNW 1-1, 1-2, 1-3, 1-5

Metallographic Examination of BNW 1-1 and 1-2. Fuel pins BNW 1-1 and 1-2 were sectioned for metallographic and burnup samples. Specimens for cladding burst tests were also obtained. The burnup samples have been submitted for chemical analysis. Rough grinding is in progress on all of the longitudinal and transverse metallography samples. All metallographic preparation is being done using nonaqueous lubricants which react minimally with the fuel and fuel-cladding interface. Between grinding steps, the metallographic specimens are stored in an evacuated system. Each burst test and spare fuel pin section is stored in individual aluminum capsules marked with sample identification and orientation. These capsules are in turn stored in other capsules under approximately 25 psig, moisture-free argon.

Preparations are being made to section BNW 1-3 and 1-5 for metallographic examination. Grinding through 600 grit, macrophotography, and $\alpha$ and $\beta$-$\gamma$ autoradiography have just been completed on samples 1-2A (longitudinal bottom of pin) and 1-2D1 and 1-2F (transverse and longitudinal respectively from the middle of the fuel column).

Sample 1-2A has a fission product ingot located in the central void approximately 3/8 in. from the original bottom of the fuel column. The ingot is located at the lower end of a mass of fuel that appears to have melted and moved down the central void. This ingot has been identified from beam analysis of gamma ray spectra data as primarily niobium. The $\beta$-$\gamma$ autoradiographs show penetration of fission products into cracks in the lower insulator pellets to a depth of about 0.2 in. The $\alpha$ and $\beta$-$\gamma$ autoradiographs reveal no evidence of extensive reaction of the fuel pellets and insulator pellets.
Sample 1-2F also shows a large ingot approximately 0.25 in. long filling the central void. Analysis of the gamma ray spectra data showed this ingot to be comprised primarily of ruthenium.

Preliminary measurements of void and columnar grain region diameters on sample 1-2D1 showed 0.075 and 0.175 in., respectively, for the 0.220 in. diameter pellet. There are many small metallic ingots in the columnar grains, as well as a concentration of these inclusions at the columnar grain terminus. Examination of these three metallography samples will continue to establish the conditions of the fuel and the movements of the fission products.

**Shielded Electron Microprobe Examination**

E. D. Jenson

Fuel pin BNW 1-6, Section E, has been partially examined in the shielded electron microprobe. This section was 6.8 in. from the lower end cap and had an average burnup of 75,200 MWD/MTM (38,000 MWD/MTM at centerline and >200,000 MWD per MTM at the surface) at an average power of 16.3 kW/ft. The fuel surface temperature was 1100 °C and the centerline temperature 2700 °C (calculated). It had cooled 12 months before microprobe examination. Figure 3.14 shows the surface examined with indications of areas and points examined.

Uranium and plutonium are known to undergo a redistribution during high temperature irradiations, with plutonium concentration increasing near the central void. Quantitative data on the concentration changes that occur are necessary for increased understanding of the processes involved. The concentration of uranium and plutonium was determined along four radii, as shown in Figure 3.14. Path R1 was analyzed using a 5 μ diameter beam at 10 μ intervals. The results, corrected for background and specimen current drift, are shown in
Figure 3.15. The concentration values are relative, being uncorrected for absorption, fluorescence, backscatter, and detector dead time. The net effect of these corrections is expected to be small. In the horizontal portion of the plots, an observed standard deviation of ±1.2% is applicable to the data. The sharp drops in both uranium and plutonium concentration are due to pores, voids, cracks, and fission product ingots. The largest dips in the curves are voids or cracks. Smaller dips may be either voids or fission product inclusions. To identify which, a simultaneous analysis for ruthenium (the most abundant element in the ingots) must be run. Both plots show depletion of uranium and plutonium in the outer fuel areas, with a decrease of uranium near the central void and a simultaneous increase of plutonium near the central void.

The columnar grain region of this section shows numerous metallic inclusions. Their composition (uncorrected) averages around 40% ruthenium, with under 10% molybdenum. Ruthenium, rhodium, technetium, molybdenum, and a very small amount of either palladium or silver were identified (in order of decreasing concentration). Large enough samples were analyzed to completely eliminate the beam penetration. No uranium or plutonium lines were excited, showing that these elements are not present in the inclusions. A plot of ruthenium and molybdenum concentrations in the inclusions against radial fraction is given in Figure 3.16. The inclusion nearest the unstructured fuel, but still in the columnar grains, shows only half as much ruthenium as inclusions further into the columnar grains. The five inclusions examined in the unstructured area showed no ruthenium, but did have 15 to 30% molybdenum. Four of these five inclusions did not appear as lustrous particles on the mosaic, but were found associated with voids or short cracks. This indicates they may be underneath the fuel surface or they may be a different physical form, such as an oxide.
The results of analysis for molybdenum in the fuel matrix are shown in Figure 3.17. Molybdenum concentrations in the fuel matrix of 1 to 4% are observed in the unrestructured fuel, dropping to 0 to 1% in the columnar grains. Ruthenium analysis across a radius indicated no measurable ruthenium (under 0.15%). However, up to 0.3% ruthenium was found in pores in the unrestructured material.

Xenon was detected and its relative distribution across line R3 determined. The xenon counts (in 100 seconds) in the outer 700 μ is shown as part of Figure 3.18. Between 700 μ and the central void, the xenon count was zero. Since a xenon standard is rather difficult to examine under vacuum, counts per 100 seconds are plotted in place of percent in Figure 3.18. The outer two maxima observed are within the unrestructured fuel while the broad, but lower maximum appears to be just outside the unrestructured region. The drop off in counting rate out to 650 μ from the fuel OD corresponds to the fuel region showing numerous pores or voids. Within the area of the columnar grains showing well developed lenticular voids, the xenon count rate is essentially zero. Counting times of 100 seconds for each point, and also the background, with 0.23 microamp specimen current were used. A statistical counting error of about 150 counts is applicable to this data (standard deviation of 75 counts).

Analysis of neodymium, zirconium, and cerium across the radius (R3) was also completed. The distribution of these elements is also shown in Figure 3.18. Each of these elements shows highest concentration in the unrestructured fuel area where the burnup was highest. The concentration drops to a minimum about half-way across the fuel, then increases near the central void and approaches the count rate observed in the unrestructured fuel, with a few isolated points exceeding the unrestructured area. The form of these plots near the central
FIGURE 3.18. Neodymium, Cerium, Zirconium, and Xenon Distribution Across Fuel Pin BNW 1-6, Section E. The xenon count rate beyond 800 μ was zero.
voids appears similar to the plutonium distribution in Figure 3.15. The distance coordinates do not appear to be the same, but after allowing for the increased path length of R3 due to not passing through the center (see Figure 3.14), the increase of plutonium, neodymium, and zirconium begin at nearly the same radial fraction, and cerium begins just before plutonium increases. Considering the interval of analysis and the variation of the plot, all four components probably increase in the same area. The increase near the central void for each element, compared to the area half-way across the radius, is given in Table 3.6.

<table>
<thead>
<tr>
<th>Element</th>
<th>% Increase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plutonium</td>
<td>~40</td>
</tr>
<tr>
<td>Neodymium</td>
<td>~40</td>
</tr>
<tr>
<td>Zirconium</td>
<td>~30</td>
</tr>
<tr>
<td>Cerium</td>
<td>~50</td>
</tr>
</tbody>
</table>

The correlation of position and concentration increase suggests that the increased fission products near the central void are simply due to fission of the increased plutonium at that location rather than transport of fission products. From the calculated burnup at the outer edge and center of the fuel (>200,000 and 38,000 MWd/MTM, respectively), one would expect a ratio of an indicator fission product at these locations of >5:1. The observed ratio of ~1:1 has not yet been explained.

At least 20 fission product elements have been identified in this pin. Table 3.7 lists the elements and the observed count rate (in the outer fuel area using a specimen current of approximately 10 microamps).
<table>
<thead>
<tr>
<th>Element</th>
<th>Count Rate for La₁ Line* (Approximately Proportional to Concentration)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strontium</td>
<td>200</td>
</tr>
<tr>
<td>Yttrium</td>
<td>100</td>
</tr>
<tr>
<td>Zirconium</td>
<td>1400</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>3000</td>
</tr>
<tr>
<td>Technetium</td>
<td>600</td>
</tr>
<tr>
<td>Ruthenium</td>
<td>1000</td>
</tr>
<tr>
<td>Rhodium</td>
<td>300</td>
</tr>
<tr>
<td>Palladium</td>
<td>1000</td>
</tr>
<tr>
<td>Tellurium</td>
<td>100</td>
</tr>
<tr>
<td>Iodine</td>
<td>(a)</td>
</tr>
<tr>
<td>Xenon</td>
<td>(b)</td>
</tr>
<tr>
<td>Cesium</td>
<td>3000</td>
</tr>
<tr>
<td>Barium</td>
<td>1500</td>
</tr>
<tr>
<td>Lanthanum</td>
<td>1100</td>
</tr>
<tr>
<td>Lanthanium</td>
<td>3400</td>
</tr>
<tr>
<td>Cerium</td>
<td>2000</td>
</tr>
<tr>
<td>Praseodymium</td>
<td>4500</td>
</tr>
<tr>
<td>Neodymium</td>
<td>(b)</td>
</tr>
<tr>
<td>Promethium</td>
<td>1500</td>
</tr>
<tr>
<td>Samarium</td>
<td>(b)</td>
</tr>
<tr>
<td>Europium</td>
<td>(b)</td>
</tr>
<tr>
<td>Gadolinium</td>
<td>(b)</td>
</tr>
</tbody>
</table>

a. Calculated from Lβ₁ or other lines for lines interfering with the La₁ line. Counting rates above and below iodine are not comparable; different detectors were used in these regions.

b. These elements were identified using point-by-point counting with high specimen current and long counting times (up to 100 seconds).
A pore near the cladding showed a lining of iron and nickel, with the interior of the pore nearest the cladding containing barium and cerium, as illustrated in Figure 3.19. The iron and nickel in the lining of the pore show the pattern of maximum iron concentration, then maximum nickel concentration with distance from the cladding. The maximum concentration of both iron and nickel observed was about 30%. Barium and cerium in the pore show some separation in the direction perpendicular to the pin radius. Barium shows a much higher concentration on one side of the pore than on the other, while the cerium concentration is highest on the side with low barium, as shown in Figure 3.20. Along the centerline of the pore (parallel to the pin radius) barium and cerium concentrations increase in an approximately parallel manner toward the center of the pin. The temperature in the area of the pore was about 1200 °C (calculated). The barium-cerium deposits do not show a metallic luster, and previous samples have shown an indication they are nonmetallic. The barium and cerium fission products should be present in the same ratio whenever they appear, unless one deposit is older than the other. The different ratios of barium to cerium may indicate deposition on one side of the pore early during irradiation, then deposition on the other side during the remainder of the irradiation, allowing that first deposited to decay and change the relative amounts of barium and cerium. Vapor phase transport is not likely since the analysis was made along what should be an isothermal line, i.e., along an arc approximately centered at the pin center.

An iron-nickel streamer extending from the cladding into the fuel is shown in Figure 3.21. The specimen current display shows a gray area indicating something unusual. The iron and nickel deposits were found bordering this area. The gray area contained the following elements in order of the X-ray
FIGURE 3.19. Specimen Current, Iron, Nickel, Barium, and Cerium Distribution in a Void near the Fuel-Cladding Interface of BNW 1-6, Section F. Isolated Dots and Lines on Iron and Cerium Displays Are Due to Background Noise. Dark Area in Lower Left-Hand Corner of 300X Specimen Current Display Is the Cladding (1000X Is 80 × 80 Microns)
FIGURE 3.20. Barium and Cerium Counting Rates (Proportional to Composition) Across a Deposit of Gray Phase in BNW 1-6, Section E.
FIGURE 3.21. Specimen Current, Iron, Nickel, and Cesium Distribution on a Streamer Extending from the Cladding into the Fuel of BNW 1-6, Section E (80 x 80 Microns)
La or Ka line intensity: iron, nickel, palladium, tellurium, cadmium, molybdenum, barium, silver, cesium, silicon, and possibly potassium.

Chromium migration from the cladding onto the fuel surface was observed in this section. Figure 3.22 shows the separation of chromium from the cladding and deposition on the fuel. No iron or nickel was found associated with the chromium removed from the cladding. Many examples of iron and nickel migration from the cladding into the fuel have been seen, but very little chromium has been associated with the iron and nickel. The most likely explanation for the difference in migration behavior of these elements appears to be the large difference in the vapor pressure of their iodides. Fission product iodine reacting with the cladding forms iron, nickel, and chromium iodides, which then diffuse through the gas phase down the gradient of chemical potential of the metal iodide into the fuel, and then decompose in the hotter regions. Chromium iodide does not migrate very far since its vapor pressure is about a factor of 300 less than iron and nickel, giving a smaller gradient of chemical potential along the path into the fuel. From the above model of iodide migration, one would expect chromium to migrate only a very short distance (compared to iron and nickel) due to the variations in the gradient of chemical potential with distance. This deposit of chromium lends support to the above mechanism of transport of cladding components into the fuel.

The cladding of this section showed bright and dark inclusions. The bright inclusions were fuel, probably deposited during the polishing operation. The dark inclusions were found to be rich in manganese. Figure 3.23 is a concentration profile across one such inclusion, showing iron, nickel, chromium, and manganese. Small amounts of molybdenum, titanium, copper, and vanadium were also identified.
FIGURE 3.22. Specimen Current, Iron, Nickel, and Chromium Distribution in a Void near the Fuel-Cladding Interface of BNW 1-6, Section E. Arrow in Iron Display Indicates Traverse in Figure 3.23
(260 x 260 Microns)
FIGURE 3.23. Concentration of Iron, Manganese, Nickel, and Chromium Across a High Manganese Inclusion in the Cladding of BNW 1-6, Section E. Fuel-Cladding Interface is at ~125μ.
Gamma Scanning of Irradiated Pins BNW 1-10 and 1-12

Gross gamma scan traces of BNW 1-10 and 1-12 each show several peaks indicating concentrations of gamma emitting isotopes. It is interesting to compare these gamma scans made at an estimated 92,200 and 96,500 MWD/MTM for BNW 1-10 and 1-12, respectively, with the gamma scans made on these two capsules during an interim examination in March 1969 at approximately 55,000 MWD/MTM (estimate) burnup for both capsules. At the lower exposure BNW 1-10 showed a high narrow peak at 1-3/4 in. from the top of the fuel. This peak has disappeared completely on the scans at 92,200 MWD/MTM (estimate). A number of smaller peaks shown on 1 to 10 at the lower exposure also do not show up at the proper axial positions on the scans at higher exposure. Three peaks at 0 to 1.5 in. from the bottom of the fuel can be seen at corresponding positions on both gamma scans. Examination of neutron radiographs made at 55,000 MWD/MTM (estimate) on 1 to 10 showed no observable concentration of materials in the central void of the fuel.

A large peak is located at the bottom of the fuel column on both gamma scans of BNW 1-12. At 96,500 MWD/MTM (estimate) the peak is broader than is found on the scan at 55,000 MWD/MTM (estimate). Three other broad activity peaks at 5, 6.5, and 8.5 in. from the bottom of the fuel correlate as to axial position on both gamma scans. There are two activity peaks at the top of the fuel at the higher exposure that do not show on the scan at the lower exposure.

The disappearance of some peaks found on gamma scans of BNW 1-10 at 55,000 MWD/MTM (estimate) and the appearance of other peaks on the gamma scan at 92,000 MWD/MTM (estimate) and the broadening of peaks on BNW 1-12 shows that significant migrations of fission products has occurred in the fuel. Detailed gamma spectra data collected at several axial positions are needed before these capsules are returned to the ETR.
for further irradiation to establish which fission products are migrating and where they migrate.

Analysis of LASL Profilometer Data

To obtain an estimate of the error on diameter measurements made by LASL of irradiated BNW fuel pins, an analysis was made on data from a special profilometer run conducted by LASL at the request of BNW. The measurements were made on a profilometer standard having five steps.

<table>
<thead>
<tr>
<th>Step</th>
<th>Diameter Range, in.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.3019 - 0.3020</td>
</tr>
<tr>
<td>2</td>
<td>0.2996 - 0.2998</td>
</tr>
<tr>
<td>3</td>
<td>0.3050 - 0.3051</td>
</tr>
<tr>
<td>4</td>
<td>0.3022 - 0.3023</td>
</tr>
<tr>
<td>5</td>
<td>0.3022 - 0.3023</td>
</tr>
</tbody>
</table>

The diameter range for each step was determined and certified as traceable to NBS standards by a measurement and inspection group at LASL. The following measuring procedure was used. The standard was positioned in the profilometer instrument where it remained for the duration of the measurements. The two profilometer linear transducer styluses at 90° to one another were positioned on the bottom step, and the two recording instruments were brought to zero. Ten profiles were obtained for both upward and downward travel of each stylus, with the instruments being brought to zero each time on the bottom step. This is the LASL standard operating procedure. Two diameter measurements are then obtained simultaneously making a total of 40 profiles for each step on the standard.

This data was analyzed statistically by T. M. Beetle of the BNW Mathematics Department to determine if significant variations occur in diameter measurements for:

- Runs made up or down.
Different transducers and recorders.

- Different steps on the standard.
- Different measurement runs or profiles.
- Determination of random variation for all of the measurements.

Although there were statistically significant variations for these parameters the maximum was 0.00003 in. The variation in the measurements due to random error was determined to be about 0.00006 in. Thus each diameter selected from a LASL profilometer trace has a standard deviation of 0.00006 in. assuming that the profilometer has been calibrated with an accurately known standard. The error for any fuel pin diameter at the 95% confidence level is ±2 standard deviations or ±0.00012 in.

Modifications to BNW 324 East Cell Profilometer

The remote profilometer for diameter measurements of irradiated fuel pins is being modified. All wiring for the electric motor controls and drive for vertical movement and rotation of the fuel pins has been completed. Startup and testing of the drive has been curtailed until a misaligned bearing on the pin rotation chuck can be replaced. Two new matched linear displacement transducers have been obtained. These are expected to improve the capability of the electronic measuring system to measure diameters to greater than ±0.0001 in. accuracy.

Two diameter standards for use in calibration of the profilometer were received. These standards were machined to give diameters of 0.245, 0.250, 0.255, 0.233, 0.238 and 0.243 in., accurate to ±0.00002 in. They have been certified by the BNW Secondary Standards Laboratory with secondary standards traceable to the National Bureau of Standards.
Work is in progress to develop the quantitative metallographic techniques required to characterize the structure of both unirradiated and irradiated mixed oxide fuel materials. These structural characterizations are formulated for use in interpreting the effects of irradiation in terms of existing calculational models for the restructuring, bubble migration, and the swelling of mixed-oxide materials. The present studies are directed toward evaluating the swelling mechanisms associated with bubble formation within the grains, the subsequent migration of the bubbles to the grain boundary, and the growth of the grain boundary bubbles.

Detailed scanning electron microscope examination of fast reactor fuel materials in cooperation with the FFTF Fuels Evaluation and Special Products Fabrication Sections has continued with the examination of a second replica from an irradiated fast reactor fuel pin (PNL 1-17) and direct examination of an unirradiated mixed-oxide fuel pellet (MEE-63-4). In addition, specifications have been written for the preparation of specimens from four irradiated fuel pins, PNL 1-19, PNL 2-19, PNL 59-5, and PNL 59-7. These specimens are being prepared under closely controlled conditions for comparison of microstructure in fuels irradiated in a fast neutron flux with similar specimens irradiated in a thermal neutron flux.

A second replica from an irradiated fuel pin (PNL 1-17), prepared by LASL for routine metallographic examination, was examined and micrographs taken at preselected locations. These included ten positions along one radius at 0.25 mm intervals.
and also at the cladding edge, midradius, and center axis along additional radii. As in the first replica studied previously, the microstructure and porosity appear consistent on each axis. However, comparison of grain size and porosity distribution show marked differences between the first replica (PNL 1-17-E) from the high flux region at the center of the fuel pin. This series is being evaluated by the FFTF Fuels Evaluation Section. Grain growth and accumulation of larger pores in the grain boundaries with depletion in the adjacent zone, and clusters of smaller pores in the grain interior (Figure 3.24) is evident. These observations support the relevance of the grain boundary swelling model selected for evaluation by this task.

Since primary data will be provided by the micrographs obtained from direct examination of plastic replicas in the scanning electron microscope, carefully controlled and documented preparation of samples is necessary to ensure accurate quantitative evaluation and comparison of void sizes and distribution in specimens irradiated under different conditions. Specimens irradiated to similar burnup levels in both a fast and a thermal neutron flux are available as shown in Table 3.8.

<table>
<thead>
<tr>
<th>Capsule</th>
<th>Burnup</th>
<th>Linear Power Rating</th>
<th>Reactor</th>
</tr>
</thead>
<tbody>
<tr>
<td>PNL 1-19</td>
<td>8,700 MWD/MTM</td>
<td>9 kW/ft</td>
<td>EBR-II</td>
</tr>
<tr>
<td>PNL 2-19</td>
<td>47,000 MWD/MTM</td>
<td>9 kW/ft</td>
<td>EBR-II</td>
</tr>
<tr>
<td>PNL 59-5</td>
<td>9,500 MWD/MTM</td>
<td>10 kW/ft</td>
<td>GETR</td>
</tr>
<tr>
<td>PNL 59-7</td>
<td>48,000 MWD/MTM</td>
<td>10 kW/ft</td>
<td>GETR</td>
</tr>
</tbody>
</table>

These specimens are being prepared in the PNL Radiometallurgy laboratory. Close control, frequent visual inspections, and documentation of all preparation procedures will provide a guide for subsequent examinations of irradiated fuel specimens.

Neg 3416  1500X

FIGURE 3.24. Micrograph of Area Midway Between Cladding Edge and Center Axis of Pellet from Irradiated Fuel Pin (PNL 1-17-E). Negative PVC Plastic Replica Has Been Coated with an Evaporated Gold Film
The reference materials from which these fuel pins were fabricated, MEE-12 and ME-22, is also being characterized. A review of the standard characterization techniques is being made to determine if specialized characterization techniques will be required for the model evaluation studies to be conducted by this task.

An unirradiated mixed oxide fuel pellet (MEE-63-4) has been examined for the Special Products Fabrication Section by both electron microprobe and scanning electron microscopy. Initial examination in the microprobe included traces along three axes and micrographs taken at ten locations along each axis. Since the magnification limit (1000X) and the resolution of the microprobe are insufficient for identification and characterization of submicron pores, examination was continued in the scanning electron microscope. There was close agreement between micrographs from both instruments but the higher magnification and better resolution of the SEM still showed no submicron pores, indicating either their absence or the need for etching to reveal the fine surface structure. Comparison of scanning electron micrographs taken in the same area before and after etching in dilute nitric acid-cerium nitrate solution, shows many submicron pores and details of the fine surface structure (Figures 3.25b and c). Figure 3.25 also shows the comparison between micrographs made in each instrument (Figure 3.25a). The effect of different conducting thin films evaporated on the specimen surface is also being evaluated to determine if the use of gold or carbon contributes to the differences in resolution.

The characterizations carried out on this project are providing the background for the specialized developments required for future quantitative evaluations of microstructural features. The photomicrographs from these studies are the observational input for developing new quantitative
FIGURE 3.25. Micrographs of the Same Area in Specimen MEE-63-4, As-Polished (a. Microprobe; b. SEM) and After Etching (c. SEM). The As-Polished Specimen Was Vapor Deposited with Carbon. The Etched Sample Was Gold Coated
characterizations of microstructure and provide the bases for establishing the limitations for making such quantitative characterizations. It has been demonstrated that the continuous magnification ranges required for statistical analysis of pore size and distribution can be readily achieved by sequential examinations using optical metallography, electron microprobe, and scanning electron microscope techniques. Similar examinations of process samples, using replica methods when necessary, will provide additional data for both the preirradiation porosity characterization and fuel swelling and analytical modeling programs, and will provide a means for comparing unirradiated and irradiated fuel materials.

An idealization of one portion of the swelling model used in the EXPAND computer code has been made for subsequent quantitative statistical evaluation. This idealization assumed that the intragranular bubbles are nucleated on dislocations with a uniform spacial distribution and that they obtain a steady-state size distribution during the early stages of irradiation. Attainment of this steady-state size distribution should be possible through adaption of the techniques used by Baroody(7) or Gruber.(8) Bubbles leave their nucleation site when they reach the critical size and breakaway from the dislocations. This critical size is a function of the temperature and the temperature gradient. Since the size distribution is assumed to be stationary and bubbles break away at a critical size, the rate of bubble break-away for migration to the grain boundaries depends upon the gas generation rate. The bubbles that migrate to the grain boundary are assumed to be immobile once they reach the grain boundary. No coalescence of bubbles during the migration is assumed, but coalescence of the bubbles as they reach the grain boundary occurs if the centers of the bubble are within a fixed region of influence.
The uniform spatial distribution of pinning sites, fixed size for bubble break-away, and stationary size distribution of intragranular bubbles results in a flux of bubbles migrating to the grain boundary. The arrival of the bubbles at the grain boundary can be described by a Poisson process. The total number of bubbles arriving per unit area of grain boundary is directly proportional to the total gas generated for the projected volume of the grain and is also proportional to the direction cosine between the temperature gradient vector and the normal to the grain boundary surface. For the proposed model of grain boundary bubble growth by only coalescence, all the bubbles are visualized as striking the grain boundary. The probabilities of no coalescence, of one or more bubbles coalescing, of two or more bubbles coalescing, etc., are determined by considering the probability distribution of nearest neighbors, next nearest neighbors, etc. Let:

- $m$ bubbles arrive per unit area.
- $R_1$ be the radius of interaction for two bubble centers.
- $R_2$ be the radius of interaction for three bubble centers if two bubbles have interacted.
- $N$ be the number of bubbles interacting.
- $P\{\cdot\}$ be the probability of interactions associated with any randomly selected bubble.

For the assumed interaction model, the resultant probabilities of the selected bubble interacting with additional bubbles are:

- $P\{N = 0\} = \exp (-\pi m R_1^2)$,
- $P\{N \geq 1\} = 1 - \exp (-\pi m R_1^2)$,
- $P\{N \geq 2\} = 1 - \exp (-\pi m R_1^2) - (\pi m R_1^2) \exp (-\pi m R_2^2)$.

These probabilities of interaction can be determined for any number of interactions. Once the number of interactions is
determined, then the size distribution can be determined by using the equilibrium conditions between surface tension, internal gas pressure, external mean stress, and conservation of fission gas.

When the bubbles that have migrated to the grain boundary are observed on a two-dimensional section, they may be categorized as being intragranular because the planar section may be inclined so as not to have the grain boundary passing through the intersection. A correction factor must be applied to account for the bubbles classified as intragranular which are really intergranular. If $R$ voids are on grain boundaries and $A$ voids are not on grain boundaries, the corrected total number estimates are $R(1 + 1/p)$ and $A(1 - lp)$, respectively. Here, $p$ is the probability that an intergranular void appears on a grain boundary when the void is sectioned by a randomly positioned and oriented plane.

The value of $p$ was calculated assuming intergranular pores are lenticular. Two alternative formulas for $p$ were derived as the method of numerical calculation of $p$ depends upon whether the pores are near spherical or not. Specifically,

$$p = \frac{a}{2} \sum_{j=0}^{\infty} B\left(\frac{3}{2}, \frac{i+1}{2}\right)b^j.$$  \hfill(1)

$$p = \frac{a}{b^2} \left[\frac{\pi}{2} + b - \sqrt{1 - b^2} \tan^{-1}\left(\frac{1 - b^2}{b}\right)\right].$$  \hfill(2)

where

$$a = \frac{2 \sin \theta_1 \sin \theta_2}{\sin \theta_1 + \sin \theta_2},$$

$$b = \frac{\sin (\theta_1 + \theta_2)}{\sin \theta_1 + \sin \theta_2}.$$
and $B(x,y)$ is the complete beta function. Here, $\theta_1$ and $\theta_2$ are the cap angles which define the shape of the lenticular pore. Formula (2) is best for near disk-like voids and (1) for near spherical voids.

**In-Reactor Swelling Measurements**

F. E. Panisko

In-reactor studies are in progress which isolate effects of fuel density, temperature, temperature gradient, fission rate and burnup on fission gas swelling of UO$_2$-25 wt% PuO$_2$. Irradiated specimens will be characterized through the use of quantitative electron microscopy in terms of analytical swelling models which are also being developed under this task. The experiment consists of simultaneously irradiating multiple (~20) fuel wafers with various thicknesses and fission atom densities to achieve different temperature gradients, burnups and fission rates. Fuel pressure and ambient temperature are controlled by externally heating a pressurized NaK annulus which surrounds the fuel capsule* and by varying insulation thickness adjacent to individual wafers.

Fuel densities in initial experiments are 94% and >99% of theoretical, the former being obtained from stocks of standard EBR-II experimental fuel and the latter produced by hot-pressing. Porosity fraction is a variable because pores and voids are expected to provide internal accommodation for much of the swelling.

Fuel temperature will be above 700 °C, a reasonable fuel surface temperature for FTR, and below ~2000 °C. Above the upper limit, all fission gas is assumed to be released. Fuel temperature influences fission gas swelling because fuel creep and pore or bubble migration are thermally activated processes.

* For a description of the controlled temperature-pressure irradiation capsule, see BNWL-SA-583 or USAEC-CONF-660511.
At higher temperatures, pores migrate more rapidly up the temperature gradient toward the fuel center.

Temperature gradient will be varied between \( \sim 0 \) and 8000 °C/cm. The lower value will be used as a basic reference; the higher value represents a maximum expected for oxide fuels in fast reactors. The combination of higher temperature and temperature gradient provides the necessary energy and force for bubble migration by a surface diffusion mechanism.

The fission rate will vary from 1.0 to \( 4.3 \times 10^{14} \) fissions per cm\(^3\)-sec. These rates are approximately the minimum and maximum achievable with available fuel and test facilities. The fission rate is expected to be most important in determining the dynamic concentration of bubbles in transit up the thermal gradient. The higher the fission rate the greater the anticipated saturation value of steady-state swelling.

Burnups for a constant time in the reactor will be varied from 20,000 to 80,000 MWD/MTM to provide a range representative of expected FTR conditions.

Fuel temperature is expected to be one of the most influential variables. Meaningful measurements require accurate knowledge of fuel temperature. Uncertainties in such parameters as neutron spectrum, cross-section, and interface heat transport are reflected in unacceptable imprecisions in calculated temperature profiles. To correct this, a prototype capsule will be irradiated during April 1970 to provide both in-pile calorimetric data and postirradiation, ceramographic evidence of fuel operating temperatures. This test will also provide an overall appraisal of procedures for capsule fabrication and postirradiation examination. Twenty-seven, 94%-dense, \( \text{UO}_2 \)-25 wt% \( \text{PuO}_2 \) fuel wafers will be included in the first test. The fuel diameter is 0.2 in. and lengths are 0.01, 0.10 and 0.20 in. The fuel contains 0.71 (normal), 30, and
93.15% enriched uranium and 11% $^{240}$Pu in the plutonium fraction. Fifteen of the fuel wafers have flat, polished surfaces which mate with flat, polished surfaces on adjacent molybdenum heat transfer plugs. This special surface preparation is expected to minimize the temperature drop across the fuel-molybdenum interfaces. To provide a comparison with standard surfaces, the remaining specimens are not polished. Table 3.9 gives dimensions, locations, enrichments, and surface conditions for each specimen included in the first capsule.

Figure 3.27 shows a molybdenum specimen holder. The holder is 0.605 in. by 9 in. long with 18-0.210 in. diameter, threaded holes. Complete, three-dimensional thermal characterization will be provided by five Chromel-Alumel thermocouples positioned inside the specimen holder and four placed outside the stainless steel cladding in the NaK.

**TABLE 3.9. Fuel Arrangement for Initial UO$_2$-25 wt% PuO$_2$ Swelling Capsule**

<table>
<thead>
<tr>
<th>Capsule Position</th>
<th>Surface Preparation</th>
<th>Top (Left) Uranium Enrichment-Length (in)</th>
<th>Center Uranium Enrichment-Length (in)</th>
<th>Bottom (Right) Uranium Enrichment-Length (in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>None</td>
<td>93%-0.013</td>
<td>93%-0.013</td>
<td>93%-0.013</td>
</tr>
<tr>
<td>2</td>
<td>Polished</td>
<td>93%-0.013</td>
<td>93%-0.013</td>
<td>93%-0.013</td>
</tr>
<tr>
<td>3</td>
<td>None</td>
<td>N-0.013</td>
<td>N-0.013</td>
<td>N-0.013</td>
</tr>
<tr>
<td>4</td>
<td>Polished</td>
<td>N-0.013</td>
<td>N-0.013</td>
<td>N-0.013</td>
</tr>
<tr>
<td>5</td>
<td>None</td>
<td>93%-0.013</td>
<td>93%-0.013</td>
<td>93%-0.013</td>
</tr>
<tr>
<td>6</td>
<td>Polished</td>
<td>93%-0.013</td>
<td>93%-0.013</td>
<td>93%-0.013</td>
</tr>
<tr>
<td>7</td>
<td>Polished</td>
<td>N-0.200</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Polished</td>
<td>N-0.013</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>Polished</td>
<td>30%-0.013</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>None</td>
<td>30%-0.013</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>Polished</td>
<td>93%-0.013</td>
<td></td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>None</td>
<td>93%-0.013</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>None</td>
<td>93%-0.013</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>---</td>
<td>No fuel</td>
<td></td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>Polished</td>
<td>N-0.100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>Polished</td>
<td>30%-0.100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>None</td>
<td>30%-0.100</td>
<td></td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>Polished</td>
<td>93%-0.100</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
FIGURE 3.26. Molybdenum Specimen Holder for UO$_2$–25 wt% PuO$_2$ Irradiation Swelling Experiment. Also shown are Thermocouples and Stainless Steel Clad.
Oxide nuclear fuel materials have a relatively low thermal conductivity in the temperature range where they operate, resulting in very steep thermal gradients when run at high specific power levels. A thermal gradient results in potential field gradients which can be considered as driving forces for mass transport. There are two main interacting components of this force which are being investigated theoretically and experimentally. These are the electronic component, characterized by the electronic thermoelectric power (electronic heat of transport) and the ionic component, characterized by the ionic heat of transport.

Flux Equation

As a first approximation, the diffusion controlled mass transport in a thermal gradient can be described by a relationship which states that the mass flux is directly proportional to the sum of the forces which are imposed upon the diffusing species. The constant of proportionality is called the diffusion coefficient. This is stated mathematically by:

\[ J = -D \Sigma \text{(Forces)} \]

Diffusion Coefficient

In a system such as UO₂ or (U,Pu)O₂ the diffusion coefficients highly depend upon composition. This can be understood by examining the form of the random-walk diffusion coefficient:

\[ D = \frac{1}{6} \Gamma \alpha^2 \]

where \( \Gamma \) is the atomistic jump frequency and \( \alpha \) is the actual jump distance. The jump distance depends upon the mechanism of diffusion and the crystal structure. The jump frequency
not only depends upon the mechanism of diffusion but more
directly upon the defect structure of the oxide and is a
probabilistic function of the ability of an ion to move from
one position through an energy col to another. Since an ion
cannot diffuse in a perfect lattice, defects must be present
for mass transport. The probability of position change of the
diffusing specie is directly related to the defect density.
The defect structure is also directly related to the degree of
nonstoichiometry and thus both the diffusion mechanism and the
compositional dependency are highly dependent upon the degree
and type of deviations from stoichiometry.

Experimental data for the diffusion of oxygen in $\text{UO}_2^{+x}$
are available (11) and the concentration dependencies have been
and are being investigated (12). The treatment by Thorn and
Winslow (12a) and experimental data by Auskern and Belle (12b)
for hyperstoichiometric $\text{UO}_2$ was used in these mass transport
calculations since they included concentration and temperature
dependencies in the equation. The diffusion coefficient for
oxygen in $\text{UO}_2$ is given by:

$$D(C,T) = 2.016 \times 10^{-2} \, e^{-\frac{1.52}{kT}}$$

$$\times \left\{ (O/M - 2) + \left[ (O/M - 2)^2 + 495 \, e^{-\frac{1.62}{kT}} \right]^{1/2} \right\}.$$ 

This equation is good only for $O/M \geq 2.0$ and cannot val-
ibly be applied to the hypostoichiometric case. (The coeffi-
cient becomes negative for some values of $T$ and $O/M < 2$).
There are no experimental diffusion coefficients in $\text{UO}_2^{-x}$. It
was assumed that for negative deviations from stoichiometry
the value for the diffusion coefficient again increases
because of an increase in the concentration of defects. The
quantitative nature of this increase is unknown so that for
the purpose of the calculation a mirror image of the composi-
tional dependency of the equation for $\text{UO}_2^{+x}$ was used for $\text{UO}_2^{-x}$. 

3.63
Forces

The simple assumption that the flux is directly proportional to the magnitude of the summation of the forces is not altogether correct. The fundamentals of nonequilibrium thermodynamics (13) state that there exist cross-coupling terms reflecting interactions between forces. However, for these purposes the cross-coupling terms will be neglected.

At the present time, three forces are being considered in the case of mass transport in oxide fuel in a thermal gradient. The first force is the chemical force or the classical concentration gradient as found in Fick's First Law for isothermal diffusion in a concentration gradient.

Chemical Force = \nabla C.

In the case of thermal diffusion, when steady-state is achieved (J = 0) it is the \nabla C_{ss} (steady-state) force which exactly balances the sum of any other forces present.

The general form for the force term is given by:\n
\text{Generalized Force} = \frac{C}{kT} \nabla E

where C is the concentration, T is the absolute temperature, and \nabla E is the potential energy gradient.

For the thermal gradient situation, the gradient of the temperature, \nabla T, must be related to the forces generated by it. These \nabla T forces at the present time are being divided into two separate components, the one resulting from electronic processes and the other resulting from ionic processes.

The ionic portion is characterized by an ionic heat of transport parameter (Q_1) and the mathematical form for this force is given by:
Ionice Thermal Force = \frac{Q^* C}{kT} \nabla T

where \nabla T is the thermal gradient.

The electronic portion of the thermal force which is exerted on the ions can be described in terms of the electronic thermoelectric force which builds an electrical potential gradient across the fuel. This force can be described by:

Electronic Thermal Force = \frac{qC}{kT} \nabla \phi

where q is the charge on the ion and \nabla \phi is the electrical potential gradient. The term \nabla \phi can be given in terms of the thermoelectric emf, \Theta_e, as:

\nabla \phi = \Theta_e \nabla T.

Using these equations the flux equation can be rewritten as:

\[ J = -D \left[ \nabla C + \frac{C}{kT} \left( \frac{Q^*}{T} + q\Theta \right) \nabla T \right]. \]

Fryxell and Aitken\(^{(14)}\) have published a value of \( \sim 1 \) eV (\( \sim 23 \) kcal/mole) for the heat of transport for \( 0^{-2} \) in \( \text{UO}_2-x \). Their experimental technique imposed a possible gas phase transfer and an electrical shorting of the electronic thermoelectric emf so that the value obtained may or may not represent the true ionic heat of transport for \( 0^{-2} \) in \( \text{UO}_2-x \). The data indicated little, if any, dependency of \( Q^* \) on concentration.

Bates and Dau\(^{(15)}\) measured the thermoelectric power of "nearly stoichiometric" \( \text{UO}_2 \) including both single and polycrystal specimens. They found a p-n switch in the sign of the thermoelectric power at about 1200 °C. The data obtained for the polycrystalline specimen were curve-fitted to the equation:
\[ \Theta = 2.021 \, e^{-0.00479T} - 0.00255 \] (volts/°K)

Since these measurements were made in a constant (but undetermined) oxygen partial pressure and small thermal gradients, it might be assumed that these values are from electronic processes alone and that a negligible concentration gradient was formed.

All of the above parameters are inserted into the calculation in a cylindrical system where the initial O/M is set at 2.00 and a parabolic temperature gradient exists along the radial direction. The central temperature was taken as 2000 °C and the outer surface temperature taken as 1200 °C. The value for the thermal gradient increases linearly from zero at the center and reaches its maximum at the surface of 1600 °C/cm.

The signs for both the ionic and electronic thermal forces indicate that for the temperature range used, the oxygen ion flux would be directed from the hot center toward the cooler skin. However, in examining the magnitude of the two forces and comparing them with reasonable values for the resulting VC, it is found that the ionic thermal force is at least one order of magnitude larger than a reasonable VC and that the electronic thermal force is at least two orders of magnitude larger than a reasonable concentration gradient.

The reasons for these discrepancies are presently under investigation. The primary contributing factor is the limited knowledge of how the vital parameters depend upon both stoichiometry and temperature.

The interpretation of the presently available literature data is also under review. The derivation of the ionic heat of transport, \( Q^*_i \), depends upon the experimental measurement of the steady-state O/M gradient which developed in \( \text{UO}_2-x \) which was maintained in a temperature gradient.\(^{14}\) In the
steady-state, the ionic flux disappears and a further assumption is made that the metal container for the experiment shorted out the electronic forces so that the third term in the flux equation is zero. Under these conditions, the relationship is given by:

$$\nabla C = \frac{Q^*}{kT^2} \nabla T.$$  

Manipulation of this differential equation gives:

$$\frac{d \ln C}{d (1/T)} = \frac{Q^*}{k}$$

which implies that the slope of the $\ln C$ versus $1/T$ curve should be constant (under the above assumptions and, further, that $Q^*_i$ is concentration and temperature independent) and yields a value proportional to the ionic heat of transport. However, the value of $1 \text{ eV}$ obtained by Fryxell and Aitken was determined from the slope of the $\ln x$ (in $\text{UO}_2-x$) versus $1/T$ plot which gives a completely different result than the equation above. A replotting of Fryxell and Aitken's data in the form of $\ln (\text{O/M})$ versus $1/T$ gives curved lines and much smaller slopes. The implications of this derivation are being investigated.

The thermoelectric power term is also being reviewed, as it is the dominant calculated force. It may be that there is a significant ionic thermoelectric emf contribution to the measured potentials. An experimental technique is being devised to investigate the nature of the electrical potentials developed in oxide fuel when subjected to both small and large thermal gradients.
REFERENCES


4.0 IRRADIATION DAMAGE TO REACTOR METALS

IRRADIATION BEHAVIOR OF STRUCTURAL MATERIALS

R. P. Marshall

Irradiation Facilities Operation

A. L. Ward

The purpose of this phase of the program has been to provide for the timely accomplishment of irradiation, testing, and subsequent processing of the data obtained from a variety of reactor structural materials. Irradiation experiments in the ETR G-7 pressurized hot water loop have been discontinued and the in-pile pressure tube has been removed.

The experiments discharged from the G-7 loop for Cycles 103 and 104 are summarized in Table 4.1 along with estimated neutron fluxes and fluences. Table 4.2 gives the operating history of the ETR and the loop for Cycles 103 and 104.

In-Reactor Measurements

E. R. Gilbert and N. E. Harding

Irradiation-induced creep in Types 304 and 316 SS has been measured in a thermal reactor (flux-tailored and enhanced test facility in a Hanford K Reactor) at total neutron fluences up to $5 \times 10^{21} \text{n/cm}^2$ ($E > 10^{-10} \text{ MeV}$). These measurements extend the temperature range over which irradiation-induced creep has been observed up to 370 °C, which is within the operating temperature range for LMFBR and FFTF components. Irradiation-induced creep is pertinent to the design and performance of stainless-steel components subjected to large amounts of intense radiation in fast-reactor cores. In some components, irradiation-induced creep is expected to be beneficial by permitting stress relaxation in cases where thermal stresses and distortions caused by irradiation-induced swelling might otherwise seriously limit reactor performance.
### TABLE 4.1. Summary of Experiments Discharged at Conclusion of ETR Cycles 103 and 104

<table>
<thead>
<tr>
<th>Experiment Designation</th>
<th>No. of Specimens</th>
<th>Specimen Type</th>
<th>Estimated Flux, n/cm²/sec (^{(a)})</th>
<th>Estimated Fluence, n/cm² (^{(a)})</th>
</tr>
</thead>
<tbody>
<tr>
<td>GEH-20-264</td>
<td>2</td>
<td>A-542, DCB</td>
<td>(4.5 \times 10^{13})</td>
<td>(2.2 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-265</td>
<td>2</td>
<td>A-542, DCB</td>
<td>(4.5 \times 10^{13})</td>
<td>(2.2 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-333</td>
<td>72</td>
<td>S/S, Tensile</td>
<td>(9.6 \times 10^{13})</td>
<td>(1.6 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-334</td>
<td>72</td>
<td>S/S, Tensile</td>
<td>(7.2 \times 10^{13})</td>
<td>(1.2 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-326</td>
<td>72</td>
<td>S/S, Tensile</td>
<td>(9.0 \times 10^{13})</td>
<td>(2.9 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-327</td>
<td>72</td>
<td>S/S, Tensile</td>
<td>(1.1 \times 10^{14})</td>
<td>(3.4 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-289</td>
<td>18</td>
<td>S/S, Tensile</td>
<td>(2.7 \times 10^{13})</td>
<td>(8.6 \times 10^{20})</td>
</tr>
<tr>
<td>GEH-20-180</td>
<td>18</td>
<td>Ni-Base, Tensile</td>
<td>(3.0 \times 10^{13})</td>
<td>(3.1 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-315</td>
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<td>S/S, Tensile</td>
<td>(1.1 \times 10^{14})</td>
<td>(3.0 \times 10^{20})</td>
</tr>
<tr>
<td>GEH-20-354</td>
<td>9</td>
<td>S/S, Tensile</td>
<td>(1.1 \times 10^{14})</td>
<td>(2.2 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-316</td>
<td>18</td>
<td>S/S, Tensile</td>
<td>(1.1 \times 10^{14})</td>
<td>(3.0 \times 10^{20})</td>
</tr>
<tr>
<td>GEH-20-312</td>
<td>20</td>
<td>S/S, Tensile</td>
<td>(1.1 \times 10^{14})</td>
<td>(1.9 \times 10^{21})</td>
</tr>
<tr>
<td>GEH-20-353</td>
<td>9</td>
<td>S/S, Tensile</td>
<td>(6.0 \times 10^{13})</td>
<td>(1.2 \times 10^{21})</td>
</tr>
</tbody>
</table>

**NOTE:** Total specimens discharged, ETR G-7 loop Cycles 103, 104 = 402. Total specimens discharged, ETR G-7 loop to date = 4500.

\(^{(a)}\) Neutrons with energies greater than 1 MeV.

The data are shown in Figure 4.1 as irradiation-induced tensile strain per unit tensile stress as a function of total neutron fluence. The temperatures and stresses are sufficiently low that after initial loading, strains saturate within a few hours and further deformation is not observed in the absence of neutron irradiation. The creep rate induced by neutron irradiation can be represented in terms of a linear stress dependence, thus allowing the normalized strain parameter to be used. Four different capsules were used to generate these data as indicated in Figure 4.1. Strain transients which occurred during the tests, such as upon changing stress or temperature in Capsule IV-36, were subtracted out to allow plotting the continuous strain increase with neutron fluence.
TABLE 4.2. Summary of Operating History for ETR and G-7
Hot Water Loop - ETR Cycles 103 and 104

<table>
<thead>
<tr>
<th>Operating History</th>
<th>ETR Cycle Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Reactor History</td>
<td>Cycle 103</td>
</tr>
<tr>
<td>Start up (a)</td>
<td>7-10-69</td>
</tr>
<tr>
<td>End of Cycle</td>
<td>8-28-69</td>
</tr>
<tr>
<td>Megawatt Days</td>
<td>5875</td>
</tr>
<tr>
<td>Effective Days at 175 MW</td>
<td>33.57</td>
</tr>
<tr>
<td>Number of Scrams (b)</td>
<td>7</td>
</tr>
<tr>
<td>Number of Shutdowns</td>
<td>2</td>
</tr>
<tr>
<td>2. G-7 Hot Water Loop History</td>
<td>Cycle 104</td>
</tr>
<tr>
<td>Maximum Temperature</td>
<td>544</td>
</tr>
<tr>
<td>Effective Days Above 200 °F</td>
<td>35.7</td>
</tr>
<tr>
<td>Effective Days at Operating Temp.</td>
<td>32.2</td>
</tr>
<tr>
<td>Operating Efficiency</td>
<td>94.1%</td>
</tr>
<tr>
<td>Temperature Efficiency (d)</td>
<td>90.3%</td>
</tr>
<tr>
<td></td>
<td>545</td>
</tr>
<tr>
<td></td>
<td>52.4</td>
</tr>
<tr>
<td></td>
<td>47.2</td>
</tr>
<tr>
<td></td>
<td>95.4</td>
</tr>
<tr>
<td></td>
<td>90.2</td>
</tr>
</tbody>
</table>

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).
FIGURE 4.1. Irradiation-Induced Tensile-Strain per Unit Tensile Stress Versus Total Neutron Fluence for Austenitic Stainless Steels in a K Reactor Flux Tailored and Enhanced Facility
The creep strain in Figure 4.1 appears to follow a transient and a steady-state behavior. The 20% cold-worked Type 316 SS underwent more transient strain than the solution-treated Type 304 SS. This observation is in qualitative agreement with UK data which showed more transient irradiation-induced creep in 20% cold-worked than in solution-treated Type 316 SS for measurements made on helical springs at 100 °C in the DMTR.\(^{(1)}\) The irradiation-induced creep strain can be represented by the McVetty Equation\(^{(2)}\)

\[
\varepsilon = A \frac{\sigma}{E} (1 - e^{-\frac{\phi t}{B}}) + C \phi t
\]  

where \(\varepsilon\) is the irradiation-induced strain, \(A\) is the transient strain amplitude which is on the order of 0.1, \(\sigma\) is the applied stress, \(E\) is the elastic modulus, \(\phi\) is neutron flux, \(t\) is time, \(B\) is a decay constant, and \(C\) is a rate constant which is on the order of \(10^{-30}\ \text{cm}^2/\text{psi}\). Fluence in place of the time parameter in the original McVetty equation has been used by Hesketh\(^{(3)}\) to describe the irradiation-induced creep strain in the austenitic stainless steel alloy EN58B in the Herald Reactor at 43 °C.

Irradiation-induced creep experiments in the form of stress-relaxation tests have resulted in more stress relaxation in tension\(^{(4)}\) than in torsion,\(^{(5)}\) as shown in Figure 4.2. Likewise, comparison of the tensile irradiation-induced creep data in Figure 4.1 with data obtained on helical springs\(^{(6)}\) in the same temperature range show more creep per unit fluence per unit stress for the tensile test. In making the comparison, the differences in the state of stress were accounted for with the conventional equations for effective strain and effective stress.\(^{(7)}\) A relationship between strain rate and stress for an isotropic material was developed by Gilbert and Straalsund\(^{(8)}\) which could account for the observed differences between irradiation-induced creep in torsion and
in tension. Whereas the conventional equations for effective strain and effective stress are based on conservative volume or a Poisson's ratio of 1/2, the derived relationship can be applied to the case where volume increases occur during creep and Poisson's ratio relating the plastic Young's modulus and the plastic modulus of rigidity can vary from 1/2 for conservative volume to -1 for isotropic swelling. For the irradiation-induced creep data on austenitic stainless steels, the analysis indicated that large ratios of $\beta$ to $\alpha$ as noted in Figure 4.2 reflect stress-induced volume changes during creep and about 5/6 of the measured creep strain in the uniaxial tensile test is due to stress-induced swelling. Less irradiation-induced creep strain is observed in the pure shear torsion test due to the absence of a hydrostatic-pressure component which could increase swelling.

![Fractional Stress Relaxation Versus Fluence](image)

**FIGURE 4.2.** Fractional Stress Relaxation Versus Fluence as Measured in Tensile and Torsional Stress Relaxation Experiments and Compiled from Tensile Creep Experiments.
A comparison of available in-reactor creep data is presented in Figure 4.3 for solution-treated austenitic stainless steels. Since these data were obtained in tests involving different states of strain and stress, the data were converted to effective strain and effective stress with the relationship described above which was not limited to conservative-volume creep.

The transient creep term in Equation (1) has been attributed to dislocation climb. Under irradiation, dislocations climb by absorbing more interstitials than vacancies. The application of stress leads to small departures from their equilibrium positions in directions which produce strain. The steady-state component has been associated with the collapse of vacancy cascades to dislocation loops, with stress providing a preference among the available planes upon which the loops may collapse. Since cascade production and loop formation depend primarily upon displacement cross sections and elastic properties of the alloys, austenitic stainless steels possessing similar displacement cross sections and elastic properties should display similar steady-state behavior.

The data shown in Figure 4.3 for DFR and K Reactor at comparable temperatures are in agreement; however, lower temperature data from Herald Reactor and DMTR show more creep. Thus it may be concluded that irradiation-induced creep decreases slightly with increasing temperature. This trend is opposite to that usually displayed by thermally-activated creep processes. Further analysis of these data which attempts to separate the temperature and the neutron-energy dependence is presented elsewhere.
Effective Strain per Unit Effective Stress Versus Neutron Fluence for Solution-Treated Austenitic Stainless Steels

**FIGURE 4.3.** Effective Strain per Unit Effective Stress Versus Neutron Fluence for Solution-Treated Austenitic Stainless Steels
Fracture Analysis
C. L. Hellerich

The purpose of this program is to gain an understanding of the deformation and fracture of the candidate materials for advanced LMFBR applications. Selected specimens from the tensile and uniaxial creep-rupture studies are being examined to determine the effects of fast neutron irradiation and environment on fracture behavior and to correlate any transitions in this behavior with the relevant mechanical property changes.

Work is currently in progress to determine the effects of aging temperature, test temperature, and irradiation conditions on the creep-rupture fracture of Type 316 SS. In order to isolate the role of aging temperature, specimens aged at 800, 900 and 1400 °F and creep-rupture tested at 1000 °F under a stress of 50,000 psi were examined in the scanning electron microscope (SEM). Test results for these specimens have been reported previously,\(^\text{(11)}\) but for convenience are shown again in Table 4.3. Examination of the fractographs in Figures 4.4, 4.5 and 4.6 shows that a transition from an intergranular fracture to a transgranular, plastically dimpled fracture occurred as the aging temperature increased. Quantitative evaluation, Table 4.3, of the mixed mode behavior showed very little difference in the fracture of the 800 and 900 °F aged material; however, at an aging temperature of 1400 °F, the fracture was entirely plastic.

Close examination of the grain boundary facets of the 800 and 900 °F aged materials, Figures 4.4b and 4.5b, showed that they were void of any recognizable plastic fracture mechanisms, and the intergranular portion of the failure must be classed as brittle, intergranular fracture. High magnification metallography, Figure 4.7, of polished and cathodically etched longitudinal sections of the material
<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Temp., F.</th>
<th>Creep Rate, in./in. hr</th>
<th>Rupture Time, hr</th>
<th>Elongation, %</th>
<th>Intergranular Fracture, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>9317</td>
<td>800</td>
<td>50</td>
<td>1000</td>
<td>631.4</td>
<td>22.1</td>
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<td>9157</td>
<td>900</td>
<td>50</td>
<td>1000</td>
<td>450.8</td>
<td>22.1</td>
</tr>
<tr>
<td>9282</td>
<td>1400</td>
<td>50</td>
<td>1000</td>
<td>377.7</td>
<td>42.22</td>
</tr>
<tr>
<td>9163</td>
<td>Sol. Treated 1000</td>
<td>40</td>
<td>2795</td>
<td>13.9</td>
<td></td>
</tr>
<tr>
<td>9134</td>
<td>Sol. Treated 1100</td>
<td>32</td>
<td>2795</td>
<td>13.9</td>
<td></td>
</tr>
<tr>
<td>9319</td>
<td>800</td>
<td>25</td>
<td>1200</td>
<td>193.4</td>
<td>39.8</td>
</tr>
<tr>
<td>9319</td>
<td>1200</td>
<td>25</td>
<td>1200</td>
<td>193.4</td>
<td>39.8</td>
</tr>
</tbody>
</table>

a. Remainder is transgranular plastic dimpling.

b. ND - Not determined.
FIGURE 4.4. Mixed Mode Fracture in Type 316 SS Solution-Treated 1950 °F/1 hr, Then Aged at 800 °F/3045 hr. Creep-Rupture Tested at 1000 °F, 50,000 psi, 22% Total Elongation. Fracture is Primarily Brittle Intergranular (70%). Remainder is Plastic Dimpling (30%)
FIGURE 4.5. Mixed Mode Fracture in Type 316 SS Solution-Treated 1950 °F/1 hr, Then Aged at 900 °F/3045 hr. Creep-Rupture Tested at 1000 °F, 50,000 psi, 20% Total Elongation. Fracture is Primarily Brittle Intergranular (65%). Remainder is Plastic Dimpling (35%)
Figure 4.6. Transgranular Plastic Dimpling in Type 316 SS Solution-Treated 1950 °F/1 hr, Then Aged at 1400 °F/3045 hr. Creep-Rupture Tested at 1000 °F, 50,000 psi, 42% Total Elongation. Region of Plastic GB Separation Can Be Seen at Arrows in a but Fracture Is Predominately Well-Defined Transgranular Plastic Dimpling as Shown in b.
FIGURE 4.7. Metallographic Longitudinal Section of Type 316 SS Solution-Treated at 1950 °F/1 hr and Aged at 900 °F/3045 hr. Creep-Rupture Tested at 1000 °F, 50,000 psi, 22% Total Elongation. Note Numerous Grain Boundary Microcracks Below Fracture Surface in a. Cathodic Etching Revealed Presence of a Continuous Platelet Form of Precipitate at the Grain Boundary Microcracks. b and c Show Platelets at Cracks Indicated by Arrows in a.
aged at 900 °F revealed the presence of uniform platelets of carbide precipitates at the grain boundaries. These were best revealed at microcracks below the actual fracture surface. It appears that the presence of this platelet form of precipitate at the grain boundaries leads to the formation of brittle intergranular microcracks during creep-rupture testing. Ultimate failure occurs by the link up of these microcracks by transgranular, plastic dimpling, as shown in Figures 4.4 and 4.5. In contrast to this behavior, the material aged at 1400 °F showed extensive carbide precipitation and agglomeration both at the grain boundaries and throughout the matrix, Figure 4.8. The formation of voids (dimples) around the precipitates is apparent in Figure 4.8b and demonstrates the dimpling process that leads to the plastically dimpled fracture in Figure 4.6. Also, grain elongation and voiding at grain boundary precipitates, indicative of a high ductility fracture, can be seen.

The observed transition in fracture behavior correlates well with an abrupt change in ductility (total elongation). It may seem a misnomer to call a fracture that has seen 22% elongation a brittle, intergranular fracture or to call a change in ductility from 22% to 42% abrupt; however, if one subtracts out the transient strain data, it can be seen that a significant portion of the total deformation occurred as time independent, plastic deformation during specimen load up. For example, a solution treated control tested at 50,000 psi and 1000 °F failed with a total elongation of 18.2% but 17.3% of this occurred as time independent, transient strain.\(^{(11)}\) Thus, the specimen strained only \(\sim 1\)% during creep-rupture, a rather low ductility event. This is contrasted to the 1400 °F aged material which had \(\sim 14\)% transient strain but failed with a total elongation of 42%. In this case, the specimen strained
Figure 4.8. Metallographic Longitudinal Section of Type 316 SS Solution-Treated 1950 °F/1 hr and Aged at 1400 °F/3045 hr. Creep-Rupture Tested at 1000 °F, 50,000 psi, 42% Total Elongation. Note Uniform Nucleation and Agglomeration of Carbides Throughout the Matrix and at Grain Boundaries a. Voiding at Both Grain Boundary and Matrix Precipitates Is Indicative of Highly Deformed Structure.
during creep-rupture. This marks a sharp ductility transition (1% to 30%) and correlates well with the observed fracture behavior transition.

To establish the effect of test temperature, the fracture surfaces of specimens tested at 1000, 1100 and 1200 °F were examined. As shown in Figure 4.9, these specimens also exhibited a transition in fracture mode from intergranular to predominately transgranular fracture by a plastic dimpling mechanism. It is of interest to note the cavities in the fracture surface of the 1200 °F test, Figure 4.9. These cavities resulted from the formation of grain boundary microcracks and subsequent plastic growth during the creep-rupture of the specimen. This sequence of events is indicated by the presence of grain boundary detail at the bottom of the cavities and is consistent with the model of microcracking at carbide platelets previously suggested.

Although it is difficult to pinpoint from these initial results just when the grain boundary platelet type of precipitates form, it is probable that they are forming during the actual creep-rupture test, especially in the solution treated and the 800 and 900 °F aged materials. Being a dynamic process which passes through the platelet stage to the formation of agglomerated carbides, the precipitation process is influenced by both the test temperature and the applied stress. It is possible that as the test temperature increases, homogeneous nucleation predominates and the precipitation process becomes so dynamic that it passes through the platelet stage, overaging to form agglomerated carbides before substantial microcracking and crack propagation can occur. Final failure then occurs as in the overaged (1400 °F aged) material. This sequence of events could lead to the mixed mode behavior and cavitation observed in the 1200 °F fracture. Work is currently being conducted to determine the precipitate morphology at the grain boundary microcracks.
FIGURE 4.9. Type 316 SS Creep-Rupture Fracture. a Mixed Mode Fracture at 1000 °F, 40,000 psi, 14% Total Elongation. Fracture Is Primarily Intergranular (70%) with Plastic Dimpling (30%) Linking Together the Brittle Intergranular Microcracks. b Plastic Fracture at 1200 °F, 25,000 psi. Fracture Is Primarily Transgranular, Plastic Dimpling. Cavities in Surface (see arrows) Formed by Plastic Growth from Intergranular Microcracks.
Work has also been started to determine the effect of irradiation on creep-rupture behavior. Specimens irradiated at 900 °F to a fluence of $1 \times 10^{22} \text{n/cm}^2$ ($E > 0.1 \text{MeV}$) and tested in creep-rupture (Table 4.4) were examined. The specimen tested at 1000 °F under a stress of 45,000 psi, Figure 4.10, exhibited a mixed mode fracture. However, as can be seen in Figure 4.10a, the mixed mode fracture occurred as two separate regions: one a predominately brittle, intergranular fracture, Figure 4.10b, and the other a predominately shear (transgranular, plastically dimpled) fracture, Figure 4.10c. The significance of this well-defined division of fracture mode is in describing the chronological sequence of events that led to final failure and in revealing the necessary occurrence of certain fracture processes. Because of the low ductility nature of the brittle, intergranular fracture, this region of failure necessarily developed during the early stages of fracture. The nearly total absence of any form of plastic fracture in this region, Figure 4.10b, is indicative of the relative ease with which grain boundary microcracks were able to link up and form a continuous, propagating flaw. Therefore, the sequence of events leading to fracture began with the early generation of intergranular microcracks which then linked up to form a propagating flaw covering ~35% of the specimen cross-sectional area. This resulted in an engineering net section stress of 69,000 psi, which is in line with the ultimate strength of the material (63,300 psi) at 1000 °F. At this point, final failure was by plastic instability resulting in the shear or transgranular, plastically dimpled fracture in Figure 4.10c.

The 1400 °F test, Figure 4.11, fractured by a single mode, intergranular. As seen in Figure 4.11a, oxidation of the fracture surface occurred during the test and resulted in the demarcation of microcracks that developed during
testing. As in the 1000 °F test, the microcracking is intergranular. Because all of the fracture surface was intergranular, however, final instability must have occurred by a rapid intergranular fracture process. Figure 4.11b is typical of the majority of the specimen and represents the region of rapid fracture. As can be seen, the failure is intergranular, but by a plastic dimpling process at the grain boundaries, i.e., a plastically dimpled, intergranular fracture.

**TABLE 4.4. Uniaxial Creep-Rupture of Irradiated Type 316 SS**

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Irrad. Temp., °F</th>
<th>Test Temp., °F</th>
<th>Stress, psi</th>
<th>Minimum Creep Rate, in./in./hr</th>
<th>Rupture Time, hr</th>
<th>% Elongation</th>
<th>% Intergranular Fracture</th>
</tr>
</thead>
<tbody>
<tr>
<td>14B66-1</td>
<td>900</td>
<td>1000</td>
<td>45,000</td>
<td>0.000230</td>
<td>107.8</td>
<td>1032</td>
<td>5.8</td>
</tr>
<tr>
<td>14B66-5</td>
<td>900</td>
<td>1400</td>
<td>10,000</td>
<td>0.000222</td>
<td>146.5</td>
<td>1050</td>
<td>5.3</td>
</tr>
</tbody>
</table>

**NOTE:** EBR-II Fluence for both specimens is $1 \times 10^{22}$ n/cm$^2$ (E > 0.1 MeV)

Thus, irradiation of Type 316 SS at 900 °F to a total fluence of $1 \times 10^{22}$ n/cm$^2$ (E > 0.1 MeV) produced two main changes in the fracture behavior during creep-rupture. First, when compared to the solution treated material, increased crack propagation characteristics were readily apparent. Secondly, the fracture mode at high temperature (1400 °F) for both the slow crack propagation and rapid unstable fracture was intergranular, whereas at 1000 °F the fracture mode during instability was transgranular. This suggests that in the irradiated material there was a transition in fracture
FIGURE 4.10. Mixed Mode Fracture in Type 316 SS Irradiated 900 °F to a Fluence of $1 \times 10^{22}$ n/cm². Creep-Rupture Tested at 1000 °F, 45,000 psi, 6% Total Elongation. Note Well-Defined Regions of Intergranular (35%) and Transgranular (65%) Fracture in a. Intergranular Fracture is Brittle as Shown in b. Transgranular Fracture, c, Is by a Plastic Dimpling Mechanism.
FIGURE 4.11. Intergranular Fracture in Type 316 SS Irradiated at 900 °F to a Fluence of $1 \times 10^{22}$ n/cm$^2$. Creep-Rupture Tested at 1400 °F, 10,000 psi, 5% Total Elongation. Note Oxidation Deposits in Area Mark Location of Intergranular Microcracks. Panel b Shows Typical Region of Rapid Intergranular Fracture. Note Dimples on Grain Facets Indicating a Plastic Dimpling Process at the Grain Boundary.
mode from transgranular to intergranular as test temperature increased, whereas in the control material the transition was from intergranular to transgranular. This contrasting behavior apparently was due to the complex interrelationship between the mechanical and metallurgical states of the material and the test conditions which together act to control the microcrack initiation process as well as the crack propagation rate and mode.

Finally, the observation of microcrack initiation and subsequent propagation in the creep-rupture tests of the irradiated materials points out a potential flaw sensitivity in these materials. Because of the importance of understanding the conditions responsible for this phenomenon, efforts directed towards defining the mechanical and metallurgical states as well as the stress conditions conducive to crack initiation and propagation have been started. These efforts have the objectives of:

- Experimentally demonstrating the crack propagation phenomena.
- Quantitatively defining the phenomena.
- Metallurgically describing the material state and its relationship to microcrack initiation and propagation.

The Stability of Voids in Neutron-Irradiated Nickel
G. L. Kulcinski, B. Mastel, H. E. Kissinger and T. K. Bierlein

As described in the previous quarterly report, voids observed in nickel after irradiation at 450 °C to a fluence of $3 \times 10^{21}$ n/cm$^2$ ($E > 0.1$ MeV) are extremely stable during high temperature annealing. In fact, very little change in either the size or the number density of voids occurs in regions greater than 15 μ from a boundary during two hour anneals at temperatures to 975 °C. The overall picture of void annealing as revealed in the present study is
one in which the outside surface of each grain becomes denuded of voids while the inside of the grain retains the character of the as-irradiated sample. This "unique" mode of void annealing must be considered in the interpretation of post-irradiation-high-temperature mechanical properties of irradiated nickel.

A particularly significant observation was made in samples which had been annealed at 1050 and 1150 °C. During these high temperature anneals, all voids had disappeared, but cavities had formed on the grain boundaries as shown in Figure 4.12. Similar cavities were not present on the grain boundaries of the unirradiated controls after annealing at 1050 or 1150 °C. Careful re-examination of the irradiated samples which were annealed at lower temperatures revealed that grain boundaries in these samples were normal and contained no cavities whatsoever. The observed cavities have several features common to bubbles. They are generally spherical instead of polyhedral. They enlarge during annealing: from 200 Å after the 1050 °C anneal to 500 Å after the 1150 °C anneal. Although an accurate count of the cavities is difficult to obtain, approximately $5 \times 10^8$ bubbles/cm$^2$ of grain boundary were present after the 1150 °C anneal. If these cavities are filled with gas to balance the surface tension forces, then the number of gas atoms required to stabilize these cavities is $\sim 1$ ppm. This is close to the 3 ppm of helium atoms which would be produced by this irradiation for an (n,α) cross-section of 1.03 b.\(^{15}\)

Figure 4.13 displays the average void diameters determined by four small angle X-ray scattering analysis techniques and compares them to the average diameter determined by transmission electron microscope (TEM) studies. It is apparent that the methods of Roess and Shull\(^{16}\) and that of Porod\(^{17}\) give sizes
FIGURE 4.12. Typical Grain Boundaries in Nickel After 2 hr Anneal at 1050 °C, Showing Cavities on Boundary
which are in particularly good agreement with the average diameters obtained by TEM. The average of the rectangular distribution of Roess and Shull shows the best agreement with the TEM results. It is particularly gratifying that the Roess and Shull and Porod diameters also reflect the observed TEM drop in void diameter from 420 to 360 Å after the 975 °C anneal.

As expected, the Guinier diameter was always 10 to 20% higher than that determined by TEM studies. The diameters obtained from the method described by Harkness (18) were consistently 20% smaller than the observed diameters. An important point of Figure 4.13 is that the SAS results substantiate the TEM result: the average void diameter is relatively constant at ≈400 Å up to the point of complete annealing (between 975 to 1050 °C).

Considerable success was also obtained in matching the void size frequency distribution determined from SAS studies to that derived from TEM studies. Figure 4.14 compares both the Maxwellian and rectangular distributions with the distribution determined by TEM techniques. The areas under all three curves were normalized to 100%. The agreement is quite acceptable considering the completely different manner in which the TEM and SAS distributions are derived. The agreement between the SAS and TEM distributions after the higher temperature anneals was also quite good.

Finally, it was found that one can use SAS to obtain semiquantitative estimates of the void density after various annealing treatments. Figure 4.15 shows that SAS techniques tend to overestimate the void density by a factor of ≈2, relative to the TEM values. Nevertheless, the general shape of the annealing curve is so similar to that determined by TEM that its shape is considered accurate.
Figure 4.14. Comparison of Void Size Distribution Determined by Transmission Electron Microscopy with Maxwellian and Rectangular Distributions Fitted to X-Ray Scattering Results. Sample: As-Irradiated High-Purity Nickel, $3 \times 10^{21} \text{n/cm}^2 (E > 0.1 \text{MeV})$ at 450 °C.
FIGURE 4.15. Densities of Voids in Irradiated Nickel as Determined by Small Angle Scattering (SAS) and Transmission Electron Microscopy (TEM) Techniques
It is felt that the present work shows that, at least for nickel, the void size distribution and the void number density can be determined by SAS techniques. While SAS techniques will never replace good TEM results, SAS measurements offer certain advantages. First of all, the time required to characterize a sample is considerably less than that required to properly thin a highly radioactive nickel sample, take proper pictures of the magnetic material, and perform the tedious task of measuring the size of several hundred voids in order to obtain an average size. An advantage of the SAS technique is that it averages over many voids ($\sim 10^{11}$) as compared to the several hundred customarily treated by TEM studies.

One of the disadvantages of SAS is that other unknown components of damage might contribute to the scattering. However, only a few transmission photomicrographs are required to settle this question. In the case of nickel, there were no other visible components of damage which could scatter X-rays and significantly alter the results. Other areas in which SAS is obviously lacking are the determination of the morphology of the voids and the detection of inhomogeneous void distributions such as grain boundary denuding. Therefore, it is felt that an in-depth study of voids in nickel after various thermal treatments can be accomplished most efficiently by combining SAS and TEM studies, the former for void size and density determinations and the latter for microstructural details and as a check on the density measurements.

It is worth noting at this point that the volume change, $0.64 \pm 0.14\%$, measured in the as-irradiated nickel by TEM techniques agrees quite well with the volume change from immersion density measurements made by Holmes,\textsuperscript{(19)} who reported a 0.7% volume change for Ni-270 irradiated in the same capsule as the foils used in this study.
There are two rather unexpected results in the annealing behavior of the voids in the nickel samples studied: the relatively high temperature (~1000 °C) required to remove all voids and the constancy of the average void size during the annealing treatments. Because both of these results were substantiated by results from three separate techniques, micro-hardness measurements, TEM, SAS, there is little doubt about the validity of these observations. However, the results are contrary to results reported by Brimhall and Mastel (20) who found that all the voids in nickel irradiated to a fluence of \(1 \times 10^{20} \text{n/cm}^2\) \((E > 0.1 \text{ MeV})\) disappeared at 800 °C and that the average size of voids increased with increasing annealing temperature.

Past studies of the annealing of voids in copper and aluminum (21,22,23) suggest that for an isolated void with nearby vacancy sinks, the shrinkage rate can be described as

\[
-\frac{dr}{dt} = \frac{D_s}{r} \left[ \exp \left( \frac{2\gamma\Omega}{rKT} \right) - 1 \right]
\]

where \(r\) is the radius, \(D_s\) is the self diffusion coefficient (including structure factor), \(\Omega\) is the atomic volume, \(\gamma\) is the surface energy, \(T\) is the absolute temperature, \(k\) is Boltzmann's constant, and \(t\) is the time.

Equation (2) assumes that whenever a vacancy is removed from one void it is absorbed by a vacancy sink and that the average void size should decrease with increasing temperature. An implicit assumption in Equation (2) is that the spacing between the voids and sinks is much smaller than the average spacing between the voids themselves and that the emitted vacancy does not significantly change the vacancy concentration \((C_v)\) from the equilibrium value \((C_v^e)\). However, when the spacing between the voids is much less than between the voids and sinks,
it is reasonable to expect that some voids will capture the vacancies emitted by other voids. Such a situation can exist in heavily neutron-irradiated metals and is thought to be the reason why previous authors have found that larger voids grow at the expense of smaller voids.\(^{(20,24)}\) This latter situation would result in an increase in average void size with increasing temperature. However, the constant average void size with increasing annealing temperature found in this study does not fit either of the above situations.

A clue to the unusual behavior of the voids may be found in a recent analysis by Bullough and Perrin\(^{(25)}\) who treated the case of partially gas-filled cavities. The word cavities is used to distinguish between voids which contain only vacancies, and bubbles which are in mechanical and thermodynamic equilibrium with external restraints. These investigators found that the shrinkage rate of a cavity is given by

\[
\frac{dr}{dt} = \frac{1}{4} \frac{D_s}{a} \left[ \exp\left( \frac{E_I(l)}{kT} \right) \right] \frac{F_m}{C_v} - \frac{C_e}{C_v}
\]

where "a" is the interatomic spacing in the direction of the jump, \(E_I(l)\) is the spacial attractive interaction energy between a vacancy and a void at a distance \(l\), usually taken as \(3a/2\), and \(F_m\) is the mechanical force tending to shrink the cavity which is equal to \(P + \frac{2}{r} - \frac{3nkT}{4\pi r^3}\), where \(P\) is the external pressure and \(n\) is the number of gas atoms in the cavity. Bullough and Perrin\(^{(25)}\) considered the case of postirradiation annealing where the vacancy concentration depends on the rate at which vacancies can diffuse away from the cavity. For \(P = 0\) they found

\[
\frac{dr}{dt} = \frac{D_s}{2a} \left[ \exp\left( \frac{2\gamma_0}{rkt} - \frac{3n\Omega}{4\pi r^3} \right) - 1 \right] \frac{E_I\left(\frac{3a}{2}\right)}{\left[1 + \frac{r}{2a} \exp\left( \frac{E_I\left(\frac{3a}{2}\right)}{kT} \right) \right]}. \tag{4}
\]
Comparing Equation (4) with Equation (3), one finds that two forces tend to slow down the shrinkage rate: (1) the pressure of the gas atoms in the cavity, and (2) the attraction between the vacancy and the cavity. Furthermore, one can see that for small $r$ and large $n$ the term $\exp\left[\frac{2\gamma}{rkT} - \frac{3n}{4\pi r^3}\right]$ can be less than 1, and it is possible for small cavities to grow and large cavities to shrink. Such behavior would result in a relatively constant average size and would tend to increase the thermal stability of the cavities. These observations are entirely consistent with the data presented in Figure 4.16, which illustrates the constancy of average void size during annealing up to 900 °C, along with the fact that annealing causes the maximum diameter to decrease and the minimum to increase. It is therefore reasonable to hypothesize that the gas atoms stabilize the cavities in nickel which has been irradiated heavily with neutrons.

Rather conclusive evidence for the presence of gas in the irradiated samples is offered by the microstructure shown in Figure 4.11. Since the amount of gas measured experimentally is consistent with that which could be produced by the $(n,\alpha)$ reaction in nickel, it is very likely that the gas is helium. We therefore conclude that the "voids" present in the irradiated nickel of this study are not true voids in the sense that they contain only vacancies, but that in fact the voids are partially gas-filled.

This conclusion would also explain the difference in annealing behavior between the nickel used in this study and that used by Brimhall and Mastel. Our samples contain $\sim$30 times more neutronically produced helium which would tend to increase the temperature stability of the cavities.
We should point out that the present work does not necessarily support the hypothesis that voids are nucleated by gas atoms. It is not possible at this time to determine whether the gas atoms entered the void after it formed or if they indeed nucleated the void.

Grain Boundary Stabilization
G. L. Guthrie and T. K. Bierlein

The purpose of this program is to study the recrystallization and grain growth characteristics of metals containing small concentrations of inert gases. It is hoped that grain boundary pinning can be achieved by small concentrations of inert gases at the boundaries. If the grain size is small initially and remains small due to boundary pinning, the ready proximity of boundary regions may provide convenient sinks for vacancies, thereby depressing the vacancy concentration during irradiation and resulting in reduced swelling.

Work has continued on the previously reported helium-ion-bombarded powder compacted by the Dynapack process. Some of the ion-bombarded Dynapacked samples have been included in a capsule which is to be irradiated in the EBR-II reactor. Meanwhile, out-of-reactor annealing studies of helium-injected Dynapacked nickel powder samples have continued, and helium gas content has been determined for several classes of specimens, including:

(1) Dynapacked specimens exhibiting grain size stabilization under annealing.

(2) Dynapacked specimens exhibiting a lack of grain size stabilization when annealed.

(3) The injected but not-yet-compacted powders used to make the Dynapacked samples of (1) and (2).

From these studies, it appears that approximately $8 \times 10^{-4}$ atm-cm$^3$ of retained helium per gram of compacted powder results in a
stabilized grain size when 4 to 7 μ powder is used as a starting material. Such compacts resisted grain growth when annealed at temperatures up to 850 °C. Finished compacts of this helium density have resulted from Dynapacking of powders at 700 °C where the starting powders contained about $2.2 \times 10^{-3}$ atm-cm$^3$ of the helium per gram of nickel powder. Noticeably lower starting concentrations (approximate factors of 2) have resulted in incomplete or even negligible stabilization at the elevated temperatures of the anneals.

The gas quantities which appear necessary to achieve stabilization are low enough to be encouraging in regard to retention of reasonable mechanical properties. The atom fraction of injected gas is of the order of 0.3 ppm on a volume basis and is of the order of one atom in 15 at the grain boundaries.

Studies of retained gas in the ion-bombarded powder as a function of injection parameters have led to the conclusion that helium pressures of less than $7.5 \times 10^{-2}$ Torr during ion-bombardment result in unreasonably long bombardment times to reach satisfactory injection quantities. Helium pressures in the range from 125 μ to 375 μ all appear satisfactory. Present evidence indicates a noncritical optimization at about 250 μ.

Total helium injection appears to be approximately proportional to injection current density for a fixed bombardment time. Increased injection is also noted at increased time, but the relationship does not appear to be directly proportional to the first power of the bombardment time. This may be due in part to recoil ejection of helium already imbedded earlier in the run.

With the present apparatus, only a small amount of the powder is exposed to the ion stream at any given time, and some of the current goes directly to the stirring mechanism. Under these circumstances, exposures of 0.4 mA for 1 hr at
250 μ have been adequate to achieve gas quantities typical of stabilization in powder loads of the order of 25 g total weight. However, it is anticipated that this exposure could be significantly decreased in a redesigned apparatus as the coulomb charge associated with the known required gas implantation is considerably less than the ampere-second quantity indicated by the bombardment-current signal.

In the helium gas release studies, release rate has been plotted as a function of time under conditions of linearly increasing temperature. This allows a qualitative measure of the binding energy of the imbedded gas or the depth of penetration of the gas. It is found that decreasing the gas pressure during bombardment and increasing the ampere setting of the bombarding apparatus both result in a delayed or higher temperature gas release, indicating deeper penetration of the helium in the imbedding process. Presumably, both correlations result from an increase in the potential difference between the powder surface and the point in space where the injected ion suffered its last inelastic gas collision prior to colliding with the metal particle.

IRRADIATION EFFECTS ON THE FRACTURE OF HEAVY SECTION PRESSURE VESSEL STEELS
C. W. Hunter and J. A. Williams

Specimen Irradiations

Specimen irradiations, including one in.-thick compact tension (1T CT) and tensile specimens, were conducted during Cycles 101 through 104 in the M-3 loop facility of the ETR reactor. The M-3 facility is capable of producing specimens irradiated to 1 to $2 \times 10^{19}$ n/cm$^2$ (E > 1 MeV) at approximately 540 °F in one reactor cycle. The irradiation capsule was designed so that removal and replacement and positioning of specimens could be accomplished during reactor outages. The
The capsule is also adaptable for use in irradiating other types of specimens. The total irradiations completed to date include 12 CT specimens of base material at approximately 1.5 to $2.5 \times 10^{19}$ n/cm$^2$ (E $>$ 1 MeV), 12 CT specimens of weld and HAZ to a fluence of approximately 1.5 to $2.5 \times 10^{19}$ (E $>$ 1 MeV), and 4 CT specimens of base material at 7 to $8 \times 10^{19}$ (E $>$ 1 MeV). A total of 64 tensile specimens have been irradiated to levels from 1.5 to $8 \times 10^{19}$ n/cm$^2$ (E $>$ 1 MeV).

At the completion of Cycle 104, the G-7 loop facility was removed from the ETR. This was of major significance to operation of the M-3 facility, since, through the use of heat exchangers, the in-pile G-7 hot water loop supplied the necessary heat input for maintaining temperature in the M-3 facility. Prior to removal of the G-7 tube, plans were initiated to install an electrical heating system in the M-3 loop facility for maintenance of test temperature. With the electrical heating system temperatures other than the current operating temperatures (500 to 510 °F) will be obtainable. To date, all designs have been completed, components ordered and installation is scheduled for Cycle 107 of the ETR during April.

The temperature distribution along the M-3 capsule for Cycle 101 has been previously described. In Cycle 102 and beyond, the inlet water temperature was increased 5 to 10 °F. Considering this change as well as the axial temperature distribution along the capsule, the tensile specimen temperature range has been 500 to 510 °F and CT specimen range has been 530 to 545 °F, with the majority of the specimens in the upper portion of the respective ranges.

The M-3 capsule cycle flux monitors for Cycles 101 through 104 have been analyzed and are plotted in Figure 4.17. The flux monitors are iron, titanium and aluminum-cobalt. Additional values will be obtained and the fluence values refined as the analysis of flux monitors located in CT specimen holes is
FIGURE 4.17. Fast Flux Distribution Over the Length of the ETR, M-3 Loop, HSST Specimen Irradiation Assembly for Reactor Cycles 101 Through 104
completed. The cycle flux monitors occupied the same position during Cycles 101 through 103, near the front of the capsule with respect to reactor center. During Cycle 104 the monitor was exchanged with a tensile specimen tube near the rear of the capsule, approximately 1-1/2 in. radial distance from the position of the previous cycles. Comparisons of flux data obtained by this method do not yield an interpretation of the radial flux gradient as was initially expected. Peak flux of Cycles 101 through 103 shown in Figure 4.17 varied a maximum of about minus 20% from the largest value. Based on other reactor data it was estimated that a decrease of as much as 30% may be expected between the monitor position of Cycle 104 and Cycles 101 through 103, which would have placed the flux values during Cycle 104 at least as low as the lowest values obtained during the previous cycles. The flux values obtained in the 104 position were, however, higher than those previously obtained.

The experimental gamma heat survey of candidate positions for irradiating a large specimen capsule in the ETR has been completed. The survey was prompted by the uncertainty of gamma heat data, a factor of major importance in the capsule design. For the farthest position from the core available the gamma heat rate would be 0.9 W/g at the front surface and 0.3 W/g at the back surface of a 4-in. thick specimen. Based upon earlier heat transfer calculations (26) this gamma heat rate would result in a temperature differential of approximately 100 °F in the specimen. In order to maintain 550 °F at the specimen center with this high gamma heat rate, a gas gap of only 0.002 in. would be required. These results and evaluations demonstrated that a lower value of gamma heat, such as that in the ATR, would be preferable. In the ATR, reflector positions are available with gamma heat rates of 0.3 to 0.1 W/g front to rear respectively through a 4-in. specimen as shown in Figure 4.18. Under these conditions the temperature differential through the specimen will be about 30 °F.
FIGURE 4.18. Gamma Heat Distribution for Positions to Be Occupied by the ATR-4T Irradiation Facility. Curves Numbered 3, 4, 5, 6, 7 Are at the Front Plane and 8, 9, 10, 11, 12 Are at the Back Plane of the Fracture Specimens Shown in Figure 4.19.
Estimates of the flux in the candidate positions of the ATR have been made and flux measurements are in progress. The highest estimate of fast flux is $2 \times 10^{12}$ n/cm$^2$/sec $(E > 1$ MeV) based on theoretical attenuation from a known position. An estimate based on the ratio of fast flux to gamma heat also yields $2 \times 10^{12}$ n/cm$^2$/sec $(E > 1$ MeV) at the center of the $4T$ capsule position. Based on these flux values, two reactor cycles with rotation of specimens between cycles would yield a specimen exposure of $1.2 \times 10^{19}$ n/cm$^2$ $(E > 1$ MeV). Ten cycles with rotation after five cycles will yield a fluence of $6 \times 10^{19}$ n/cm$^2$ $(E > 1$ MeV). The proposed ATR-4T irradiation capsule is shown in Figure 4.19. With this arrangement of two capsules with two specimens each the following irradiations could be produced:

- 2 base plate, WR orientation, $6 \times 10^{19}$ n/cm$^2$ $(E > 1$ MeV).
- 4 base plate, WR orientation, $1.2 \times 10^{19}$ n/cm$^2$ $(E > 1$ MeV).
- 4 base plate, RW orientation, $1.2 \times 10^{19}$ n/cm$^2$ $(E > 1$ MeV).
- 2 weld metal, $1.2 \times 10^{19}$ n/cm$^2$ $(E > 1$ MeV).

Irradiations 2, 3 and 4 would run concurrently with irradiation 1. The lowest estimate of fast flux, obtained from the ratio of fast to thermal flux, is approximately $9 \times 10^{11}$ n/cm$^2$/sec $(E > 1$ MeV). Irradiations at this level would require twice as many cycles to attain the similar goal exposures discussed above.

The ATR-4T irradiation facility in Figure 4.19 will replace ten reflector position filler pieces. The capsule support structure will be approximately 9.5-in. $\times$ 22.5-in. $\times$ 57-in. long. Two capsules will be irradiated concurrently with each capsule containing two $4T$ fracture specimens, and the specimen grip holes and notches will be filled with tensile specimens, charpy specimens, flux monitors, and thermal monitors. The capsules
FIGURE 4.19. Schematic of ATR-4T Irradiation Facility
will be instrumented with thermocouples. Temperature control will be obtained by regulation of gas mixture supplied to the capsule.

**Base Material Studies: Annealing and Fluence Effects**

A cursory examination was conducted of the recovery of irradiation damage of ASTM A533 B tensile properties. The objective of this study was to determine the temperature range in which significant recovery occurred and the extent of recovery which could be expected. As previously reported herein, the irradiation temperature of tensile specimens in the M-3 loop is between 500 and 510 °F while maximum temperatures at the center of the 1T CT specimens was between 530 and 545 °F. It was therefore desirable to determine if tensile properties at the lower irradiation temperature were similar to those which would be obtained at the higher temperature.

Tensile specimens irradiated to a higher fluence, \( \sim 4.3 \times 10^{19} \) n/cm\(^2\) (\( E > 1 \) MeV), were also tested and compared to the specimens which were irradiated at approximately \( 2 \times 10^{19} \) n/cm\(^2\) (\( E > 1 \) MeV). These will be supplemented with future tests to determine the effect of fluences to \( 8 \times 10^{19} \) n/cm\(^2\) (\( E > 1 \) MeV) on tensile and fracture properties.

The results of these studies are presented in Table 4.5 and Figures 4.20, 4.21 and 4.22. Tensile specimens irradiated to 1.7 to 2.4 \( \times 10^{19} \) n/cm\(^2\) (\( E > 1 \) MeV) at a temperature between 500 and 510 °F were annealed at 540, 600, 650 and 725 °F for 130 hr. The tensile specimens annealed at 725 °F had been previously annealed at 650 °F for 130 hr. The specimens were tested in a range from room temperature to 500 °F. As shown in Figure 4.20, approximately 70% of the change in room temperature yield strength due to irradiation was recovered after annealing at 650 °F. Increasing the annealing temperature to 725 °F did not yield any significant
<table>
<thead>
<tr>
<th>Specimen Identification</th>
<th>Test Temperature, °F</th>
<th>Test Temperature, °C</th>
<th>Plate(^{(a)}) Position</th>
<th>Fluence, (10^{19} \text{n/cm}^2 (E &gt; 1 \text{MeV}))</th>
<th>Yield Strength, 0.2% Offset (10^3 \text{psi})</th>
<th>Ultimate Strength (10^3 \text{psi})</th>
<th>Annealing (^{(b)}) Temperature, °F</th>
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</thead>
<tbody>
<tr>
<td>02GA 156</td>
<td>90</td>
<td>32</td>
<td>5/19</td>
<td>2.26</td>
<td>93.9</td>
<td>110.6</td>
<td>540</td>
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<tr>
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<td>260</td>
<td>5/19</td>
<td>2.40</td>
<td>82.3</td>
<td>104.4</td>
<td>540</td>
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<tr>
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<td>90</td>
<td>32</td>
<td>9/19</td>
<td>1.8</td>
<td>80.2</td>
<td>98.7</td>
<td>600</td>
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<tr>
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<td>250</td>
<td>121</td>
<td>9/19</td>
<td>1.9</td>
<td>76.0</td>
<td>94.1</td>
<td>600</td>
</tr>
<tr>
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<td>500</td>
<td>260</td>
<td>9/19</td>
<td>1.97</td>
<td>70.7</td>
<td>94.9</td>
<td>600</td>
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<td>121</td>
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<td>88.2</td>
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<td>9/19</td>
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<td>650</td>
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<td>02GA 186</td>
<td>84</td>
<td>29</td>
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<td>75.7</td>
<td>97.1</td>
<td>725(^{(c)})</td>
</tr>
<tr>
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<td>560</td>
<td>260</td>
<td>7/19</td>
<td>2.40</td>
<td>67.5</td>
<td>92.4</td>
<td>725(^{(c)})</td>
</tr>
<tr>
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<td>-100</td>
<td>-73</td>
<td>7/19</td>
<td>4.31</td>
<td>118.1</td>
<td>130.2</td>
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<td>118.1</td>
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</tr>
<tr>
<td>02GA 168</td>
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<td>6/19</td>
<td>4.31</td>
<td>96.4</td>
<td>112.7</td>
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</tr>
<tr>
<td>02GA 191</td>
<td>500</td>
<td>260</td>
<td>7/19</td>
<td>4.41</td>
<td>86.3</td>
<td>107.2</td>
<td>---</td>
</tr>
</tbody>
</table>

\(^{a}\) Fraction of thickness at specimen center line.

\(^{b}\) Annealing time, 180 hr.

\(^{c}\) Re-annealed after annealing 130 hr at 850 °F.
Figure 4.20. Recovery of Room Temperature Yield Strength During Postirradiation Annealing for 130 hr. ASTM A533-B Base Material from HSST Program Plate 02.
FIGURE 4.21. Yield Strength of ASTM A533-B HSST Program Plate 02 at Two Fluence Levels and After Postirradiation Anneal
FIGURE 4.22. Ultimate Tensile Strength of ASTM A533-B HSST Program Plate 02 at Two Fluence Levels and After Postirradiation Anneal
additional recovery over the 650 °F anneal. No significant recovery of irradiated properties was obtained below 550 °F. The results of Figure 4.20 are typical of both the yield strength and the ultimate strength over the temperature to 500 °F, as demonstrated by Figures 4.21 and 4.22.

If indeed the change in yield strength is a measure of the change in fracture properties, then one could expect significant recovery of fracture properties at annealing temperatures as low as 600 °F. Similarly, it may be expected that appreciably less degradation in toughness will occur during irradiation at 600 °F. The fact that as much residual damage exists after annealing at 725 °F as at 650 °F suggests that much higher temperatures would be required to recover all the irradiation damage, if such an event is possible at all in the range of practical interest. Studies at NRL reveal that Charpy-V notch ductility of 6-in., A302-B steel was observed to exhibit approximately 70% recovery after annealing at 800 °F for 168 hr. Varying degrees of recovery of notch ductility for a number of materials and conditions are reported elsewhere. (27, 28, 29)

The lack of any observed recovery of tensile properties at 540 °F lend confidence to the results of the 500 to 510 °F tensile irradiations being closely representative of tensile properties which might be obtained when irradiating at 540 °F. If any significant difference were to be expected at the 540 °F irradiation temperature, then one would also expect a significant change as a result of postirradiation heat treatment at the 540 °F temperature.

The tensile specimens irradiated to a fluence of 4.3 to $4.4 \times 10^{19}$ n/cm$^2$ (E > 1 MeV) were tested from -100 °F to 500 °F. Test results showed an approximately 6 to 7% increase in room temperature ultimate and yield strength over those properties observed after irradiation to 1.7 to $2.4 \times 10^{19}$ n/cm$^2$ (E > 1 MeV).
Fatigue Crack Preparation in CT Specimens

In linear elastic fracture toughness determination, the critical stress intensity ($K_{IC}$) is that for fracture initiation from a sharp fatigue crack. Hence, the preparation of a fatigue crack in the fracture specimens is a necessary preliminary to actual toughness determinations. Consideration of the loading conditions during such fatigue crack preparation provides an opportunity to extract some information on fatigue crack behavior. To be sure, these loading conditions were not intended to provide pure propagation data, such as the crack growth rate per cycle (da/dN) versus cyclic stress intensity ($\Delta K$) relationship developed by Paris. (30) Nevertheless, this information does offer some examples of and insight into the practical situations of crack initiation, stalling, and re-initiation, (incubation) as well as propagation.

The specimens were IT CT specimens. The materials were the same 12-in. thick, A533-B HSST program plate and subarc weldment used throughout this investigation. The base material specimens were RW orientation from quarter through center positions. The fracture planes of the weldment specimens were parallel to the weld and located at positions A, B, and C throughout the weldment. As previously described, (26) A is the center of the weld metal, B is the weld dilution region (on the weld side of the fusion line), and C is the austenitic grain growth region in the HAZ (on the base metal side of the fusion line). All load cycling was from zero to tension to provide the intended $\Delta K$ and was performed at 1800 cycles/min in air at room temperature.

The fatigue cracks were initiated from a machined notch of 0.002 to 0.005 in. radius and subsequently grown a total of 0.200 in. in several steps at different levels of $\Delta K$. Three different patterns of stress intensity were employed; the various steps in each of these three patterns are listed.
in Table 4.6. The crack lengths for each step in Table 4.6 are the distance from the point of load application to the crack tip in the specimens. All three patterns were used in the fatiguing of the base material specimens. Only Pattern III was employed with the weldment specimens. With Patterns I and II, the $\Delta K$ was substantially reduced in Step 2 after crack initiation in Step 1. Typically, this reduction greatly reduced the propagation rate and in some instances stalled it. In the specimens of stalled propagation, the higher $\Delta K$ of Step 3, Pattern II was employed to re-initiate propagation. After re-initiation, Steps 4 and 5 of Pattern II were similar to Steps 2 and 3 of Pattern I. Therefore, Patterns I and II were the same except that Pattern II incorporated a step to re-initiate the stalled cracks which sometimes occurred during Step 2. Stalling was not prevalent in Pattern III because the $\Delta K$ decrease from Step 1 to 2 was not so large.

TABLE 4.6. Basic Stress Intensity and Propagation Patterns Employed in the Fatigue Crack Preparation of 1T CT Specimens of A533-B Plate and Weld

<table>
<thead>
<tr>
<th>Steps</th>
<th>Pattern I</th>
<th>Pattern II</th>
<th>Pattern III</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 (from machined notch)</td>
<td>26 ksi/\text{in.}</td>
<td>26 ksi/\text{in.}</td>
<td>26 ksi/\text{in.}</td>
</tr>
<tr>
<td>2</td>
<td>0.78 to 0.80 in.</td>
<td>0.78 to 0.80 in.</td>
<td>0.78 to 0.80 in.</td>
</tr>
<tr>
<td></td>
<td>17.5 to 20</td>
<td>17.5</td>
<td>21 to 24</td>
</tr>
<tr>
<td></td>
<td>0.80 to 0.89 in.</td>
<td>crack propagation stalled at 0.80 in.</td>
<td>0.80 to 0.90 in.</td>
</tr>
<tr>
<td>3</td>
<td>16.5 to 19</td>
<td>21 to 22</td>
<td>17 to 19</td>
</tr>
<tr>
<td></td>
<td>0.89 to 0.98 in.</td>
<td>crack propagation restarted to 0.84 in.</td>
<td>0.90 to 0.97 in.</td>
</tr>
<tr>
<td>4</td>
<td>18 to 20</td>
<td>13</td>
<td>0.97 to 0.99 in.</td>
</tr>
<tr>
<td>5</td>
<td>0.84 to 0.89 in.</td>
<td>16.5 to 19</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.89 to 0.98 in.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The corresponding values of effective crack growth rate and ΔK for each step are plotted in Figure 4.23 for the base material specimens and in Figure 4.24 for the weldment specimens. It must be emphasized that these values of crack extension rate per fatigue cycle include periods when the crack was initiating, stalled, and re-initiating as well as pure propagation. The typical relationship between ΔK and pure crack propagation is indicated in Figures 4.23 and 4.24.

The extent by which the data in Figures 4.23 and 4.24 fall below the band for typical propagation is a measure of the amount of hindrance provided by initiation, etc. The crack initiation in Step 1 from a machined notch is hindered a reasonable amount. The large ΔK decrease between Steps 1 and 2 (26 to 17.5 ksi√in.) in Patterns I and II always hindered extension. The ΔK decrease from Step 1 to 2 in Pattern III (26 to 21 ksi√in.) effected far less hindrance. Steps 3 in Patterns I and II and Step 5 in Pattern II were not appreciably hindered. With the base material specimens, Step 4 of Pattern III was not hindered, even though it involved a ΔK decrease from 19 to 13 ksi√in. Apparently, both a ΔK decrease and a fairly high prior value of ΔK are requisites for re-initiation hindrance.

The behavior of the weldment specimens during Step 4 (13 ksi√in.) is quite varied. The specimens near the fusion line (areas B and C) exhibited cracking rates approaching typical propagation behavior. However, the cracking in the center of the weld metal (area A) was markedly hindered. A possible explanation for the hindered cracking in the weld metal may be the presence of rounded microinclusions which blunt the fine crack tip.

The direction which a crack wanders in the fusion region is also of potential significance. Table 4.7 lists the positions of the original machined notch and final crack tip in specimens near the fusion line (areas B and C). The distance,
Neg 700453-4

**FIGURE 4.23.** Effective Fatigue Crack Extension Behavior in 1T CT Specimens Subjected to Different Patterns of Cyclic Stress Intensity. ASTM A533-B Base Material from HSST Program Plate 02.
FIGURE 4.24. Effective Fatigue Crack Extension Behavior in 1T CT Specimens in Weld Metal and near the Fusion Line. ASTM A533-B Submerged Arc Weldment from HSST Program Weldment 51A.
in mils, into either area B or C for both sides of the cracks are indicated. Based on these results, neither B or C areas offer a preferential path for fatigue cracks.

**TABLE 4.7. Position of the Machined Notch and Fatigue Crack on the Two Sides of the CT Specimens with Respect to the Fusion Line**

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Machined Notch Region</th>
<th>Fatigue Crack Root Region</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>B</td>
<td>C</td>
</tr>
<tr>
<td></td>
<td>Weld-Dilution</td>
<td>Region of γ Grain Growth</td>
</tr>
<tr>
<td>51A 5021</td>
<td>5,10</td>
<td></td>
</tr>
<tr>
<td>51A 5022</td>
<td>1</td>
<td>20</td>
</tr>
<tr>
<td>51A 5023</td>
<td>10,20</td>
<td></td>
</tr>
<tr>
<td>51A 5024</td>
<td>1</td>
<td>10</td>
</tr>
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<td>51A 5025</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>51A 5040</td>
<td>15,40</td>
<td></td>
</tr>
</tbody>
</table>

**FUEL SUBASSEMBLY MATERIALS**

T. T. Claudson

**EBR-II Irradiations**

P. K. McDaniel

The EBR-II irradiations program is to provide coordination and direction for the timely accomplishments of fast reactor irradiations on structural materials.

Structural material irradiation experiments are in various stages of design, construction and irradiation in EBR-II. Several changes have been made in scheduled EBR-II experiments. The schedule for the FFTF prototypical materials subassembly and control rod swelling materials experiment (Pins BNW B41, B42 and B43) originally scheduled to be inserted in EBR-II by February 1, 1970, has been changed. Two pins of the original 3-pin experiment (Pins BNW B41 and B42) have been combined with BNW Subassembly 5 (metallurgical variables swelling experiment). Pin BNW B43 which is to contain the tantalum control rod alloys for swelling studies experiment is still pending final AEC approval.
Pin BNW 24, the third in a series of five swelling experiments directed at defining effects of cold work, fluence and temperature on Types 304 and 316 SS, Inconel 600 and Nickel 200 in Subassembly X021 will be removed at the end of run 40 after accumulating about $3 \times 10^{22} \text{ n/cm}^2 (E > 0.1 \text{ MeV})$. An identical pin, BNW 25, will replace BNW 24 for an exposure of about $1 \times 10^{22} \text{ n/cm}^2 (E > 0.1 \text{ MeV})$.

An updated summary of experiments in EBR-II is given in Table 4.8. The accumulative exposure values in the past have been total exposure to the center of the subassembly. These values are now given as accumulative exposure based on fluence for neutrons having energies greater than 0.1 MeV. The new fluence estimates are based on unfolded spectra from the run 31F dosimetry experiment.

**Damage Analysis**
J. L. Straalsund, J. F. Bates and H. R. Brager

The objectives of this effort are to establish the radiation-induced swelling characteristics of FFTF alloys, relate fast-reactor induced substructural 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. 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 Type 316 SS irradiated to $7.8 \times 10^{22} \text{ n/cm}^2$ total fluence. Volume changes of this magnitude, if not taken into account, are sufficient to cause distortion in reactor components.
**TABLE 4.8. Summary of Experiments in EBR-II**

<table>
<thead>
<tr>
<th>Subassembly Designation</th>
<th>Reactor Position</th>
<th>Date Charged</th>
<th>Estimated Discharge Date</th>
<th>Type of Specimens</th>
<th>Material</th>
<th>Accumulative(^{(a,b)})</th>
<th>Goal Exposure, n/cm(^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X018B</td>
<td>4E2</td>
<td>10-2-69</td>
<td>Oct. 70</td>
<td>Density</td>
<td>504, 316 SS</td>
<td>5.17 x 10(^{21})</td>
<td>3.0 x 10(^{22})</td>
</tr>
<tr>
<td>X020</td>
<td>6B5</td>
<td>1-13-67</td>
<td>March 70</td>
<td>28 Tensile 8 Uniaxial Creep</td>
<td>304 SS</td>
<td>3.3 x 10(^{22})</td>
<td>5.6 x 10(^{22})</td>
</tr>
<tr>
<td>X021B(^{(c)})</td>
<td>2D1</td>
<td>2-27-67</td>
<td>Dec. 70</td>
<td>501 Tensile or Uniaxial Creep 65 Biaxial Creep</td>
<td>304 SS, 316 SS, 348 SS, 321 SS(^{(d)}) Ni Base Alloys</td>
<td>5.47 x 10(^{22})</td>
<td>1 x 10(^{23})</td>
</tr>
<tr>
<td>Pin 24</td>
<td></td>
<td>March 70</td>
<td></td>
<td></td>
<td>Pin 24</td>
<td>2.68 x 10(^{22})</td>
<td>3 x 10(^{22})</td>
</tr>
<tr>
<td>X021</td>
<td>4B3</td>
<td>11-21-67</td>
<td>July 70</td>
<td>5 Tensile 3 Uniaxial Creep</td>
<td>304 SS</td>
<td>3.8 x 10(^{22})</td>
<td>5 x 10(^{22})</td>
</tr>
<tr>
<td>X041</td>
<td>7C4</td>
<td>7-24-68</td>
<td>June 70</td>
<td>713 Tensile or Uniaxial Creep 74 Biaxial Creep</td>
<td>304 SS, 316 SS, 321 SS, 348 SS</td>
<td>2.1 x 10(^{22})</td>
<td>3 x 10(^{22})</td>
</tr>
<tr>
<td>X057</td>
<td>2B1</td>
<td>2-24-69</td>
<td>March 71</td>
<td>577 Tensile or Uniaxial Creep 164 Biaxial Creep 58 Density</td>
<td>304 SS, 316 SS, Ni Base Alloys</td>
<td>2.68 x 10(^{22})</td>
<td>6 x 10(^{22})</td>
</tr>
</tbody>
</table>

\(^{a}\) Accumulative to end of run 89C.

\(^{b}\) Fluxes greater than 0.1 MeV.

\(^{c}\) Original X021 subassembly with two single pin interchanges. Pin 84 in now.

\(^{d}\) Variety of other alloys.
Maximum target fluences for FFTF are as high as $10^{24}$ n/cm$^2$. 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.

Density measurements have been completed on a pin (A4) which was used to contain tensile specimens for irradiation in Row VII (Subassembly X010 position 7F3) in the EBR-II. During irradiation the inside of the pin was exposed to helium and the outside to coolant sodium. The pin is made of mill annealed Type 304 SS. The results of the measurements are shown in Figure 4.25. In the figure, the density and estimated exposure are plotted as a function of distance from the midplane. The error bands represent the spread of 5 independent measurements for each point.

Two aspects of the results are different than had been expected:

- The maximum swelling was significantly less than would be obtained from the swelling equation$^{(32)}$ for solution-treated stainless steel. While the irradiation temperature is not very well known, it is certain that the temperature at the midplane was no less than the inlet temperature, 700 °F. Using 700 °F and a fluence of $3 \times 10^{22}$ n/cm$^2$ ($E > 0.1$ MeV) in the swelling equation gives a value of 0.5% compared to the observed value of about 0.3%.

- The gradient in swelling along the axial length appears to be much less pronounced than one would expect. For example, the temperature may be expected to increase somewhat as one proceeds from the bottom of the pin to the midplane, but
FIGURE 4.25. Irradiation Dose and Swelling Profile for Pin A4, Irradiated in Row VII in EBR-II
one may estimate a minimum expected change in swelling by assuming no change in temperature. Doing this and using the estimated fluences in Figure 4.25 with the fluence exponent of 1.7 in the swelling equation, one may calculate a value of 2.6 for the minimum expected ratio of swelling at the midplane to swelling 8 in. below the midplane. As may be seen from Figure 4.25, however, very little difference exists in the amount of swelling observed at the two positions.

The above features may possibly be due to a flux rate effect or perhaps a spectrum effect. It is noteworthy that this is the first set of data that we have obtained from Row VII in the EBR-II. For positions this far from the core center, the flux is much lower, and the energy spectrum is considerably different. However, the irradiation conditions and material characterization need to be defined more completely before any meaningful interpretations may be made.

Empirical equations are presently being developed to describe the effects of neutron exposure and irradiation temperature on void sizes and concentration in Types 304 and 316 SS. The purpose of this work is to develop a set of three self-consistent equations describing the void diameter, void concentration, and void volume, or swelling, over as wide a range of experimental conditions as possible. A consistent set of equations accounting for all of the observed trends will provide more confidence in extrapolating to the higher exposures expected in the FTR. The best fits obtained to date for the void concentration data developed at PNL are:

$$\rho = (3.31 \times 10^{-5})(\frac{\phi t}{10^{22}})^{0.90}(\rho^{7030}/T - 1.0). \quad (1)$$

$$\rho = (1.21 \times 10^{-5})(\frac{\phi t}{10^{22}})^{-0.45 + 0.0019T}(\rho^{7740}/T - 1.0) \quad (2)$$
where
\[
\phi_t = \text{n/cm}^2 \ (E > 0.1 \ \text{MeV})
\]
\[
\rho = \text{voids/cm}^3 \times 10^{-15}
\]
\[
T = \text{temperature, ^oK.}
\]

These equations are based on transmission microscopy results from 15 specimens that were irradiated over a temperature range from 370 to 620 °C and fluence from 0.4 to 5.9 \times 10^{22} \text{ n/cm}^2 \ (E > 0.1 \ \text{MeV}).

Because of the limited amount of data presently available and the rather large degree of uncertainty associated with the irradiation conditions, it is difficult to determine which equation provides the better fit with the data. The essential difference between the two equations is that Equation (1) indicates a power relationship between fluence and void concentration with a constant exponent of 0.9, whereas Equation (2) indicates the same relation except for a temperature dependent exponent which varies from about 0.76 at 370 °C to 1.26 at 620 °C. Both equations appear to predict "high" values at high irradiation temperatures indicating that the form of temperature dependence chosen for the intercept (\(\rho A/T-1\)) should be changed. Several equations have been developed which provide good fits to the void size data. Two examples are:

\[
d = 417 + 0.87T \quad (3)
\]

\[
d = 4150 \left(\frac{\phi_t}{10^{22}}\right)^{0.082} \rho^{-2202/T} \quad (4)
\]

While different in form, these equations predict very similar values over the range of conditions for which we have data. Equation (3) actually fits the data over the whole range of conditions slightly better than Equation (4) which was included here to emphasize the insensitivity of void size to neutron dose. The estimated standard deviation on the fluence
exponent in Equation (4) was 0.042, half as large as the exponent itself. This indicates that statistically, the fluence dependency of the void size data is almost insignificant.

This effect is shown in Figures 4.26 and 4.27. Figure 4.26 illustrates the structure observed in Type 304 SS after a dose of $7 \times 10^{21} \text{ n/cm}^2 (E > 0.1 \text{ MeV})$ at 565 °C, whereas Figure 4.27 illustrates the structure after $3 \times 10^{22} \text{ n/cm}^2 (E > 0.1 \text{ MeV})$ at 595 °C. Despite the large differences in fluence, approximately the same void size was observed. It is noteworthy, however, that for the low fluence, the voids are predominately located on precipitates. Extraction replicas have identified only the complex carbide $\text{M}_2\text{C}_6$. At present, this phenomenon has been observed only at high irradiation temperatures.

The low correlation between void size and fluence is an important feature which must be accounted for in a theoretical description of void formation and growth. It appears that the voids may be considered to nucleate and grow, in small regions independent of all other regions, to a size range which is limited only by irradiation temperature. The interstitial loops which are also formed during irradiation may play a major role in this limitation. The loops increase both in size and spacing with irradiation temperature as shown in Figures 4.28 and 4.29. Consequently, if one assumes that close proximity of a loop prevents growth of a void, the increased void size with increased irradiation temperature may be explained by the increasing loop spacing.

An important corollary to the above argument is that stainless steels with high stacking fault densities produced by thermomechanical treatment or aging should have improved swelling resistance. Austenitic stainless steels with these structures have been developed for this purpose at BNW. These materials were developed under Task A of the IRDM Program, but irradiation experiments designed to determine their swelling
FIGURE 4.26. Absorption Contrast Condition Showing Voids and Rod-Shaped Precipitates in Type 304 SS irradiated at 1050 °F to $0.7 \times 10^{22}$ n/cm$^2$ ($E > 0.1$ MeV)
FIGURE 4.27. Large Faulted Loops in Same Material as Shown in Figure 4.25, Except Oriented for Diffraction Contrast, $g = [T11]$, [W11] Zone Axis

Neg 8516-C

69,420X
NEG 8712-B2

FIGURE 4.28. Small Irregular Faulted Loops in Type 304 SS Irradiated at 700 °F to $0.4 \times 10^{22}$ n/cm$^2$ ($E > 0.1$ MeV). The Foil Is Oriented to the Diffraction Contrast for $g = [002]$, [110] Zone Axis

78,000X
FIGURE 4.29. Voids in Type 304 SS Irradiated at 1100 °F to $3.0 \times 10^{22}$ n/cm² ($E > 0.1$ MeV)
behavior have been initiated in a cooperative effort between the Cladding Evaluation Section and the Metallurgy Section of the Chemistry and Metallurgy Division.

One may use Equations (1) through (4) to calculate the void volume based on transmission electron microscopy. If this is done, and Equation (2) is used for the void concentration, a temperature-dependent fluence exponent for void volume is obtained, suggesting similar behavior should be observed in the bulk density change data. Close examination of the data indicates that such a trend may be present.

Mechanical Testing of Fuel Pin Cladding
R. L. Fish

The purpose of this program is to provide a basis for evaluating the combined effects of fuel cladding interaction, fluence, and operating temperature on the postirradiation burst and stress-rupture properties of fuel pin cladding.

Burst properties of fuel cladding sections from PNL-1 series fuel pin irradiations at 900 °F have been given in previous quarterly reports. The most important finding in these previously reported studies was a very low strength and ductility region above the reactor midplane. The low strength is thought to be due to grain boundary attack during testing of sensitized cladding. The nature of the corrodingant has not been identified.

Additional fueled cladding specimens prepared in a dry, inert atmosphere, but stored for about 10 months in hot cell air after puncture of cladding for fission gas analysis, have revealed intergranular cracks during leak testing. These specimens were from the upper fueled regions of PNL 1-16 and 1-18 pins.
The origin of the stresses thought to be necessary to cause this cracking is not known, but the crack orientation suggests the pins were bent during storage. The intergranular failure in PNL 1-16 and 1-18 occurred at ambient temperatures which demonstrates the boundary weakness effect is not confined to elevated temperatures.

The cause of the boundary weakness is very important to fast reactor technology. If the weakness is due to storage environment, then pin handling procedures must be improved in order to preserve data. Alternately, if the effect is inherent in fuel pin performance, then the cladding will not be capable of supporting the loads which appear safe from unfueled irradiation experiments.

In order to clarify the mechanism involved with the boundary weakness, a number of definitive experiments have been conducted and additional experiments are planned.

The electron microprobe has been used to examine grain boundaries in failed areas to determine the extent of average penetration by fission products.

Figure 4.30 illustrates the typical cladding integrity found at the fissures in the fueled specimens from above the reactor midplane and also shows the results of the electron microprobe examination performed on the same specimen from PNL 1-18. The scan across the grain boundary indicated in the micrograph revealed the presence of cesium. There was no fuel associated with this cesium as is the case along the inside diameter of the cladding. Cesium was found in one other grain boundary area in the cladding and was again unassociated with fuel. No definite conclusion can be drawn from these observations, however, because of the possibility of cesium penetration or deposition along the grain boundaries subsequent to specimen failure.
PNL 1-18
MICROPROBE RESULTS

CLADDING MICROSTRUCTURE AT FAILURE

MICROPROBE SCAN ACROSS GRAIN BOUNDARY INDICATED ABOVE

FIGURE 4.30. Electron Microprobe Results from Above Fuel Midplane in PNL 1-18
Initial results were obtained from a ring test, on a specimen cut from a biaxial test specimen which failed by grain boundary fracture prematurely in a biaxial test. In the ring test, a 1/4-in. long section of cladding is pulled in a tensile machine in such a way as to create tangential stress in the cladding wall. Fuel and fission products are removed prior to testing at 900 °F. The metallography on these ring test ruptures is shown in Figure 4.31. The good ductility and strength exhibited by this cladding with the fuel and fission products removed contrasts greatly with the very low ductility (<1.0% ΔD/D), intergranular failures observed at the same test temperature in fueled cladding from the same region of the fuel pin. This indicates that the premature failures are due to corrosive attack from within the fuel pin and are not a result of external attack or inherent cladding grain boundary weakness. Two burst tests (one fueled and one with fuel removed) are planned to verify the effects observed in these ring tests. Carbon extraction replicas are also being obtained from the ring test samples for further characterization of their microstructure.

Fuel pins PNL 1-9, 1-12, 1-13, and 1-14 were helium leak tested for possible fissures. These pins have not been punctured nor otherwise intentionally compromised. This testing did not reveal any fissures or leaks. The outside surface of the cladding on these same fuel pins was replicated with cellulose acetate plastic. This replication did not reveal any intergranular cracking or other cladding perforations. The only significant characteristic revealed by the replicas was a pronounced delineation of the grain boundaries in the upper fueled region of PNL 1-14 (Figure 4.32). This is believed to be heavy carbide precipitation and is not unexpected in view of the calculated temperatures (≈900 °F) of this cladding during irradiation.
(a) Irradiation Conditions: $\phi t = 3.7 \times 10^{21}$ n/cm$^2$ (E > 0.1 MeV) 
$T_{ave} = 945 ^\circ F$

(b) Irradiation Conditions: $\phi t = 2.8 \times 10^{21}$ n/cm$^2$ (E > 0.1 MeV) 
$T_{ave} = 940 ^\circ F$

**FIGURE 4.31. Microstructure of Ring Test Samples (Test Temp = 900 ^\circ F)**
FIGURE 4.32. Precipitate Delineation of Cladding Grain Boundaries in Upper Fueled Region of PNL 1-14

FAST REACTOR SUPPORTING STUDIES

Acceptance Testing and Clad Characterization

M. M. Paxton

The clad characterization program is being conducted to ensure that high quality stainless steel cladding is obtained and used throughout the FFTF fuels development and associated irradiation testing program. Under this program, selected mechanical properties are being determined and analyzed to evaluate fabrication variables and assist in vendor qualification.

The characterization of PNI tube lots E, F, G and H are essentially complete, except for a few long-term, stress-rupture tests. The characterization of PNI lot N-1, Carpenter mill release numbers 83628 and 83629, and PNI lot N-2, Carpenter
mill release number 83626, 83627, and 83631 has been initiated and is progressing satisfactorily. The current status of all PNL lots being characterized is contained in Tables 4.9 through 4.13.

A check analysis has been completed by Lukens Steel Company on PNL lots N-1 and N-2, Tables 4.14 and 4.15. This analysis is in agreement with that supplied by the vendor.

The volume of data being generated on each lot of tubing and its distribution to the individuals that use the data has shown the need for a data handbook for each lot of tubing. Such a handbook containing all available data on a particular lot of tubing is presently being assembled. The first distribution is expected about April 15, 1970.

Tensile Studies of Types 304 and 316 Stainless Steels
A. L. Ward

The objective of this program is to provide mechanical property and metallurgical stability data for candidate LMFBR vessel and core-structural materials in order to define adequate lifetime performance under anticipated service conditions.

The effect of fast reactor irradiation on the ductility of solution-annealed AISI Types 304 and 316 SS has been investigated. Ductility losses caused by low fluence irradiation (less than about $10^{22}$ n/cm$^2$) have been extensively studied in many metals. Usually the losses are described in terms of two temperature domains divided approximately by one-half the absolute melting temperature ($T_m/2 \approx 560$ °C). At irradiation and test temperatures below $T_m/2$, irradiation-produced point defect clusters increase the flow stress and lead to the early onset of plastic instability. Above $T_m/2$, displacement damage anneals rapidly and does not lead to irradiation hardening. However, at such temperatures, helium generated from various (n,α) reactions readily migrates to grain boundaries. Gas bubbles formed by agglomeration of helium atoms
### TABLE 4.9. Mechanical Properties PNL Tube Lot E, Mill-Annealed Type 304 SS, 0.250-in. OD x 0.218-in. ID

<table>
<thead>
<tr>
<th>Test Temp. °F</th>
<th>Burst Test Press. psi</th>
<th>Max Hoop Stress psi</th>
<th>%ΔD/D</th>
<th>0.2% Yield Strength psi</th>
<th>Ultimate Tensile Strength psi</th>
<th>Uniform Elong. %</th>
<th>Total Elong. %</th>
<th>Max Hoop Stress</th>
<th>Rupture Time, hr</th>
<th>ΔD/D, 1/hr</th>
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<tbody>
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<td>470</td>
<td>10,700</td>
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<td>-</td>
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<td>79,270</td>
<td>-</td>
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<td>51,500</td>
<td>95,110</td>
<td>40.6</td>
<td>61.7</td>
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<td>-</td>
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<td>41.1</td>
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<td>29,000</td>
<td>44.5</td>
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<tr>
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<td>1200</td>
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<td>45,950</td>
<td>0.01</td>
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<td>48,550</td>
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*Note: ΔD/D represents the change in diameter relative to the original diameter.*
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<th>Stress Rupture</th>
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TABLE 4.10. Mechanical Properties PNL Tube Lot F, Mill-Annealed Type 304 SS, 0.250-in. OD x 0.218-in. ID
### TABLE 4.11. Mechanical Properties PNL Lot G, Mill-Annealed Type 316 SS, 0.250-in. OD x 0.218-in. ID

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<td>Ultimate Tensile Strength, psi</td>
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### TABLE 4.12. Selected Mechanical Properties PNL Tube Lot H, Mill-Annealed Type 316 SS, 0.250-in. OD × 0.218-in. ID

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<th>%Δ/D</th>
<th>Tensile Test</th>
<th>Yield Strength, psi</th>
<th>Ultimate Tensile Strength, psi</th>
<th>Uniform Elong., %</th>
<th>Total Elong., %</th>
<th>Stress Rupture Max Hoop Stress, psi</th>
<th>Rupture Time, hr</th>
<th>%Δ/D</th>
<th>(ΔD/D), μ/hr</th>
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### TABLE 4.13. Selected Mechanical Properties on PNL Tube Lot N-1, MFG's Lot 83628 and 83629, 20% CW Type 316 SS, 0.280-in. OD × 0.200-in. ID

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<th>0.2% Yield Strength, psi</th>
<th>Ultimate Tensile Strength, psi</th>
<th>Uniform Elong., %</th>
<th>Total Elong., %</th>
<th>Stress Rupture Max Hoop Stress, psi</th>
<th>Rupture Time, hr</th>
<th>%ΔD/D</th>
<th>ΔD/D/hr</th>
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4.78
### TABLE 4.14. Chemical Analysis PNL Lot N-1, by Lukens Steel Company

<table>
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<th>Tube No.</th>
<th>N</th>
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<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Cb</th>
<th>Ti</th>
<th>Cu</th>
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<td>2.52</td>
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<td>0.011</td>
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<td>0.014</td>
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<td>0.49</td>
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<td>16.60</td>
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<td>0.05</td>
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### TABLE 4.15. Chemical Analysis PNL Lot N-2, by Lukens Steel Company

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<th>Tube No.</th>
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<th>C</th>
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<th>P</th>
<th>S</th>
<th>Si</th>
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<th>Cr</th>
<th>Mo</th>
<th>Cb</th>
<th>Ti</th>
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<td>26-162</td>
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<td>0.010</td>
<td>0.056</td>
<td>1.47</td>
<td>0.008</td>
<td>0.011</td>
<td>0.49</td>
<td>13.50</td>
<td>16.75</td>
<td>2.45</td>
<td>0.06</td>
<td>0.002</td>
<td>0.07</td>
<td>0.0019</td>
</tr>
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<td>27-113</td>
<td>0.006</td>
<td>0.008</td>
<td>0.054</td>
<td>1.47</td>
<td>0.005</td>
<td>0.011</td>
<td>0.49</td>
<td>13.36</td>
<td>16.79</td>
<td>2.50</td>
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<td>0.002</td>
<td>0.07</td>
<td>0.0025</td>
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<td>27-158</td>
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<td>0.005</td>
<td>0.053</td>
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<td>0.007</td>
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<td>16.82</td>
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<td>0.05</td>
<td>0.002</td>
<td>0.075</td>
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<td>0.005</td>
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<td>0.010</td>
<td>0.48</td>
<td>13.44</td>
<td>16.77</td>
<td>2.65</td>
<td>0.05</td>
<td>0.002</td>
<td>0.075</td>
<td>0.0040</td>
</tr>
<tr>
<td>31-100</td>
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<td>0.009</td>
<td>0.056</td>
<td>1.48</td>
<td>0.005</td>
<td>0.010</td>
<td>0.48</td>
<td>13.36</td>
<td>16.90</td>
<td>2.52</td>
<td>0.04</td>
<td>0.002</td>
<td>0.07</td>
<td>0.0025</td>
</tr>
</tbody>
</table>
result in weakened boundaries and subsequent premature failure. At high fluences \(10^{22} \text{n/cm}^2\) and above) these concepts must be modified. In this fluence region displacement damage in the form of dislocation loops and voids accumulates to levels which can cause appreciable strength increases at irradiation temperatures as high as \(0.7 T_m\). Thus, at high fluences, ductility loss may be controlled by the combined effects of helium embrittlement and displacement damage.

In order to clarify the roles of helium and displacement hardening, test results from irradiated specimens were compared with those from identical specimens annealed for 1 hr at 1100 °C, a temperature sufficient to removal all displacement damage. The very low solubility of helium in metals causes the helium embrittlement process to remain operative after annealing, so that the role helium plays in ductility loss is reflected in the tests conducted after annealing. Once the effects of helium are known, the relative roles of helium and displacement can be deduced.

Type 316 SS specimens were taken from a group irradiated in an experimental subassembly in the Experimental Breeder Reactor-II (EBR-II) to a peak fluence of \(2.7 \times 10^{22} \text{n/cm}^2\) \((E > 0.1 \text{ MeV})\) at temperatures ranging from 390 to 620 °C. Type 304 SS specimens were obtained from the EBR-II, 5C3 control rod thimble irradiated to a peak fluence of \(3.3 \times 10^{22} \text{n/cm}^2\) \((E > 0.1 \text{ MeV})\) at temperatures between 370 and 470 °C. These specimens contain helium concentrations ranging from 2 to 16 ppm (atomic).

Postirradiation annealing produces nearly complete restoration of preirradiation tensile properties in specimens tested below \(T_m/2\) (Table 4.16). In Type 304 SS specimens tested at 370 and 480 °C \((0.38 \text{ and } 0.45 T_m)\), the strength and flow properties returned to those typical of the unirradiated material, which indicates that the postirradiation
TABLE 4.16. Effect of Postirradiation Annealing at 1100 °C on the Tensile Properties of AISI Types 304 and 316 SS

<table>
<thead>
<tr>
<th>Condition</th>
<th>Irrad. Temp, °F</th>
<th>Test Temp, °F</th>
<th>Fluence, n/cm²(a)</th>
<th>Yield Stress, ksi</th>
<th>Ultimate Stress, ksi</th>
<th>Uniform Elong., %</th>
<th>Total Elong., %</th>
<th>Reduction of Area, %</th>
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<td></td>
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<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>As-Irradiated</td>
<td>400</td>
<td>370</td>
<td>$3.3 \times 10^{22}$</td>
<td>107.5</td>
<td>108.3</td>
<td>0.7</td>
<td>1.9</td>
<td>41.7</td>
</tr>
<tr>
<td>Annealed</td>
<td>400</td>
<td>370</td>
<td>$3.3 \times 10^{22}$</td>
<td>19.5</td>
<td>67.0</td>
<td>40.9</td>
<td>42.9</td>
<td>73.2</td>
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<td>370</td>
<td>---</td>
<td>19.2</td>
<td>62.3</td>
<td>35.0</td>
<td>50.0</td>
<td>62.5</td>
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<tr>
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<td>480</td>
<td>$3.3 \times 10^{22}$</td>
<td>95.8</td>
<td>96.0</td>
<td>0.8</td>
<td>1.7</td>
<td>29.9</td>
</tr>
<tr>
<td>Annealed</td>
<td>400</td>
<td>480</td>
<td>$3.3 \times 10^{22}$</td>
<td>16.8</td>
<td>61.2</td>
<td>42.1</td>
<td>43.4</td>
<td>72.6</td>
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<tr>
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<td>---</td>
<td>480</td>
<td>---</td>
<td>18.0</td>
<td>59.0</td>
<td>36.0</td>
<td>41.5</td>
<td>69.0</td>
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<tr>
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<td>56.3</td>
<td>0.4</td>
<td>0.5</td>
<td>7.9</td>
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<tr>
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<td>600</td>
<td>$3.3 \times 10^{22}$</td>
<td>16.1</td>
<td>38.6</td>
<td>13.8</td>
<td>15.1</td>
<td>43.7</td>
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<tr>
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<td>600</td>
<td>---</td>
<td>16.5</td>
<td>46.3</td>
<td>27.5</td>
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<tr>
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<td>$3.3 \times 10^{22}$</td>
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<td>0.2</td>
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<td>22.5</td>
<td>5.8</td>
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<tr>
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<td>74.6</td>
<td>22.9</td>
<td>27.1</td>
<td>61.4</td>
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<tr>
<td>Annealed</td>
<td>460</td>
<td>480</td>
<td>---</td>
<td>16.3</td>
<td>61.3</td>
<td>34.4</td>
<td>36.1</td>
<td>53.3</td>
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<tr>
<td>Un-Irradiated</td>
<td>---</td>
<td>480</td>
<td>---</td>
<td>18.0</td>
<td>59.0</td>
<td>36.0</td>
<td>41.5</td>
<td>69.0</td>
</tr>
<tr>
<td>As-Irradiated</td>
<td>460</td>
<td>600</td>
<td>$0.38 \times 10^{22}$</td>
<td>40.0</td>
<td>49.0</td>
<td>8.9</td>
<td>10.4</td>
<td>39.5</td>
</tr>
<tr>
<td>Annealed</td>
<td>460</td>
<td>600</td>
<td>$0.38 \times 10^{22}$</td>
<td>12.7</td>
<td>35.5</td>
<td>18.4</td>
<td>19.3</td>
<td>42.1</td>
</tr>
<tr>
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<td>---</td>
<td>600</td>
<td>---</td>
<td>16.5</td>
<td>46.3</td>
<td>27.5</td>
<td>33.2</td>
<td>46.0</td>
</tr>
<tr>
<td>As-Irradiated</td>
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<td>700</td>
<td>$0.38 \times 10^{22}$</td>
<td>26.7</td>
<td>30.1</td>
<td>5.0</td>
<td>7.9</td>
<td>16.4</td>
</tr>
<tr>
<td>Annealed</td>
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<td>700</td>
<td>$0.38 \times 10^{22}$</td>
<td>12.3</td>
<td>22.4</td>
<td>9.5</td>
<td>11.1</td>
<td>34.1</td>
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<tr>
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<td>700</td>
<td>---</td>
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<td>14.0</td>
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<td>46.9</td>
<td>2.0</td>
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<td>11.4</td>
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<tr>
<td>Annealed</td>
<td>580</td>
<td>650</td>
<td>$2.7 \times 10^{22}$</td>
<td>14.1</td>
<td>33.4</td>
<td>9.6</td>
<td>10.4</td>
<td>19.5</td>
</tr>
<tr>
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<td>---</td>
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<td>---</td>
<td>16.8</td>
<td>49.0</td>
<td>28.0</td>
<td>42.0</td>
<td>47.5</td>
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<tr>
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<td>515</td>
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<td>50.6</td>
<td>52.5</td>
<td>1.3</td>
<td>2.7</td>
<td>8.1</td>
</tr>
<tr>
<td>Annealed</td>
<td>515</td>
<td>650</td>
<td>$1.6 \times 10^{22}$</td>
<td>14.4</td>
<td>33.5</td>
<td>9.7</td>
<td>10.6</td>
<td>22.8</td>
</tr>
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<td>650</td>
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<td>51.5</td>
<td>0.8</td>
<td>2.5</td>
<td>13.8</td>
</tr>
<tr>
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<td>480</td>
<td>650</td>
<td>$1.1 \times 10^{22}$</td>
<td>14.1</td>
<td>34.2</td>
<td>10.5</td>
<td>11.4</td>
<td>21.1</td>
</tr>
<tr>
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<td>650</td>
<td>$0.9 \times 10^{22}$</td>
<td>42.4</td>
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<td>2.6</td>
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<td>21.1</td>
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<tr>
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<td>650</td>
<td>$0.9 \times 10^{22}$</td>
<td>13.9</td>
<td>35.0</td>
<td>11.7</td>
<td>12.6</td>
<td>18.7</td>
</tr>
</tbody>
</table>

a. Neutrons with energies greater than 0.1 MeV.
anneal eliminated all displacement damage. At these temperatures, the helium embrittlement process is not active, and no residual ductility losses are expected once displacement damage is removed. At test temperatures greater than $T_m/2$, the postannealing data presented here show a marked departure from low fluence results. At low fluences, ductility is not significantly improved after annealing because of the insolubility of the helium. In contrast, substantial increases in ductility are observed in the high fluence case (Table 4.16). However, a significant nonrecoverable ductility loss remains. The ductility improvement is attributed to the removal of dislocation loops and voids as a result of the annealing, while the nonrecoverable loss is attributed to the classical helium embrittlement process.

The magnitude of the nonrecoverable ductility loss is consistent with results obtained by Kramer et al.\(^{33,34}\) from studies in which helium concentrations from 0.1 to 30 ppm were introduced by cyclotron injection of alpha particles. Indirect evidence previously published by Holmes\(^{35}\) suggested that the helium embrittlement mechanism, acting independently of displacement damage, controls high-fluence, elevated-temperature ductility. The present findings show that in the range from 593 to 704 °C, both processes contribute to the total ductility loss and suggest that helium embrittlement dominates only at sufficiently high temperatures to eliminate the displacement damage.

**Uniaxial Creep-Rupture Studies of Types 304 and 316 Stainless Steel**

A. J. Lovell

The objectives of this program are to evaluate the effects of fast neutron irradiation and environment on the postirradiation 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.

The effects of fast neutron irradiation to $1.2 \times 10^{22} \text{ n/cm}^2$ (total) at $\approx 900^\circ \text{F} (\approx 480^\circ \text{C})$ on the postirradiation creep-rupture properties of Types 304, $^{(12)}$ 316, $^{(12)}$ and 348 $^{(36)}$ SS have been reported. The comparison of these three sets of results at a test temperature of 1000 $^\circ \text{F}$ (538 $^\circ \text{C}$) are shown in Figures 4.33 and 4.34. In the unirradiated condition, the three alloys have substantially different rupture life and minimum creep rates. After irradiation, the rupture life of all alloys is reduced, with the Types 348 and 316 SS materials exhibiting larger reductions than the Type 304 material. Minimum creep rates of the Types 348 and 316 alloys are increased as a result of irradiation. The end result is that these alloys, which were quite different prior to irradiation, now exhibit similar creep-rupture properties after irradiation.

Weldment Studies
A. L. Ward and A. J. Lovell

Postirradiation tensile test results are presented in Tables 4.17 and 4.18 for base metal (AISI Type 304 SS) and all-weld specimens representing the submerged-arc, TIG, MIG, and stick-electrode weld processes. The specimens included in this study were irradiated in Pins No. 35, 36, and 37 of Sub-assembly X067 in Row 4 of the EBR-II. Listed fluences are based on a peak of $8.75 \times 10^{21} \text{ n/cm}^2$ (total) and a fluence versus axial distance profile established by dosimeter activation measurements in another Row 4 irradiation experiment. The irradiation temperature for specimens represented in Table 4.17 was 800 $^\circ \text{F}$ and as indicated for those in Table 4.18. Reported temperatures are based on heat transfer calculations. Figures 4.35 and 4.36 show yield strength and total elongation, respectively, as functions of neutron fluence ($E > 0.1 \text{ MeV}$) for
Figure 4.33: Postirradiation ruptured properties of stainless steels. Irradiated to 1 x 10^22 n/cm^2 (total) at ~900 °F (482 °C). Tested at 1000 °F (538 °C).
Neg 701101-1

**FIGURE 4.34.** Postirradiation Creep Properties of Stainless Steels Irradiated to $1 \times 10^{22}$ n/cm$^2$ (total) at $\sim 900$ °F ($\sim 482$ °C), Tested at 1000 °F (538 °C)
<table>
<thead>
<tr>
<th>Specimen</th>
<th>Type</th>
<th>Test Temp., °F</th>
<th>$\sigma_t$ ($10^{21}$ n/cm$^2$)</th>
<th>Proportion of Elastic Limit, psi</th>
<th>Yield, psi</th>
<th>Ultimate, psi</th>
<th>Uniform Elong., %</th>
<th>Total Elong., %</th>
<th>Reduction of Area, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>35-T-B22A</td>
<td>Base</td>
<td>600</td>
<td>4.4</td>
<td>3.2</td>
<td>56,000</td>
<td>69,600</td>
<td>77,900</td>
<td>8</td>
<td>54.0</td>
</tr>
<tr>
<td>35-T-B25A</td>
<td>Base</td>
<td>700</td>
<td>4.4</td>
<td>3.2</td>
<td>51,200</td>
<td>69,600</td>
<td>77,900</td>
<td>8</td>
<td>54.0</td>
</tr>
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<td>3.2</td>
<td>50,000</td>
<td>67,500</td>
<td>70,800</td>
<td>8</td>
<td>54.0</td>
</tr>
<tr>
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<td>Base</td>
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<td>76,100</td>
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<td>MIG</td>
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<td>2.6</td>
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<td>3.8</td>
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<td>900</td>
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<td>2.6</td>
<td>44,700</td>
<td>59,100</td>
<td>77,600</td>
<td>8</td>
<td>54.0</td>
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</table>

**TABLE 4.17.** Tensile Properties of Weldment Materials Irradiated in the EBR-II at 800 °F
### TABLE 4.18. Tensile Properties of Weldment Materials Irradiated in the EBR-II

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Type</th>
<th>Test Temp., °F</th>
<th>$\sigma$ (10^2 n/cm^2) Total</th>
<th>Proportion of Elastic Limit, psi</th>
<th>Yield, psi</th>
<th>Ultimate, psi</th>
<th>Uniform Elong., %</th>
<th>Total Elong., %</th>
<th>Reduction of Area, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>35-T-B21A(a)</td>
<td>Base</td>
<td>600</td>
<td>1.3</td>
<td>0.61</td>
<td>15,800</td>
<td>21,700</td>
<td>65,000</td>
<td>45.0</td>
<td>52.6</td>
</tr>
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<td>36-T-B37A(a)</td>
<td>Base</td>
<td>700</td>
<td>1.3</td>
<td>0.61</td>
<td>13,700</td>
<td>23,700</td>
<td>64,500</td>
<td>45.2</td>
<td>52.7</td>
</tr>
<tr>
<td>35-T-B18A(b)</td>
<td>Base</td>
<td>800</td>
<td>1.3</td>
<td>0.61</td>
<td>10,000</td>
<td>18,500</td>
<td>64,600</td>
<td>44.8</td>
<td>51.9</td>
</tr>
<tr>
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<td>Base</td>
<td>900</td>
<td>1.3</td>
<td>0.61</td>
<td>12,000</td>
<td>20,100</td>
<td>61,000</td>
<td>37.0</td>
<td>44.0</td>
</tr>
<tr>
<td>35-T-W9(a)</td>
<td>TIG</td>
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<td>1.1</td>
<td>0.54</td>
<td>45,500</td>
<td>58,300</td>
<td>71,800</td>
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<td>16.5</td>
</tr>
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<td>0.54</td>
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<td>56,800</td>
<td>71,300</td>
<td>10.7</td>
<td>16.5</td>
</tr>
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<td>35-T-W6(a)</td>
<td>TIG</td>
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<td>0.54</td>
<td>36,600</td>
<td>51,300</td>
<td>67,800</td>
<td>12.8</td>
<td>19.2</td>
</tr>
<tr>
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<td>TIG</td>
<td>900</td>
<td>1.1</td>
<td>0.54</td>
<td>59,000</td>
<td>53,900</td>
<td>68,500</td>
<td>11.8</td>
<td>17.3</td>
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<tr>
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<td>Sub-Arc</td>
<td>600</td>
<td>1.5</td>
<td>0.75</td>
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<td>43,100</td>
<td>73,000</td>
<td>19.9</td>
<td>23.0</td>
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<tr>
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<td>Sub-Arc</td>
<td>700</td>
<td>1.1</td>
<td>0.54</td>
<td>28,500</td>
<td>41,500</td>
<td>70,500</td>
<td>18.7</td>
<td>19.1</td>
</tr>
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<td>66,100</td>
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<td>Sub-Arc</td>
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<td>1.1</td>
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<td>39,100</td>
<td>67,600</td>
<td>22.1</td>
<td>23.8</td>
</tr>
<tr>
<td>35-SE1-W17(a)</td>
<td>St Elect</td>
<td>600</td>
<td>1.1</td>
<td>0.54</td>
<td>43,000</td>
<td>49,800</td>
<td>71,800</td>
<td>18.5</td>
<td>23.7</td>
</tr>
<tr>
<td>35-M1-W15(a)</td>
<td>MIG</td>
<td>600</td>
<td>1.1</td>
<td>0.54</td>
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<td>42,600</td>
<td>66,700</td>
<td>21.6</td>
<td>27.0</td>
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</tbody>
</table>

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*a. Irradiation Temperature 700 °F*

*b. Irradiation Temperature 800 °F*
Neg 700190-1

**FIGURE 4.35.** Yield Strength of Fast Reactor Irradiated AISI Type 304 SS as a Function of Neutron Fluence (E > 0.1 MeV)
Neg 700190-2

FIGURE 4.36. Tensile Ductility of Fast Reactor Irradiated AISI Type 304 SS as a Function of Neutron Fluence (E > 0.1 MeV)
Type 304 SS specimens from several EBR-II irradiation experiments (open symbols). These plots represent data selected on the basis of:

- Test temperatures in the range from 700 to 1000 °F
- Fluences from $6 \times 10^{20}$ to $3.7 \times 10^{22}$ n/cm$^2$ ($E > 0.1$ MeV)
- Irradiation temperatures from 700 to 900 °F.

Results from the weldment study (closed symbols) represent test temperatures of 600, 700, 800, and 900 °F.

The weld-deposited materials show smaller irradiation-induced yield strength increases and smaller ductility losses than the base metal. Note, however, that the preirradiation yield values were higher and the ductilities lower in the weld material. At fluences of 5.4 and $6.1 \times 10^{20}$ n/cm$^2$ ($E > 0.1$ MeV), properties are essentially unaffected by irradiation, while at $2.6 \times 10^{21}$ n/cm$^2$ yield stress increments of about 20,000 psi and significant reductions in total elongation are observed for all welds except the stick-electrode. Corresponding base metal specimens exhibited ~30,000 psi yield stress increases and 2 to 15% reductions in total elongations (Figures 4.35 and 4.36).

Tensile test results are reported from a study to determine whether a larger diameter specimen would improve the reproducibility of test results and also be more representative of the bulk weld material being studied. Table 4.19 lists the results from the larger specimens (0.250-in. gage diameter, 1.125-in. gage length) tested in the range from 600 to 900 °F. The data is in general agreement with previously reported results for the miniature (0.125-in. gage diameter, 1.25-in. gage length) specimen used in the weldment irradiation experiment. However, no consistent improvement in the magnitude of data scatter is observed upon comparison of the two sets of results. Therefore,
<table>
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<th>Specimen</th>
<th>Type</th>
<th>Proportion of Elastic Limit, psi</th>
<th>Yield, psi</th>
<th>Ultimate psi</th>
<th>Uniform Elong., %</th>
<th>Total Elong., %</th>
<th>Reduction of Area, %</th>
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<td>52,600</td>
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<td>19.5</td>
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<td>R.T.</td>
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<tr>
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<td>28,500</td>
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<td>65,600</td>
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<td>34,900</td>
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<td>67,400</td>
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<td>39,900</td>
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<td>13.5</td>
<td>19.5</td>
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</table>
present plans for future irradiation experiments involving weldment materials call for continued use of the miniature specimen.

Results from creep-rupture tests on weldment specimens irradiated to $\sim 8 \times 10^{21}$ n/cm$^2$ (total) at $\sim 1100$ °F are presented in Table 4.20. This irradiation had little effect on the rupture life of the submerged arc weldment. The TIG weldment material, on the other hand, showed substantial losses in rupture life after irradiation. However, irradiation specimens from the TIG weldment failed at the extreme end of the gage length. Since this failure may actually occur at the weld/base metal interface, these results should be considered preliminary until the fracture is better defined.
### TABLE 4.20. Uniaxial Creep-Rupture Properties of Irradiated Stainless Steel Welds

<table>
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<th>Specimen No.</th>
<th>Fluence, n/cm² (total)</th>
<th>Irrad. Temp., °F</th>
<th>Test Temp., °F</th>
<th>Stress, ksi</th>
<th>Minimum Creep Rate, in./in./hr x 10⁵</th>
<th>Rupture Time, hr</th>
<th>Total Elongation, %</th>
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<td></td>
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<td></td>
<td></td>
<td></td>
<td>Irrad.</td>
<td>Control</td>
<td>Irrad.</td>
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<td></td>
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<td></td>
</tr>
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<td>1100</td>
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<td>1100</td>
<td>45</td>
<td>BOL (a)</td>
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(a): Broken on Loading.
REFERENCES


5.0 FAST REACTOR DOSIMETRY AND DAMAGE ANALYSIS

DAMAGE FUNCTIONS AND DATA CORRELATION

W. N. McElroy, R. L. Simons, and L. D. Blackburn

Previous reports\(^{(1-5)}\) discussed the development and study of a method for generating energy-dependent damage functions for the correlation and prediction of neutron-induced property changes.

A preliminary form for a damage function, \(G(E)\), is given in Figure 5.1 for a change in yield strength (YS) to 60 ksi in 304 SS irradiated and tested at \(\sim 450 \text{ °C}\). The solid curve represents the most reasonable of a number of possible solutions for the semi-empirically derived \(G(E)\).

The dashed curve represents a vacancy cluster (greater than 7) production rate model developed by Russcher\(^{(6,7)}\) for austenitic steel. Based on the solid curve in Figure 5.1, damage from low-energy neutrons appears to be more prevalent than indicated by Russcher's input model.

Russcher's curve, the multiple foil-derived spectra for approximately 20 measured data points,\(^*\) and the corresponding fluence estimates for a change to a 60 ksi yield strength were read into the SAND-II code to derive the solid curve solution.\(^{(8)}\) Preliminary results of a solution uniqueness study suggest reasonably good definition for neutron energies above \(\sim 10^{-3} \text{ MeV}\) and poorer definition below this point. This was expected since there is only a single thermal reactor data point.

---

\(^*\) Use of multiple foil-derived values rather than calculated values of spectra and flux levels for the PNL and ANL data made some significant differences (up to a factor of \(\sim 2\)) in total fluence estimates, particularly for out-of-core data points.

5.1
Figure 5.1. Absolute Damage Function for a Change in Yield Strength to 60 ksi in 304 Stainless Steel Irradiated and Tested at \( \sim 450 \) °C.
The experimental data used to derive the damage function, Figure 5.1, are presented in Figure 5.2.* Except for the single ETR thermal reactor, data point 42, all data points are from irradiations in EBR-II.

To translate the experimental data into a meaningful form,** it was necessary to establish for each data point (each spectrum) a value of fluence to produce a change in yield strength to 60 ksi. Grouping data points into three spectral groups (see Figure 5.2) made it possible to establish a shape for a single curve that would approximate the property change versus total fluence for each data point (each spectrum); that is, a single shaped curve (solid lines in Figure 5.2) whose lateral displacement at the 60 ksi level represents the energy dependence of damage for these data points.

Analytically, this curve can be represented by the equation

\[ YS(ksi) = 1856 - 89.49 \ln \phi t + 1.062 (\ln \phi t)^2, \]  

where \( \phi t \) is the total fluence \((E > 10^{-10} \text{ MeV in units of n/cm}^2)\) and \( \beta \) is a coefficient (representing the neutron energy dependence of damage), which determines the intersection of the curve with the dashed horizontal line at the 60 ksi level. The use of Equation (1) is limited to yield strengths in the range from \( \approx 20 \) to 100 ksi.

---

For these PNL and ANL data, the initial undocumented compilation (with references) provided by the FFTF project (dated December 16, 1968 by J. W. Helm and R. Moen of PNL) has been modified and expanded by L. D. Blackburn. New and/or modified references will be provided for all of these data in future publications.

The data used in this study were selected after a careful review to eliminate, as far as possible, all variables except spectra as a source of the difference in property change versus fluence.

5.3
FIGURE 5.2. Yield Strength (YS) Versus Total Fluence for Different Reactor Spectra
The derived $G(E)$, Figure 5.1, is used directly (or in a group-averaged form) with any neutron spectrum, $\phi(E)$, of interest to determine the fluence required to cause a yield strength change to 60 ksi.\(^4\) This fluence, $\phi t$, is then used to define the coefficient $\beta$ for the spectrum, $\phi(E)$, using the equation

$$\beta = 1.90 \times 10^{22}/\phi t.$$ \hspace{1cm} (2)

Substitution of the value of $\beta$ into Equation (1) yields an equation which can be used to estimate the yield strength (within the range of 20 to 100 ksi) resulting from an exposure $\phi t$ in the specified spectrum $\phi(E)$. Such estimates are useful in reactor design, damage analysis studies, and irradiation program planning.

**SAND-II SOLUTION ACCURACY STUDY FOR EBR-II SPECTRA**

R. L. Simons and W. N. McElroy

The accuracy of the integral flux greater than a specified energy $\phi(>E)$, determined by using the SAND-II code, depends on uncertainties in cross sections, measured reaction rates, and uniqueness of solution. A study of the effect of these uncertainties on the accuracy of derived energy-dependent integral fluxes for the EBR-II run 31F dosimetry test has been initiated. For a specified integral flux solution $\phi(>E)$, propagation of errors using a computerized technique will be applied to determine the error due solely to uncertainties in cross sections and reaction rates.

This report considers the contribution of lack of solution uniqueness to the overall inaccuracy of energy-dependent integral fluxes, $\phi(>E)$. Previous error-estimate studies suggest that the combined errors from all sources should introduce no more than about a 10 to 30% absolute uncertainty in integral flux values.\(^{9,10}\)
The SAND-II code calculates

$$\phi^K(\geq E) = \int_{E}^{18 \text{ MeV}} \phi^K(E) \, dE,$$  \hspace{1cm} (1)

where $\phi^K(E)$ is the energy dependent differential flux calculated from a set of $N$ reaction rate integral equations

$$A_i = \int_{10^{-10} \text{ MeV}}^{18 \text{ MeV}} \phi^K(E) \sigma_i(E) \, dE, \quad i = 1, 2, 3, \ldots N.$$  \hspace{1cm} (2)

$A_i$ is the measured reaction rate of the $i^{th}$ reaction, $\sigma_i(E)$ is its corresponding energy-dependent cross section, and $K$ refers to the number of iterations to obtain the $\phi^K(E)$ solution.

This study is intended to show the uncertainty of SAND-II $\phi(\geq E)$ solutions for EBR-II run 31F dosimetry data using the available set of 14 reactions, Table 5.1. Data were analyzed from four bounding locations, Rows 2 and 7 at midplane and 59.2 cm above midplane, of the dosimetry test to establish the limit of uniqueness uncertainty at these positions and intermediate locations. The uniqueness limits were determined by obtaining solutions with an extreme input and comparing the results with either a known or a best solution.

**TABLE 5.1. Foil Reactions Used for Determining Integral Fluxes**

<table>
<thead>
<tr>
<th>Reaction</th>
<th>Reaction</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{235}\text{U}(n,f)$</td>
<td>$^{24}\text{Na}(n,\gamma)$</td>
</tr>
<tr>
<td>$^{237}\text{Np}(n,f)$</td>
<td>$^{54}\text{Fe}(n,p)$</td>
</tr>
<tr>
<td>$^{238}\text{U}(n,f)$</td>
<td>$^{58}\text{Ni}(n,p)$</td>
</tr>
<tr>
<td>$^{58}\text{Fe}(n,\gamma)$</td>
<td>$^{46}\text{Ti}(n,p)$</td>
</tr>
<tr>
<td>$^{59}\text{Co}(n,\gamma)$</td>
<td>$^{47}\text{Ti}(n,p)$</td>
</tr>
<tr>
<td>$^{197}\text{Au}(n,\gamma)$</td>
<td>$^{48}\text{Ti}(n,p)$</td>
</tr>
<tr>
<td>$^{63}\text{Cu}(n,\gamma)$</td>
<td>$^{27}\text{Al}(n,\alpha)$</td>
</tr>
</tbody>
</table>
The extreme input was required to be a physically reasonable approximation to the correct spectral shape. This approximation for each position was determined by inputting to the SAND-II code an initial fission spectrum form above 3 MeV, a constant form from 3 MeV down to \(10^{-4}\) MeV, and a form proportional to energy below \(10^{-4}\) MeV and the calculated or measured reaction rates for each location. The iterative solution to this initial input was expected to be approximately correct in magnitude at the \(^{197}\text{Au}(n,\gamma)^{198}\text{Au}\) and \(^{59}\text{Co}(n,\gamma)^{60}\text{Co}\) major resonances, and for threshold monitors above \(\sim 1\) MeV. Thus, an extreme (but physically reasonable) input could be defined from the initial solution by extrapolating an E form below the gold resonance energy, using straight line log-log interpolations between the gold and cobalt resonance energies and between the cobalt resonance energy and 1 MeV, and the initial solution shape above 1 MeV. This spectrum should be a reasonable approximation of the real spectrum in each case but still could be considered as an extreme input form because it is defined entirely by the experimental data; that is, it has no features derived from reactor physics calculations.

Two approaches were taken in determining the uniqueness bounds. First, a completely analytical case was studied in which there were no errors in reaction rates or cross sections and the true form of the spectrum was known. This was done by calculating reaction rates for four known 2DB-calculated\(^{(11)}\) spectra (for the bounding locations) using the current SAND-II cross sections. These calculated reaction rates and the SAND-II cross sections were then used with the extreme input for each of the four locations to obtain integral flux solutions. The integral flux solutions were compared with the 2DB-calculated integral fluxes by plotting the ratio of solution \(\phi(E)/2DB\) calculated \(\phi(E)\) versus energy. A ratio of one indicates that the solution \(\phi(E)\) and 2DB-calculated
\( \phi(>E) \) are in agreement, Figure 5.3. The second approach used measured reaction rates from the 31F dosimetry test with their associated experimental errors (estimated to be \( \sim 7\% \) of one standard deviation for each of the 14 reactions) and the extreme input to obtain solutions at the four bounding locations. These results are compared to Figure 5.4 with the best solutions by plotting the ratio of the extreme input solution \( \phi(>E) \)/best input solution \( \phi(>E) \) versus energy. The best solutions were obtained using the same four sets of measured reaction rates, but with the best available input spectral forms (assumed to be the four known calculated 2DB spectra).

Figure 5.3 shows that the maximum differences in \( \phi(>E) \) along the reactor midplane (\( z = 0 \)) and the top bounding position (\( z = 59.2 \) cm) are \( \sim 5\% \) and \( \sim 25\% \), respectively. These absolute differences are converted to statistical uncertainties for subsequent error propagation with reaction rate and cross section errors under the conservative assumption that the differences are limits of error at the \( 2\sigma \) (95\% confidence) level. Consequently, at a \( 1\sigma \) (67\% confidence) level, the estimated solution uniqueness uncertainties are 2.5\% and 12.5\% at midplane and at the top of the rows, respectively. Since there were no errors in the reaction rates or cross sections used in this analytical case, these uncertainties result from the lack of solution uniqueness associated with the use of 14 foil reactions rather than an infinite set.

For the second case studied, Figure 5.4 shows that the maximum differences in \( \phi(>E) \) along midplane and at the top locations are \( \sim 8\% \) and \( \sim 50\% \), respectively. At the \( 1\sigma \) confidence level, these uncertainties are \( \sim 4\% \) at midplane and \( \sim 25\% \) at the top locations.* The results in Figure 5.4 show the effect of uniqueness uncertainty due to errors in measured activities and

* In Figure 5.4, the results for the \( r = 5.2 \) cm, \( z = 59.2 \) cm position seem to be excessive relative to the \( r = 33.8 \), \( z = 59.2 \) cm results. These results are being rechecked.
FIGURE 5.3. Integral Flux (>E) for Extreme Input/True Integral Flux
FIGURE 5.4. SAND-II Integral Flux (>E) for Extreme Input/SAND-II Integral Flux (>E) for 2DB Integral Flux Input
cross sections as well as the finite size of the foil set used. 
These results show that the uncertainties due to lack of solu-
tion uniqueness can be increased by a factor of \( \sim 2 \) (in the 
cases studied) by errors in reaction rates and cross sections 
if extreme, but still physically reasonable, input forms are 
used.

For both cases, the ratios in Figures 5.3 and 5.4 indicate 
that the greatest uniqueness uncertainty exists in the energy 
range from \( \sim 0.05 \) to 1 MeV. This is a result of the lack of 
proper sensitivity of any of the present monitors to neutrons 
in this region of the spectrum.*

In conclusion, the contribution to the limit of inaccuracy 
of \( \Phi(\geq E) \) from lack of solution uniqueness for EBR-II run 31F 
using 14 activation monitors is estimated to be no more than 
4 to 25\%, 1\( \sigma \), in the absence of the use of any knowledge about 
the real form of the spectrum. If available reactor physics 
results were used, however, the errors should be much less. 
The lower number would be typical for core and bordering 
regions. In the blanket, the uncertainty would progressively 
increase to about 25\% at a distance of \( \sim 59 \) cm from the core.

**FAST REACTOR MATERIALS DOSIMETRY CENTER**

J. A. Ulseth and J. L. Jackson

The data file of the Fast Reactor Materials Dosimetry 
Center has been enlarged and new data handling and reporting 
methods have been incorporated into the center.

Since last reported,(12) the data file has been enlarged 
by an inclusion of unfolded neutron spectra for 13 axial loca-
tions (0 to 76 cm above midplane) for each of 6 radial 
positions (0 to 33 cm) from core center out into the inner

* Recent studies suggest that the addition of the \( ^{238}\text{Pu}(n,f) \) 
reaction will improve foil coverage in the 0.1 to 1 MeV 
range.
blanket region -- bringing the total of unfolded spectra to over 100. These spectra were unfolded (using the SAND-II code) (13) from 14 measured monitor reaction rates from the run 31F dosimetry experiment. (14) The 14 reaction rates at each reactor position are in the form of a $10 \times 18$ matrix. (15)

The spectral data are stored in the form of printed output that includes absolute differential and integral flux for 620 energy points ranging from $10^{-10}$ to 18 MeV, spectral-averaged cross sections for 35 different monitor reactions, and the energy ranges over which the 35 monitors respond in that particular spectrum. The differential flux is punched on IBM cards and is plotted for 620 energy groups.

As requests to the dosimetry center for flux and/or fluence analyses for EBR-II irradiations increase, the data handling and analysis of data become more complicated and time consuming. To facilitate usage, automated procedures have been written for rapid retrieval of information from the expanding data file, and axial and radial flux and fluence distributions for the EBR-II core and blanket can now be rapidly obtained from it. For example, if the distribution of flux is desired for a particular radius, the integral flux above or below any specified energy (up to 20 different energies) are printed in a table suitable for publication. Any or all of the columns in the table can be automatically plotted to give an axial distribution of the integral flux above or below the selected specified energy. Similarly, fluence can be estimated if the megawatt seconds of irradiation are input to the program, and radial flux or fluence distribution in the form of tables or plots can be provided. Two examples of the type of output that can now be obtained from the data file are shown in Figures 5.5 and 5.6. Figure 5.5 is a typical table showing axial distribution of integral flux above various lower energy limits. Figure 5.6
### EDH-II RUN 31F - RADIUS = 5.2CM

**INTEGRAL FLUX ABOVE SPECIFIED ENERGIES**

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<th>CM FROM REACTOR MIDPLANE</th>
<th>ENERGY OF SPECTRUM (MEV)</th>
<th>TOTAL FLUX (N/cm²*sec)</th>
<th>NORMALIZED INTEGRAL FLUX ABOVE (&gt;1,0-01) OR BELOW (&lt;1,0-01) SPECIFIED ENERGIES (ENERGIES IN MEV)</th>
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<th>&gt;1.0-01</th>
<th>&gt;4.0-01</th>
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</tbody>
</table>

Neg 701383-7

**FIGURE 5.5.** Example of Tabular Computer Data Output
EBR II RUN 31F - RADIUS = 5.2CM
INTEGRAL FLUX ABOVE SPECIFIED ENERGIES

\(10^{16}\)
\(10^{15}\)
\(10^{14}\)
\(10^{13}\)
\(10^{12}\)

\(\bigcirc \) E > 0.1 MeV
\(\triangle \) E > 1.0 MeV
\(\star \) E > 1.0-10 MeV

DISTANCE ABOVE MIDPLANE, CM

FIGURE 5.6. Example of Plotted Computer Data Output
is a plot of the flux greater than specified energies as a function of axial distance above midplane.

Any or all of the 14 saturated reaction rates can be plotted (as a function of r or z) from the 31F dosimetry test reaction rate matrix. Thus with this aid, an experimenter can determine activity levels for monitors to be included in future EBR-II irradiations. Flux levels at any core or blanket position can also be estimated.

The 31F dosimetry test was conducted during the last quarter of 1968. The general configuration of the core and the inner blanket has not changed to this date, therefore the 31F saturated reaction rates and flux levels continue to be valid.

COMPUTER SIMULATION OF RADIATION DAMAGE
D. G. Doran

The computer simulation of displacement cascade overlap in α-iron has continued. The purpose is to determine how the presence of defect clusters in a lattice influences the defect production in displacement cascades that are subsequently produced.

The results of the simulation of the production and short-term annealing of nine 20 keV cascades were reported previously; these runs will be called Pass 1. The procedure adopted here was to select the final defect configuration from the high temperature annealing simulation of one of these spikes (No. 2051) and modify it for use as the initial damage state. The modification consisted of removing the mobile defects and rearranging the clusters into more stable configurations. Specifically, 13 single vacancies (designated $V_1$) were removed and the $V_5$ and $V_{21}$ clusters were transformed into compact polyhedra; 17 single and 4 di-interstitials were removed, and the two $I_3$, the $I_4$, 5.15
and the \( I_5 \) clusters were transformed into compact platelets in \{110\} planes. Note that the initial numbers of vacancies and interstitials are not equal.

This cluster configuration was then used as the initial damage state in the CASCADE-CLUSTER code for the simulation of nine new displacement spikes (Pass 2). The take-off points and initial directions of the primary knock-on atoms (PKA) into a perfect crystal for Pass 2 were the same as for the previously reported Pass 1.

The resulting cascades were treated with a simulated short-term anneal with the HAP-4 (Hybrid Anneal Program) code. This code, written for a BNW hybrid computer, includes all the operations of the ANNEAL code used previously, plus a MODE 4 in which simultaneous interstitial and vacancy migration are permitted.* Each spike was run in MODE 4 until interactions were infrequent, generally 5000 interstitial time steps. The spike was then run in MODE 3 in which only vacancy migration is permitted for 1000 vacancy time steps.**

The CASCADE-CLUSTER runs (the 0 °K configurations) were only weakly affected by the lack of crystal perfection. For 6 of the 9 spikes, the collision histories of the primary atom were identical in Passes 1 and 2, and the collision histories of higher order knock-ons were altered only slightly. The average number of defects per spike was just the average number after Pass 1 plus the number present in the initial damage state.

* One potential source of error in operating HAP should be noted. A random noise generator is the ultimate source of the random numbers used in determining the order of defect selection, whether a jump shall occur, and the jump direction. This instrument was found to require frequent adjustment to compensate for long term drift. If this is not done, an observable trend for defects to migrate in certain preferred directions can result. The drift during a single run is considered negligible.

** An interstitial (vacancy) time step is defined as one-tenth the mean jump time of an uncorrelated single interstitial (vacancy).
This result is coincidental, however, because a weak trend toward lower production of defects in 8 of 9 spikes was exactly counterbalanced by a large increase in one spike (not 2051).

During the simulation of a short-term high temperature anneal, the evolution of the defect configuration from Pass 2 followed closely that of Pass 1. The most notable difference was the generally lower fraction of mobile defects throughout the simulation. The apparent annihilation of interstitials and vacancies dropped from 79% for both to 77% and 73% respectively. These differences, however, are almost wholly due to the initial damage state present for Pass 2. The number of annihilation events actually increased 5%.

The annealed configurations for several of the spikes are shown in Figure 5.7 (the initial damage state is shown for comparison). The cluster size distributions for Passes 1 and 2 are compared in Figures 5.8 and 5.9.

Several observations can be made concerning the differences in the distributions and the relation of these differences to the initial damage state. The initial large cluster, $A_{V_{21}}$ (the largest cluster observed after Pass 1), grew in 5 spikes, shrank in 3, and remained dormant in 1. Final sizes of this cluster range from $V_{15}$ to $V_{33}$, the latter occurring in No. 2051. The smaller vacancy cluster initially present, $a_{V_{5}}$, survived in 6 of 9 spikes. Although 2 vacancy clusters were present initially, the Pass 2 runs average 1.3 more clusters ($V_{>4}$) than in Pass 1. The incidence of large vacancy clusters correlates roughly with the proximity of the PKA take-off direction to that of the PKA of spike No. 2051. It was noted above that the fraction of mobile defects was lower in Pass 2 than in Pass 1. Figure 5.8 makes it evident that this relative decrease of
FIGURE 5.7. CRT Displays of Simulated 20 keV Displacement Cascades Produced in Previously Damaged Lattice and Subsequently Annealed. (Vacancies and interstitials indicated by 0 and X symbols respectively. Take-off point at origin in direction of arrow. Edge length of box is 50 half-lattice units.)
Neg 701383-3

**FIGURE 5.8.** Comparisons of Vacancy Cluster Distributions Produced in Simulated Displacement Cascades
FIGURE 5.9. Comparisons of Interstitial Cluster Distributions Produced in Simulated Displacement Cascades
small clusters extends generally to vacancy clusters of size 15 or less. The fraction of vacancies in larger clusters increased significantly.

Pass 2 of spike No. 2051 produced the largest interstitial cluster \((I_{12})\) yet recorded in this work. It resulted through growth of the \(I_5\) which was present initially. This \(I_5\) survived in 7 of the 9 spikes but grew only in No. 2051. The other large interstitial clusters resulted from the growth of smaller clusters present in the initial damage state. In 6 of the 9 spikes, the interstitial cluster size distribution extends to larger sizes in Pass 2 than in Pass 1. The average number of interstitial clusters \((I_{>3})\) per spike in Pass 2 is only 1.6 more than in Pass 1, although there were 4 clusters present initially. This fact, together with the \(\approx 20\%\) decrease in the fraction of mobile interstitials, emphasizes the increase in average cluster size.

As discussed in the previous quarterly report,\(^3\) the HAP-4 code allows parameter variations to be made with relative ease. One of the Pass 2 runs was performed with the interstitial-vacancy recombination criteria replaced by a simple distance criterion. Previous work had shown that a recombination distance of \(\leq \sqrt{19}\) hlu (half lattice units), extending through 7th neighbors and including 88 sites, was too small. For the present run, the distance was increased to \(\leq \sqrt{20}\) hlu, extending through 8th neighbors and including 112 sites. The annealing behavior appears to be indistinguishable from the standard runs in which the 62-site, bow-tie shaped region shown in Figure 5.10 was used.
COMPUTER SIMULATION OF IRRADIATION PROCESSES

G. E. Russcher

The development of the SLIDES code continues through the debugging process. Several different test problems are being used to detect weak logic procedures and improper computational techniques.

As the code has been refined, many logic tests and diagnostic information sources have been added. These additions overloaded the available computer memory storage. Consequently, the code has been modified to use the Memory Allocation Processor (MAP), which now operates the SLIDES code in three segments. The main program and necessary common-data subroutines are in continuous operation, but most of the subroutines are segregated by function into:

- Lattice structure description and data preparation.
- Atomic interaction calculations.
- Edit, catalog, and output of the disarranged-lattice description.

COMPUTER TABULATION OF DOUNREAY FAST REACTOR FLUX AND REACTION RATE DATA

R. E. Dahl, Jr., R. D. Bourquin, and N. J. Graves

A great deal of experience in fast reactor fuels and structural materials has been gained through irradiations performed in the Dounreay Fast Reactor (DFR). A comprehensive report\(^\text{17}\) has been issued which gives the results \(\phi(E)\) for 16 energy groups in a \(r,z\) matrix which is \(28 \times 23\) of a multi-group calculation for fluxes in the DFR core and reflector regions. These data have been incorporated in a computer program, and neutron flux and spectrum can be recalled for any point of the matrix. Thus, a highly detailed, flux profile for any subassembly or pin in the reactor can be obtained very
quickly. This is very useful in analyzing stainless steel swelling data and fuel element dilatation and in studying fuel element modeling.

In addition to the flux data presented in the reference document, other sets of previously presented detailed data were incorporated into the library program. These include lineal heat generation rates for various fuel elements commonly irradiated in the DFR, fission rates for a number of fissile isotopes, activation rates for common flux monitors, and a tabulation of expected damage rates in stainless steel according to the Thompson-Wright model. Access to the data may be obtained by request to the dosimetry center.

**COMPUTER SIMULATION STUDIES OF DISLOCATION CORE STRUCTURE**

R. E. Dahl, Jr. and R. D. Bourquin

To quantify models of swelling and irradiation-induced creep in stainless steels, an experimenter requires a knowledge of atomistic scale parameters which define damage production and diffusion parameters in the disordered structure. Data on the strength of interstitial and vacancy sinks, which include grain boundaries, dislocation loops and voids, comprise part of the most needed information. Diffusion coefficients and activation energies for vacancy and interstitial migration to loops and voids, and data which would define a mechanism for void growth are also essential. Dislocation loops play an extremely important role in determining swelling and plastic deformation of metals. Therefore, an initial and extremely necessary step in providing the required data was to develop the facility to simulate dislocation core structure. The GRAINS\(^{18}\) program has been modified and tested so that edge dislocations, partial or complete, can be constructed and relaxed to equilibrium configurations.
So far the simulation has been limited to fcc gamma iron. The principal slip system [(111) plane, (112) direction] for fcc metals has been simulated by constructing cells in which the frontal planes are 112 planes and the dislocation lines are in a 112 direction. The dislocations are set up initially using the method of Doyama and Cotterill,\(^{(19)}\) and are then moved to positions dictated by either isotropic or anisotropic elasticity theory using the equations given by Gehlen, Rosenfield, and Hahn.\(^{(20)}\) The atoms are then relaxed (moved) to equilibrium positions according to classical mechanics. The dislocation structure is maintained throughout the computational cell by a technique whereby the atoms which have three degrees of freedom (those within the interior of the computational cell) move and then are frozen, while atoms in crystallographic equivalent planes on the front and rear surfaces of the computational cell are moved to corresponding positions. The process is repeated until the potential energy of the computational cell is at a minimum. Figures 5.11 and 5.12 show the core structure before and after relaxation. Figure 5.11 is a (112) plane in which the atoms are located in positions dictated by isotropic elasticity theory according to their distance and direction from the dislocation. The atoms in Figure 5.12 have been relaxed, and this is a basic structure upon which the initial computer simulation studies for swelling and creep will be conducted.

In these figures, the hexagons are atoms which were completely moveable during the computations, and the squares represent atoms that were initially moved to their positions according to elasticity theory but which remain immobile during the relaxation process. The exact position of each atom falls in the center of its symbol, or at the end of the vertical center-line of the symbol.

The results obtained to date and illustrated in these figures corroborate the results of Cotterill and Doyama\(^{(19)}\) and
Neg 700240-13

FIGURE 5.11. Computer-Drawn Plot of Computational Cell Core Structure Before Relaxation
FIGURE 5.12. Computer-Drawn Plot of Computational Cell Core Structure After Relaxation
of Gehlen et al.\textsuperscript{(20)} in that the core region of a dislocation actually has a fairly high degree of order. This is also shown in Figure 5.13 which is a computer drawn plot of the plane just below the slip plane for the edge dislocation. (The three types of symbols show the ABC stacking of the fcc (111) planes.) This also illustrates that the atoms in the system relax to a fairly well ordered structure within close proximity to the dislocation core. The results shown may be of interest since atomic positions are located quite exactly through the plotting routine. The next stages in this investigation will be to determine changes in volume and pressure caused by the addition of interstitial atoms to jogs. This will have a direct relationship to swelling studies since the absorption of interstitials onto loops is a very probable mechanism causing dilatation.

**FLUENCE DETERMINATIONS USING TIME-VARYING FLUX**

R. D. Bourquin

Historically, flux and fluence have been calculated assuming a constant reactor power level for an irradiation. Since no reactor operates continuously, the values calculated for flux and fluence would be more precise if the effects of reactor cycling were considered. Thus, especially for a short half-life monitor, the flux calculated by assuming a constant power level could be significantly in error if the power level fluctuates.

For example, consider a hypothetical irradiation using monitors whose daughter nuclide half-life is (1) 10 days (typical of fission monitors) and (2) 300 days (typical of threshold monitors). In addition, assume:

- Spectral-averaged cross section of 1.
- Activity of 1.0 dps/nucleus.
- One hundred and fifty-day irradiation having a power history as shown in Figure 5.14.
- No burnout of daughter nuclide.
Neg 701383-8

A. Before Relaxation

Neg 701383-6

B. After Relaxation

FIGURE 5.13. Computer-Drawn Plot of Plane Just Below Slip Plane for Edge Dislocation
The resulting ratios would be as shown below.

<table>
<thead>
<tr>
<th></th>
<th>Short (10 day)</th>
<th>Long (300 day)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\frac{\phi(\text{constant power})}{\phi(\text{actual})}$</td>
<td>1.480</td>
<td>0.977</td>
</tr>
<tr>
<td>$\frac{\phi(\text{TIMH})}{\phi(\text{actual})}$</td>
<td>1.000</td>
<td>1.000</td>
</tr>
</tbody>
</table>

The first line gives the result of the constant power assumption and the second line the result corrected for reactor power history using the TIMH code. For a long half-life monitor then, either method is well within the error limits of activation counting data, but as seen for the short half-life monitor, accounting for actual power level fluctuations is necessary to avoid excessively large errors in flux and fluence calculations.

A computer program is now available that uses reactor power-history data for the determination of flux and fluence by foil activation analysis. This program, TIMH,\(^{(21)}\) has successfully calculated flux and fluence using activation data from EBR-II irradiations. Additional information may be obtained from the Dosimetry Center.

REFERENCES


12. See pp. 5.5-5.7 in Reference 3 above.

13. See p. 15 in Reference 9 above.


15. See pp. 5.4-5.5 in Reference 3 above.


6.0 FAST REACTOR ABSORBER MATERIALS

G. L. Tingey

RADIATION EFFECTS ON BORON CARBIDE AND TANTALUM

A. L. Pitner and D. E. Baker

Irradiation in a Thermal Flux

The irradiation of boron carbide at 500 to 1200 °F in Hanford thermal reactors has been completed. The boron carbide samples were in five forms: 60 and 80% TD powders, and 65, 80, and 99% TD pellets. Pellet sample size was nominally 0.25 in. diameter × 1 in. long. The irradiations were conducted in two subassembly groups. Subassemblies 1, 2, and 3 operated at temperatures of 550 to 1200 °F, while subassemblies 4, 5 and 6 ran at 500 to 750 °F. Subassemblies 1, 2, and 4 have thus far been disassembled and examined for helium gas release and B\(^{10}\) burnup. Mass-spectroscopic analyses of isotopic ratios indicate that the average exposure for Subassemblies 1, 2, and 4 are 7 × 10\(^{20}\) captures/cm\(^3\) (≈4% B\(^{10}\) burnup), 21 × 10\(^{20}\) captures/cm\(^3\) (≈12% B\(^{10}\) burnup), and 15 × 10\(^{20}\) captures/cm\(^3\) (≈8% B\(^{10}\) burnup), respectively. Because of the high thermal cross section of B\(^{10}\), however, nearly all of the reaction occurs on the surface of the samples, while the inner cores remain relatively unreacted. Consequently, burnup in affected areas is always very nearly 100%, and the gas release fractions are not expected to vary significantly with total irradiation exposure.

Gas release values for the three subassemblies are shown in Figure 6.1. Each data point is accompanied by a number indicating which subassembly the sample resided in. In general, the results from Subassembly 1 show higher gas release fractions than do the data from Subassemblies 2 and 4. Since Subassembly 1 had the lowest exposure, the total amount of gas generated was less than in the other two subassemblies, and the accuracy of
the gas pressure measurements would be correspondingly lower. Consequently, these values are probably the least reliable and were ignored in drawing the curves. The 99% TD pellets release the lowest fraction of gas. The 65 and 80% TD pellets do not appear to vary significantly in their gas release behavior, but both release a higher gas fraction than the 99% TD pellets. The percent of gas released from boron carbide appears to increase rapidly when the irradiation temperature exceeds 1000 °F. There is considerable scatter in the gas-release values for the powders, and no attempt was made to draw a curve through them. The gas-release fractions in the powders are, however, generally higher than in the pellets.

Postirradiation examination will continue on remaining Subassemblies 3, 5, and 6. The estimated exposure for each of these is $25 \times 10^{20}$ captures/cm$^3$ ($\approx 14\%$ B$^{10}$ burnup). Swelling measurements and postirradiation annealing/gas release studies are also planned for most of the samples.

Apparatus for Annealing Studies of Boron Carbide

An apparatus was constructed to measure helium gas release from boron carbide during postirradiation annealing. The apparatus consists of a high vacuum system, gas burette, manometer, and Toepler pump. The sample is heated by either a 10 kW induction generator or a resistance tube furnace. Boron carbide samples were heated to 2100 °C and the evolved helium was transferred with a Toepler pump to a calibrated volume for gas pressure measurement. Initial operation of the apparatus indicates that it will perform as designed and will be useful in studies of the kinetics of helium release from irradiated boron carbide.

Fast Reactor Irradiation

When the EBR-II tantalum capsule TA-4 was disassembled and the inner capsule cladding rupture observed, it was determined
that an identical capsule, TA-5 with the same irradiation history was probably similarly ruptured. Analysis of a gas sample taken from the annulus between inner and outer capsules confirmed the rupture. The TA-5 capsule was subsequently disassembled and the rupture observed in a position corresponding to that of one of the sets of melt wires.

Although TA-4 contained only melt wires for temperature monitoring, TA-5 contained a silicon carbide temperature monitor located at the hottest position in the capsule in addition to the melt wires. Postirradiation annealing of the SiC pellet indicated a maximum irradiation temperature of 2100 °F, a temperature considerably higher than 1650 °F which was estimated from heat transfer calculations.

The length and density of each of the samples in TA-5 have been measured, and the results are shown in Table 6.1. Previous measurements from TA-4 are also included for comparison. The results from the two capsules agree reasonably well and show an unusually high temperature dependence. Samples irradiated at the lower temperatures exhibited the greatest swelling, even though their exposure were lower than the higher-temperature samples.

Metallography was performed on the tantalum holder that contained the silicon carbide pellet in TA-5 to determine if any interaction occurred during the irradiation. No definite indication of interaction was observed. A out-of-reactor test at 1000 °C also continues to support the compatibility of tantalum with silicon carbide. After 1000 hr at temperature, weight changes in the silicon carbide were within the accuracy of the weighing.

The tantalum replacement capsules, BTA-6 and BTA-8, have been loaded into the X042 subassembly in EBR-II and are now under irradiation. Based on the temperature measurement from
the SiC pellet in TA-5, tantalum samples in BTA-6 and BTA-7 are expected to reach maximum temperatures of 2300 °F. Silicon carbide monitors have been included throughout the length of these capsules and should yield more accurate temperature profiles along the capsules. After BTA-6 has received an exposure of $\sim 10^{22}$ nvt, it will be replaced with an identical capsule (BTA-8) and be disassembled. Upon completion of the irradiation, BTA-7 and BTA-8 will have received exposures of $\sim 3.4$ and $2.4 \times 10^{22}$ nvt, respectively.

**TABLE 6.1. Swelling of Tantalum Irradiated in EBR-II**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Est. Exposure $(E &gt; 0.1 \text{ MeV})$</th>
<th>Temp., °F</th>
<th>$\Delta L$, %</th>
<th>$\Delta \rho$, %</th>
<th>$\Delta L$, %</th>
<th>$\Delta \rho$, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>$3.0 \times 10^{21}$</td>
<td>1325</td>
<td>+0.13</td>
<td>-0.69</td>
<td>+0.18</td>
<td>-0.88</td>
</tr>
<tr>
<td>2</td>
<td>$4.6 \times 10^{21}$</td>
<td>1600</td>
<td>+0.13</td>
<td>-0.68</td>
<td>+0.13</td>
<td>-0.72</td>
</tr>
<tr>
<td>3</td>
<td>$6.7 \times 10^{21}$</td>
<td>1875</td>
<td>+0.11</td>
<td>-0.85$^a$</td>
<td>+0.07$^a$</td>
<td>-0.47</td>
</tr>
<tr>
<td>4</td>
<td>$8.2 \times 10^{21}$</td>
<td>2025</td>
<td>-</td>
<td>-0.58</td>
<td>+0.06</td>
<td>-0.53</td>
</tr>
<tr>
<td>5</td>
<td>$8.6 \times 10^{21}$</td>
<td>2100</td>
<td>+0.01</td>
<td>-0.42</td>
<td>+0.02</td>
<td>-0.29</td>
</tr>
<tr>
<td>6</td>
<td>$8.2 \times 10^{21}$</td>
<td>2025</td>
<td>+0.05</td>
<td>-0.45</td>
<td>+0.03</td>
<td>-0.25$^a$</td>
</tr>
<tr>
<td>7</td>
<td>$6.7 \times 10^{21}$</td>
<td>1875</td>
<td>+0.06</td>
<td>-0.62</td>
<td>+0.10$^a$</td>
<td>-0.61</td>
</tr>
<tr>
<td>8</td>
<td>$4.6 \times 10^{21}$</td>
<td>1600</td>
<td>+0.09</td>
<td>-0.63</td>
<td>+0.11</td>
<td>-0.82$^a$</td>
</tr>
<tr>
<td>9</td>
<td>$3.0 \times 10^{21}$</td>
<td>1325</td>
<td>+0.13</td>
<td>-0.72</td>
<td>+0.09$^a$</td>
<td>-0.58</td>
</tr>
</tbody>
</table>

(a) Plating from nearby melt wire apparently caused measurement to be in error.
FIGURE 6.1. Helium Gas Release of Boron Carbide Irradiated in a Thermal Flux
7.0 NUCLEAR GRAPHITE

G. L. Tingey

DIMENSIONAL CHANGES OF GRAPHITES AT HIGH NEUTRON FLUENCES
AND HIGH TEMPERATURES
A. L. Pitner and W. J. Gray

Conventional nuclear graphites as well as several experimental graphites are being irradiated to very high neutron fluences at high temperatures primarily to characterize the dimensional changes that take place under these conditions. Data obtained so far have disclosed two noteworthy items:

- Post-turnaround expansion of at least some graphites is less rapid at temperatures above 1200 °C than at temperatures of 900 to 1200 °C.
- Graphites manufactured by POCO Graphite Inc., exhibit smaller dimensional changes and less rapid expansion at high fluences than do conventional graphites.

Figure 7.1 shows data at three temperatures for NC8 graphite, a conventional needle-coke grade. The 650 to 750 °C data have been previously published and are included only for comparison. At 1200 to 1300 °C the post-turnaround expansion rate in the perpendicular direction is very much less than at 950 to 1050 °C. Our data indicate that the marked temperature dependence occurs around 1200 °C, but in the range from 900 to near 1200 °C, the curves approximate that shown for 950 to 1050 °C. Suddenly, however, at temperatures above 1200 °C, the post-turnaround expansion rate is markedly reduced.

The data in the parallel direction at high temperatures is badly scattered. Sample numbers have been included beside some of the data points so that the length-change trends can be more readily seen. Initial contraction rates are greater at 1200 to 1300 °C than at 950 to 1050 °C. Post-turnaround behavior has not yet been well established. However, the
FIGURE 7.1. Irradiation-Induced Length Changes of NC8 Graphite
mechanisms that operate to produce the slow post-turnaround expansion rate at 1200 to 1300 °C in the perpendicular direction should also apply to the parallel direction.

The high-temperature data shown for NC8 graphite in Figure 7.1 is typical of at least three grades as follows:

- CSF, a non-needle coke graphite.
- RC5, a gilsonite coke graphite.
- RP4, a composite of both needle and non-needle cokes.

The most likely explanation for the slow post-turnaround expansion rate at high temperatures seems to be related to irradiation-induced creep. At all temperatures, rapid expansion of the crystallites in the c-axis direction is partially accommodated at low fluences by Mrozowski cracks that form during cool-down from graphitization temperatures. This, together with contraction of the crystallites in the a-axis direction, results in net contraction for the polycrystalline graphite. The Mrozowski cracks, however, eventually become filled. Further expansion of the crystallites in the c-axis direction and contraction in the a-axis direction can then only be accommodated by formation of new cracks or by localized plastic deformation. At temperatures below ~1200 °C, the irradiation creep constant is apparently not high enough to prevent crack formation. Therefore, the graphite expands rapidly. Above 1200 °C, enough plastic deformation is possible to accommodate the crystallite strains so that no cracks are formed and only a very slow net expansion is observed.

The seven grades of POCO graphite that are being irradiated in this program are generally described as fine-grained, isotropic, high-strength, high CTE materials. They vary in density from 1.5 to 1.9 g/cm³, and have experienced graphitization temperatures of 2300 to 2800 °C. All seven grades show generally similar irradiation behavior that is typified in Figure 7.2, which presents the results for AXF-8Q1. This
FIGURE 7.2. Irradiation Induced Length Changes of POCO Graphite AXF-8Q1
Graphite was graphitized at 2800 °C and has a density of 1.8 g/cm³. The dotted curves in the figure show the comparative behavior of a conventional, needle-coke graphite, NC8 at 1000 °C. At irradiation temperatures around 1100 °C, the POCO graphite shows expansion from the onset of irradiation. This expansion, however, saturates after an exposure of \( \sim 5 \times 10^{21} \text{ n/cm}^2 \), and the material does not begin expanding significantly again until the fluence exceeds \( \sim 10^{22} \text{ n/cm}^2 \). The expansion rate in this latter stage is seen to be considerably less than that shown by conventional graphites after turnaround in this temperature regime. Above 1200 °C the POCO graphite contracts slightly with irradiation until a fluence is reached of \( \sim 10^{22} \text{ n/cm}^2 \), after which the trend appears to be toward slow expansion. The maximum linear contraction of the various POCO grades is usually on the order of 1%.

**COMPUTER CALCULATIONS OF GRAPHITE OXIDATION IN HTGR**

R. C. Giberson

Additional data have been received from Gulf General Atomic concerning the 1100 MW\(_e\) HTGR. This data indicates a leak rate of \( 4.2 \times 10^{-2} \) lbs of H\(_2\)O/hr and a purification factor of \( 9.9 \times 10^{-5} \) fraction per cycle. Using this new data we have reevaluated the steady-state gas compositions to be anticipated in the 1100 MW\(_e\) reactor. Table 7.1 presents the results of these evaluations compared with the 330 MW\(_e\) (PSC) reactor.

The steady-state water vapor in an HTGR is given approximately by:

\[
PH_{2\, C}^{ss} \approx \frac{PH_{2\, OLK}}{\Delta W + PF},
\]

where: \( PH_{2\, OLK} = \) water in leakage rate.

\( PF = \) purification factor (fraction of gas purified per cycle).

\( \Delta W = \) change in water vapor in passing through the reactor core.
For the reactions considered in the present computer program,\(^{(1)}\) any change in water vapor concentration gives an equal but opposite change in hydrogen concentration; therefore, the hydrogen steady state is:

\[
PH_{2ss} = \frac{\Delta W \cdot (PH_2O)}{PF} \quad (2)
\]

and

\[
\left( \frac{PH_2}{PH_2O} \right)_{ss} = \frac{\Delta W}{PF} \quad (3)
\]

**TABLE 7.1. Steady-State Gas Compositions in HTGR's**

Thermal Reactions only, \(K = 1.0K^{(a)}\)

<table>
<thead>
<tr>
<th>Reactor</th>
<th>Concentrations in mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>(MW_e)</td>
<td>(\text{H}_2\text{O})</td>
</tr>
<tr>
<td>330 (^{(b)})</td>
<td>(7.24 \times 10^{-2})</td>
</tr>
<tr>
<td>1100</td>
<td>(1.16 \times 10^{-2})</td>
</tr>
</tbody>
</table>

\(^{a}\) Refer to normal TSX graphite rate constants

\(^{b}\) Values reported for the 330 MW\(_e\) are slightly different than given in the previous quarterly. This is a result of a change in the temperature simulation equation.

As shown in Table 7.1, calculated by the computer program, \(\Delta W\) is significantly higher in the 1100 MW\(_e\) than in the 330 MW\(_e\) reactor. This is a result of the higher graphite temperatures in that reactor.

To a first approximation, Equation (3) can be used to determine the effect of increased purification system capacity on the hydrogen/water vapor ratio. It also suggests that this ratio is relatively independent of leak rate. However, precise effects must be calculated because \(\Delta W\) is not immune to water leakage.
The results given in Table 7.1 are calculated neglecting radiation-induced reactions. It is noted, however, that radiation effects increased the $H_2/H_2O$ ratio in the 330 MW$_e$ HTGR,$^1$ and a similar effect is expected for the 1100 MW$_e$. Data have also been obtained from GGA on some postulated accident conditions. These will be analyzed and reported in the next quarterly.

**RADIOLYSIS OF H$_2$-CO$_2$-He MIXTURES**

D. E. Clark and G. L. Tingey

The apparent stability of CO$_2$ toward ionizing radiation can be attributed to the rapid recombination of intermediates.$^2$ However, in the presence of chemical scavengers which interfere with the recombination reactions, decomposition of CO$_2$ does occur to a significant extent. As an example of such a substance, hydrogen gives the overall reaction $H_2 + CO_2 \rightarrow CO + H_2O$. We have studied the reaction of equimolar amounts of $H_2$ and CO$_2$ in helium, the reactive gas concentrations ranging from ppm levels to 50 mole%. Both static and single-pass flow methods of irradiation were used and gave comparable results.

The relative yield of CO, Y(CO), is shown in Figure 7.3 as a function of reactant concentration. The dose rate must be known in order to calculate the G-value for the reaction (the number of molecules formed per 100 electron-volts absorbed). We have determined the dose rate with two different chemical dosimeters, the formation of $N_2$ from $N_2O$ $[G(N_2) = 9.3]$ and $H_2$ from $CH_4$ $[G(H_2)= 5.7]$. Agreement between the two was good, giving a value for the current dose rate of $5.5 \times 10^{17}$ electron-volts per second per mole of electrons. With reference to Figure 7.3, then, this leads to the expression $G(CO) = 0.018 Y(CO)$. 

7.7
FIGURE 7.3. Irradiation of Flowing Gas Stream Yield of CO (Relative Units) Versus CO₂ Concentration
In mixtures containing less than about 10,000 ppm of reactants, energy is absorbed mainly by helium which results in energy-rich forms of He*:

$$\text{He} \xrightarrow{\gamma} \text{He}_i^*$$

These may be de-excited through the emission of radiation

$$\text{He}_i^* \xrightarrow{k_2i} \text{He} + h\nu,$$

or through the transfer of energy to reactive species

$$\text{He}_i^* + \text{CO}_2 \xrightarrow{k_{3i}} \text{He} + \text{CO}_2^*,$$

which then undergo chemical change to form the observed products. The latter reaction occurs at a significant rate down to the ppm level of reactive gas concentration, thus implicating relatively long-lived metastable states of atomic or molecular helium as the energy-transfer species. At these concentration levels, the rate of direct activation of the reactants, such as by the reaction \( \text{CO}_2 \xrightarrow{\gamma} \text{CO}_2^* \), is relatively slow and can be ignored. Our results also indicate that transfer of energy to \( \text{H}_2 \) does not significantly affect the reaction rate, but rather that the rate depends primarily on the transfer of energy to \( \text{CO}_2 \).

We are presently studying the effect of pressure and temperature on the observed reaction. Assuming a steady-state concentration of \( \text{He}^* \), the following expression is obtained:

$$\frac{d}{dt} (\text{CO}) = \frac{k_1k_4(\text{He})(\text{CO}_2)}{k_2 + k_4(\text{CO}_2)} + k_3(\text{CO}_2)$$
Thus, depending on the relative values of $k_1$, $k_2$, $k_3$, and $k_4$, the overall reaction rate should depend on the pressure of He, $P_{\text{He}}$, or on $P_{\text{He}} \times P_{\text{CO}_2}$ in the concentration range where one mode of de-excitation predominates over the other. The yield was shown to increase with pressure in the range of $\sim 1000$ ppm but does not obey the simple $P$ or $P^2$ relationship. At lower relative concentrations, it is expected that a linear dependence on $P$ will be observed.

REFERENCES


8.0 NONDESTRUCTIVE TESTING

EDDY CURRENT METHODS
H. L. Libby

A simulated practical tube testing problem has been solved using the multiparameter eddy current tester. When tubes must be tested after they are installed in a system, large signals are often received from known causes such as tube supports or iron inclusions in concrete surrounding the tube. It is often impossible to test for flaws on the inner (ID) and outer (OD) surfaces of a tube which are near or under a tube support by using a conventional single frequency eddy current tester. This study shows that the multiparameter eddy current tester can be used to test for flaws on both the inner and outer surface of a tube even though the flaws are under simulated tube supports. However, this report does not intend to imply that the study shows that all eddy current testing problems can be solved by using the multiparameter eddy current tester.

A Hanford prototype multiparameter eddy current tester was used to demonstrate its advantage over the use of a conventional single frequency tester for detecting flaws in tubing which occur coincident with tube supports. The same tester, flaw standard, probe and simulated tube supports were used for all of the tests described in this study. For the single frequency test, the tester was operated at a single frequency (125 kHz). For the multiparameter tests, the tester was operated at 125 kHz and 250 kHz, simultaneously. The flaw standard was a piece of 0.5 in. ID, 0.050 in. wall, 304 SS tubing with a 0.023 in. deep OD notch, 0.015 in. deep ID notch, a kink, and an area of suspected ID intergranular corrosion. Two simulated tube supports were used: (1) a piece of flat, 3/8-in. thick mild steel, and (2) a piece of 1/2-in. thick, flat stainless steel, each with a 41/64 in. diameter hole in it. The simulated tube supports fit the tube loosely enough
to be moved along the tube, but were not a sloppy fit. The test probe was a standard encircling type, inside differential coil arrangement.

The in-phase and quadrature signal component channels of a single frequency (125 kHz) eddy current tester are shown in Figures 8.1 and 8.2. The signals of each channel, from left to right, are caused by:

- 0.023 in. deep OD notch.
- Mild steel simulated tube support.
- Stainless steel simulated tube support.
- Suspected ID intergranular corrosion.
- Kink.
- 0.015 in. deep ID notch.

The tester was adjusted to produce minimum signal output for the mild steel tube support on the lower trace in both Figures 8.1 and 8.2. In Figure 8.1 all of the signals are separate, distinct signals. Notice that even though discriminated against, the mild steel tube support produces a large residue signal on the lower trace. Next, the mild steel tube support was physically placed over the OD notch giving the results shown in Figure 8.2. Now, the iron tube support signal and the OD notch signal appear together, and the OD notch signal is unreadable.

Figures 8.3 and 8.4 are strip chart recordings of the output signals from the multiparameter eddy current tester operated simultaneously at 125 and 250 kHz. The same tube standard, simulated tube supports and test probe were used for these tests as were used for those related to Figures 8.1 and 8.2. The top trace in Figures 8.3 and 8.4 is a reference trace and is used only to show the relative position of the tube supports with respect to the notches. Notice in Figure 8.3 that all flaws and supports occur at distinct (separate) positions and that both of the tube supports signals have been discriminated
FIGURE 8.1. Two-Channel Output of a Single-Frequency Eddy Current Tester Showing Null Against the Mild Steel Tube Support Signal on the Lower Trace

FIGURE 8.2. Two-Channel Output of a Single-Frequency Eddy Current Tester When the OD Notch is Under with the Mild Steel Tube Support
FIGURE 8.3. Multiparameter Eddy Current Tester Output Showing Discrimination Against Mild Steel and Stainless Steel Tube Support Signals on the Lower Trace

FIGURE 8.4. Multiparameter Eddy Current Tester Output Showing OD Notch Signal and the ID Intergranular Corrosion Signal when the Mild Steel Tube Support is over the OD Notch and the Stainless Steel Tube Support is over the ID Intergranular Corrosion Flaw
against on the lower trace, leaving small residues. In comparing the lower trace of Figure 8.4 to the lower trace in Figure 8.3, notice that the OD notch signal and ID corrosion signal in Figure 8.4 are essentially the same as in Figure 8.3, even though the mild steel tube support has been moved over the OD notch and the stainless steel tube support has been moved over the intergranular corrosion area on the tube.

Figures 8.5 and 8.6 were made with the same test equipment, standards and probe as were Figures 8.3 and 8.4. The top traces in each figure are again only reference traces used for showing the position of the tube supports with respect to the notch signals. The lower trace in Figure 8.5 shows that both tube support signals and the OD notch signal have been discriminated against. The residues from these signals are very small. Here again the tube support signals and the notch signals are separate. In Figure 8.6 the stainless steel support has been moved over the intergranular corrosion flaw. Notice that the intergranular corrosion signal appears essentially the same on the lower trace in Figure 8.6 as it did in Figure 8.5.

Figures 8.7 and 8.8 are photographs of the complex voltage waveforms for all of the signals shown in Figures 8.1 through 8.6. Figure 8.7 was made using a test frequency of 125 kHz and a differential probe. Figure 8.8 was made using a test frequency of 250 kHz with the same probe. The probe motion axis is horizontal in each of these figures. The two largest signals in Figures 8.7 and 8.8 are from the tube supports. The stainless steel tube support signal is the largest in both figures. The magnitude of the stainless steel tube support signal is approximately 15 times larger than the largest notch or intergranular corrosion signal at 125 kHz. All of the notch, kink, and corrosion signals appear clustered at the center of the photograph. These two figures are intended to show the relative magnitudes and nonlinear nature of the signals.
FIGURE 8.5. Multiparameter Eddy Current Tester Output Showing Discrimination Against OD Notch Signals, Mild Steel Tube Support Signals and Stainless Steel Tube Support Signals on Lower Trace When Signals Occur Separately

FIGURE 8.6. Multiparameter Eddy Current Tester Output Showing the Intergranular Corrosion Signal When the Stainless Steel Tube Support Is Over the Intergranular Corrosion Flaw
FIGURE 8.7. Complex Voltage Waveforms for Single Frequency Eddy Current Test at 125 kHz

FIGURE 8.8. Complex Voltage Waveforms for Single Frequency Eddy Current Test at 250 kHz
ULTRASONIC METHODS

Phase Measurements of Reflected Ultrasonic Waves near the Rayleigh Critical Angle
F. L. Becker

The nature of the reflected ultrasonic wave at the Rayleigh critical angle has been the subject of much research. Several researchers\(^{(1,2,3)}\) have measured the sharp minimum which occurs at the Rayleigh critical angle but have been unable to explain this effect with a suitable model. Others\(^{(4)}\) have investigated the phase of the reflected wave for cases of low material attenuation or low frequencies. Through our research of the effects of attenuation on the propagation of ultrasound,\(^{(5)}\) we have developed a mathematical model\(^{(6)}\) which describes both the phase and amplitude of the reflected wave. Verification of the model's accuracy in predicting the amplitude has been previously reported.\(^{(7,8)}\) This work presents phase measurements which fully confirm the predictions of our model. These predictions are in agreement with those formulated by V. M. Merkulova\(^{(9)}\) and G. Mott.\(^{(10)}\)

These predictions differ substantially from nonattenuating theories in that the phase and rate of change in phase with incident angle are sensitive to attenuation and frequency in the reflecting medium. Above the frequency of least reflection, the phase no longer shifts from approximately 0 to +360°, rather it shifts from 0 to +60 then -60° and back to zero again. The frequency of least reflection (FLR) is the frequency for which the reflection at the Rayleigh critical angle is a minimum, approximately zero. This frequency has been found to be dependent on the shear wave attenuation per wave length in the reflecting media.\(^{(8)}\)

The phase and amplitude of a plane wave reflected at a liquid solid interface (as in References 6-8) can be written as follows:

8.8
\[
\frac{Z_{112}}{Z_{111}} = \left(\frac{C_{22}}{C_{12}}\right)^2 \frac{\sin^2 T_{221} \sin^2 T_{121} + \cos^2 2T_{221} + \frac{\rho_1}{\rho_2} \frac{C_{11}}{C_{12}} \frac{\sin T_{121}}{\sin T_{111}}}{\sin^2 T_{221} \sin^2 T_{121} + \cos^2 2T_{221} - \frac{\rho_1}{\rho_2} \frac{C_{11}}{C_{12}} \frac{\sin T_{121}}{\sin T_{111}}}.
\]

Equation (1) is very similar to those found in existing theories \(^{(1,11)}\) except that the velocities \(C_{ij}\) and angles \(T_{ij}\) can be complex, which results in a complex reflection factor. The amplitude and phase are the modulus and phase, respectively, of this complex number. The development of and solutions for this equation are given in detail in other reports.\(^{(6,8)}\)

Measurements of the change in phase of a reflected wave with angle requires that the ultrasonic path length be very stable; for example, an error of 0.001 in. would result in a phase change error of 92° at 15 MHz. To overcome this limitation, a dual angle goniometer was designed and constructed which has an error of less than ±80 μin. between 30 and 35°, which is equivalent to ±7° phase change at 15 MHz. This error was evaluated by measuring the phase shift from a brass sample which has a phase change of less than 1° over this angle range. The physical details and operation of this device are similar to that described by Fountain.\(^{(12)}\)

The phase was measured by the same technique described by VanBuren and Breazeale,\(^{(4)}\) with two exceptions: first, single frequency pulse bursts of 10 μsec duration were used and second, a vector voltmeter was used for phase readout. The use of the voltmeter has two advantages: (1) a calibrated analog output is obtained and (2) the actual phase lead or lag is measured, rather than the difference in phase (as in the case where Lissajous patterns are used). In this method, the phase of a cw reference signal is adjusted to match the received pulse,
and this shifted signal is then measured relative to an unshifted reference signal, point by point for each incident angle.

The feasibility of using a cw ultrasonic signal was also investigated and found to be equally suitable and much faster than the pulse method. In this technique, the phase of the cw signal, relative to a fixed reference, was read directly by the voltmeter and recorded automatically as the incident angle was varied. Within the limits of the stability of the measurement (approximately ±5° due mostly to vibration and electrical noise), the two methods gave the same results.

The results of the phase measurements for frequencies of 10, 14.5 and 18 MHz are shown in Figure 8.9 for Type 304 SS. The cw method was used for these curves. The reflected amplitude was also recorded to show its relationship to the phase at the Rayleigh critical angle (minimum amplitude point). At 10 MHz the phase changes from approximately 0 to 360° and is 180° at the critical angle. These results are similar to existing theories. Significant differences occur at higher frequencies. For a frequency of 14.5 MHz which is slightly below the FLR (note amplitude minimum), the slope becomes very steep. Above the FLR, 18 MHz, the phase rises to approximately +60°, falls to -60° and then approaches zero as the incident angle passes through critical.

It is easy to see that the phase change is indeed a phase lead by observing a pulse signal on an oscilloscope relative to a fixed reference. Figure 8.10 depicts these phase changes for small changes in the incident angle for frequencies of 10 and 18 MHz. Although the changes are difficult to determine from this figure, it is possible to show that the transient portion of these signals (first one or two cycles) does not move forward in time but that the steady-state portion does. Previous negative phase predictions(8) are actually consistent
FIGURE 8.9: Reflected Phase ($\phi$) and Amplitude for a Water Type 304 SS Boundary. Frequencies of 10, 14.5, and 18 MHz are shown.
**FIGURE 8.10.** Received Ultrasonic Signal Showing Phase Advance as the Incident Angle Is Varied Through Critical. Both 10 and 18 MHz Pulses Are Shown Relative to a Fixed cw Reference Signal.
with these results, if the definition of the traveling wave used is considered. The wave, exp $i\omega \left( \frac{N_c R}{c} - t - \phi \right)$, was used and in this system a negative $\phi$ actually represents a phase lead in time.

The anomalies (slope changes and reversals) noted above can be explained by using a model such as Equation (1) which considers the attenuation in both media. The predicted and measured phase changes in the area of the critical angle are shown in Figure 8.11 for Type 304 SS in water. The attenuations and other material constants used in these calculations are given in Reference 8. Two facts should be noted from this figure. First, the general shapes of the predicted and measured phases are very similar. Second, the slope of the phase curve decreases for frequencies away from the FLR. The difference in angular position of the measured curves is caused by a thin surface layer of work-hardened material (which occurred during the sample preparation) and results in a critical angle shift.\(^{(13)}\) This shift is dependent on the frequency, as the depth of penetration of these waves depends on the wave length.\(^{(14)}\)

The otherwise good agreement between measured and predicted phase along with the previously verified amplitude relationships substantiates the accuracy and applicability of the model. The sharp phase change for frequencies just below the FLR present several possibilities for materials testing or characterization. The Rayleigh critical angle has been shown\(^{(8,13)}\) to be sensitive to changes in material properties such as elastic modulus and density. The steep slope of the phase curve would be capable of much higher resolution than existing methods which use amplitude detection only.
FIGURE 8.11. Calculated and Measured Phase of Reflected Ultrasonic Waves at a Water Stainless Steel Interface. Frequencies of 10, 12, 14, 15, 18 and 20 MHz Are Shown.
Ultrasonic Measurement of Cold Work
R. L. Trantow

Our preliminary studies of the application of Rayleigh critical angle techniques to cold work and grain size determinations in Type 316 SS have established this test as a very sensitive indicator of a material's metallurgical state. The studies have also shown, however, that the Rayleigh critical angle and reflected beam attenuation are sensitive to specimen alignment and to changes in the transmitted beam configuration. Because of the high degree of curvature in the actual fuel cladding, the effects of alignment and beam geometry become very critical. Therefore, to ensure the accuracy of the critical angle measurement on our present flat plate specimens, and eventually on the actual fuel cladding, a two-part program was initiated. First to be developed was a reliable alignment procedure to be used with our present dual angle goniometer. The second part of the program, which is continuing presently, involves the study of the actual transmitted ultrasonic beam to determine its spatial energy distribution and to study the effect of that distribution on the reflected critical angle beam shape. This second part of the program will be discussed more fully here.

During early critical angle measurement of our characterized Type 316 SS flat specimens, we noticed that in some combinations of transmitter and receiver transducers, the critical angle null region of our data became distorted. In some cases, the distortion would reach proportions where multiple or false null points could be obtained. Our initial studies into the causes of this distortion were directed toward scanning the transmitter beam to determine if anomalies in beam symmetry or uniformity were present. One of our 1-in. diameter transducers (one that had presented some problems in system alignment and data uniformity) was selected for beam scanning. An isometric
representation of the energy distribution in a plane normal to the beam axis and 3.9 in. from the transducer face is shown in Figure 8.12. In this representation, the high amplitude peaks have been clipped to more clearly show the lack of symmetry and beam nonuniformity. This anisometric effect is probably due to areas of reduced sensitivity caused by lense unbonds or dead spots in the transducer material. Near-field effects and internal reflections are highly probable contributors as this scanning distance is well within the near field region. However, their effects would be expected to produce a more uniform beam interference. Although the reflected critical beam resulting from this transducer has not been scanned as yet, it appears safe to assume that the perturbations in the transmitted beam will be present in the reflected beam. Therefore, their presence will partially explain the distortion and multiple nulls in the critical angle data which were taken. An example of a uniform and symmetric beam energy distribution is shown in Figure 8.13.

![Energy Distribution Scan](image1)

**FIGURE 8.12.** Energy Distribution Scan of a 15 MHz 1 in.-Diameter Transducer with Tone Burst Excitation

![Symmetrical Energy Distribution](image2)

**FIGURE 8.13.** Symmetrical Energy Distribution of a 15 MHz 1/4 in.-Diameter Transducer with Tone Burst Excitation
This photograph shows an isometric representation of the energy distribution from a smaller, 1/4-in. diameter PZT transducer driven by a 15 MHz tone burst. As the scanning distance is approximately 4 in. away from the transducer face coinciding with the first beam maximum, this scan is in the far-field zone. The critical reflection resulting from bouncing this beam off a stainless steel block has also been scanned (see Figure 8.14). The notch or saddle between the two peaks is the result of critical reflection, and as the critical angle of incidence is approached, it can be seen to sweep through the peak of reflected energy. The null occurs over a very small range of incident beam angle and is, therefore, a very good indicator of the critical reflection condition. It is interesting to note that there are a series of secondary peaks or interferences scattered about the bases of the two major peaks. These are possibly caused by reflections within the specimen and/or by transducer near-field effects since in this experiment the beam-plate interception point occurred well within the transmitted beams near-field zone.

Since the possibility still exists that these side lobes may interfere with the saddle position and with the minimum amplitude though superposition of energy, we attempted to minimize the near-field effect by applying a pulse rather than a tone burst to the transmitter transducer. The energy distribution resulting from a 10 MHz period pulse applied to the same 1/4-in. transducer discussed above is shown in Figure 8.15. The energy distribution now became much broader and very smooth. The scan of critical reflection energy distribution of the initial beam is shown in Figure 8.16. The scale of this scan is the same as all previous scans shown in Figures 8.13 through 8.15. A broad smooth saddle point appeared and seemed to be free of extra interfering radiation lobes. Although the pulsed critical angle technique appears
**FIGURE 8.14.** Energy Distribution of the Critical Reflection of the Beam Described in Figure 8.13

**FIGURE 8.15.** Energy Distribution of the Transducer with a 10 MHz Period Pulse Excitation

**FIGURE 8.16.** Energy Distribution of the Critical Reflection of the Beam Described in Figure 8.15
to yield the most uniform and interference-free data, it must also be realized that the position and depth of the critical angle null point is frequency sensitive and that a pulse has high harmonic content.

Flaw Identification and Characterization
N. E. Dixon

An ultrasonic signal reflected from an object is believed to contain all the necessary geometric properties of the object such as size, shape, contour, etc. and that by processing the data in an appropriate manner (unknown as yet) this information can be extracted. The present approach to accomplish this consists of utilizing broad bandwidth pulses for interrogation of the objects and computerized spectral analysis techniques for identification and characterization.

In order to determine if the size, shape, etc. of parameters of an object can be extracted from the data, a series of experiments were undertaken which made use of three very simple flaws. These were a series of flat disks, spheres, and ribbons. The diameter of the disks were 0.030, 0.050, and 0.075 in. in diameter. The spheres were 0.150 in. in diameter and the ribbons were 0.025 × 0.300 and 0.050 × 0.300 in. Each of the flaws was embedded in the center of a 2-in. glass cube.

Figures 8.17 through 8.23 show a partial set of data from a pulse with a relatively broad bandwidth reflected from each of three types of flaws. In Figure 8.17, the photo insert on the computer spectral content graph shows the received pulse from a back surface of a glass block without a flaw and is intended as a reference signal. This type of reflector reflects the entire beam from which a normalized spectrum can be obtained. The spectral frequency coordinate (horizontal) is from 1 through 40 MHz. The peak spectral amplitude, (vertical coordinate) was normalized to one on the spectral distribution graph.

8.19
FIGURE 8.17. Received Pulse Displayed on a Sampling Scope from the Back Surface of a 1 in. Glass Plate and the Computer Analyzed Power Density Function from 1 Through 40 MHz
FIGURE 8.18. Received Pulse from 0.030 in. Diameter Disc 1 in. Deep in Glass and the Power Density Function from 1 Through 40 MHz
FIGURE 8.19. Received Pulse from 0.050 in. Diameter Disc 1 in. Deep in Glass and the Power Density Function from 1 Through 40 MHz
FIGURE 8.20. Received Pulse from 0.075 in. Diameter Sphere 1 in. Deep in Glass and the Power Density Function from 1 Through 40 MHz
FIGURE 8.21. Received Pulse from 0.150 in. Diameter Sphere 1 in. Deep in Glass and the Power Density Function from 1 Through 40 MHz
Neg 700548-4

FIGURE 8.22. Received Pulse from 0.025 by 0.300 in. Ribbon 1 in. Deep in Glass and the Power Density Function from 1 Through 40 MHz
**FIGURE 8.23.** Received Pulse from 0.050 by 0.300 in. Ribbon 1 in. Deep in Glass and the Power Density Function from 1 Through 40 MHz
Figure 8.18 shows the signal from a 0.030-in. diameter disc. Note the shape of the spectral distribution including the relative increase in the high frequency components as compared to Figure 8.18. Figure 8.19 shows the signal from a 0.050-in. diameter disc. Note the relative increase in amplitude in the lower spectral frequency components compared to Figure 8.18. The relative signal amplitude between Figures 8.18, 8.19 and 8.17 are noted as an attenuation factor just beneath the photo insert -- the higher attenuation (Figure 8.17) being the stronger signal.

Figure 8.20 shows the signal from a 0.075 in. diameter sphere. Note the spectral peaks at approximately 3.6 MHz intervals and the overall spectral pattern change compared to the flaws of Figures 8.17 through 8.19. Figure 8.21 shows the signal from a 0.150-in. diameter sphere. Note the difference in the spectral peaks which are now approximately doubled those in Figure 8.20. Also, the lower frequency spectral components of Figure 8.21 have increased in the overall curve shape compared with Figure 8.20.

Figure 8.22 shows the signal from a 0.025 × 0.300-in. ribbon. Note the change in spectral pattern shape compared to the disc, (Figures 8.18, 8.19) and the sphere, (Figure 8.20, 8.21). Also, the lower spectral frequency content increases compared to a disc or sphere. Figure 8.23 shows a 0.050 × 0.300-in. ribbon. Note the broader spectral peaking bands compared to Figure 8.22 and the increase in relative amplitude.

These and associated data indicate the following:

- Spectral information from broadband-width ultrasonic pulses reflected from flaws appears to be a basis for determining flaw size and geometry.
• Further development and optimization of pulse techniques and understanding of broadband-width pulse-wave propagation behavior is mandatory for practical implementation of the spectral analysis techniques for flaw identification.

THERMAL METHODS
D. R. Green

Research and development on new thermal and infrared techniques is continuing, and further applications of the techniques have been made. These applications include testing FFTF fuel pin simulators and carbon/carbon-composite cones. A full-scale experimental prototype of a thermal-image transducer for testing conical specimens was fabricated for Sandia Laboratories.

Figure 8.24 shows the full-scale, conical thermal transducer. Air pressure is used to press the transducer surface into close contact with the conical test specimens. Testing the area covered by the transducer requires 3 sec, not including the time required to get the cone into position. A bank of ultraviolet lamps and three solenoid-operated cameras that are used to record the thermal image are not shown in Figure 8.24 so that the details of the transducer itself can be seen.

Automatic analysis of data from thermal-image transducers requires rapid return to equilibrium upon terminating the heating pulse. Previous attempts to perform automatic image analysis were made on an air-convection cooled transducer. Because equilibrium was attained too slowly with this type of transducer, it was redesigned to incorporate water cooling. In addition to allowing automatic data analysis, the water cooling allows a test to be repeated sooner, if desired. Excellent images of defects were obtained with the water-cooled transducer. However, the thermally sensitive phosphor used to produce the image darkened after a few tests. We believe
FIGURE 8.24. Full-Scale, Experimental Prototype of a Thermal-Image Transducer for Testing Conical Specimens
that this darkening is due to volatile materials, contained in the binder used with the phosphor, that are vaporized by the heat during a test and condense on the water-cooled, optically-transparent transducer backing. Phosphor applied to the transducers is presently being outgassed at elevated temperatures in a vacuum to reduce the concentration of volatile vapors.

High frequency response is desired in the thermal image transducer so that it can be applied to test bonds under thin coatings. A special low-thermal inertia foil for use in the transducers has been obtained. This foil should improve the frequency response of the thermal transducer so that it exceeds 25 Hz. Methods of mounting the foil and coating it with thermally sensitive phosphor are being developed.

Another application for the thermal transducer method is emittance-independent, noncontacting measurement of surface temperature. The thermal transducer comprises a thin, blackened foil suspended between the specimen surface and a backing as shown in Figure 8.25. Blackening of the foil is not essential, but increases sensitivity. The backing surface is water cooled to provide a constant temperature of 25 °C. We will assume values of surface temperatures and emittances as shown in Figure 8.25 for the purpose of computations to demonstrate the feasibility. In a practical surface temperature transducer, such assumptions would not be necessary, as we shall see later.

![Figure 8.25](image)

**FIGURE 8.25.** Physical Arrangement of the Thermal Transducer and Specimen of Which the Surface Temperature is to be Measured
If $T_1 = T_3$, all of the heat flow, $q$, must pass through thermal resistance $R_1$ (Figure 8.25) to the backing, since the temperature drop across $R_2$ is zero and thus no heat flows through it. Let us consider the foil temperature that results from various amounts of electrical power injected into the foil, assuming for the moment that the specimen temperature is always somehow adjusted to be equal to the foil temperature. (The reason for this apparently trivial assumption will become clear later.) Then, at equilibrium

\[
\text{Electrical Power into Foil per Unit Area} = \frac{\text{Radiant Energy Lost per Unit Area From Foil to Cold Backing}}{1}
\]

assuming the foil is large enough and close enough to the backing and to the specimen that edge effects do not occur in the center of the foil (where the foil temperature is monitored). Values of foil temperature that result from some various amounts of injected electrical energy are tabulated in Table 8.1.

**TABLE 8.1. Foil Temperatures Resulting from Electrical Heating, $q$, When the Specimen Temperature and Foil Temperature are Equal**

<table>
<thead>
<tr>
<th>$q$, W/cm²</th>
<th>$T_1$, °F</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.100</td>
<td>716</td>
</tr>
<tr>
<td>0.115</td>
<td>755</td>
</tr>
<tr>
<td>0.135</td>
<td>807</td>
</tr>
<tr>
<td>0.152</td>
<td>850</td>
</tr>
<tr>
<td>0.175</td>
<td>894</td>
</tr>
</tbody>
</table>

These temperature values are approximate; they were calculated on a radiation slide rule to save time.

Now, consider the foil temperature resulting from electrical power input when the specimen temperature is not equal to the foil temperature. In this case,
Energy Injected + Foil Radiantly from Specimen per Unit Area or

\[ q + \frac{1}{\varepsilon_1 + \frac{1}{\varepsilon_3} - 1} \sigma (T_3^4 - T_1^4) = \frac{1}{\varepsilon_1 + \frac{1}{\varepsilon_2} - 1} \sigma (T_1^4 - T_2^4). \] ...

(2)

Again, as above, we tabulate the resulting values of foil temperature resulting from electrical heating. However, this time we must assume a specimen temperature and surface emittance as indicated in Tables 8.2 and 8.3.

**TABLE 8.2.** Foil Temperatures Resulting from Electrical Heating, \( q \), When the Specimen Temperature and Emittance are 824 °F and 0.10, Respectively

<table>
<thead>
<tr>
<th>( q, \text{ W/cm}^2 )</th>
<th>( T_1, ^\circ\text{F} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.100</td>
<td>770</td>
</tr>
<tr>
<td>0.115</td>
<td>796</td>
</tr>
<tr>
<td>0.135</td>
<td>822</td>
</tr>
<tr>
<td>0.152</td>
<td>841</td>
</tr>
<tr>
<td>0.175</td>
<td>862</td>
</tr>
</tbody>
</table>

**TABLE 8.3.** Same as Table 8.2, but Specimen Surface Temperature and Emittance are 716 °F and 0.10, Respectively

<table>
<thead>
<tr>
<th>( q, \text{ W/cm}^2 )</th>
<th>( T_1, ^\circ\text{F} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.100</td>
<td>716</td>
</tr>
<tr>
<td>0.115</td>
<td>739</td>
</tr>
<tr>
<td>0.135</td>
<td>767</td>
</tr>
</tbody>
</table>

Figure 8.26 shows a plot of the foil temperatures as a function of electrical heating for the loaded case (the case where specimen temperature is not necessarily equal to foil temperature) and nonloaded case (where specimen temperature is always equal to foil temperature). The shaded areas represent a band of error of about ±4 °F resulting from the use of a radiation...
slide rule in the calculations. More precise computations could easily be carried out, but would not add anything to the discussion here.

![Graph](image)

**FIGURE 8.26** Foil Temperature Versus Electrical Power Injection

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8.33
As we would expect, the loaded and nonloaded transducer curves in Figure 8.26 cross at the point where the foil temperature and specimen temperature are equal. Hence, if the electrical power input to the foil is swept through a range of values, the corresponding loaded foil temperature will only arrive at an unloaded value when the foil temperature is passing through the sample temperature. The unloaded values of foil temperature are, of course, predetermined by placing the transducer foil near a specimen that is always maintained at a temperature equal to the foil temperature while the electrical input to the foil is swept through its range of values. In this manner, the transducer is calibrated according to its own internal resistance. The point at which the foil passes through the unloaded transducer response curve can either be determined graphically or by an automatic electronic method which uses an analog curve follower or a digital memory.

Specimen emittance has no effect on the temperature measurement, except for sensitivity, since the emittance influences only the slope of the loaded curves and not the crossover points between the unloaded curve and the loaded curves. In practice, the unloaded curve would be best determined experimentally rather than computed, since real materials do not always behave according to the Plank radiation function that was used in the computed example above.

The computed curves in Figure 8.26 are given only as evidence of the sensitivity that could be expected. Somewhat lower values of specimen emittance than those used in the computations are possible, but the sensitivity of the transducer should be more than adequate since the foil temperature should be measurable with an accuracy of better than ±1.0 °F.
NEW TECHNIQUES

Holographic Methods
F. R. Reich

Applications of holography in nondestructive testing have been successfully demonstrated, using both cw (continuous wave) and pulsed ruby laser illumination. The main effort has been directed toward reflection illumination holography and the ability to do classical interferometry with complexly shaped objects not optically smooth or flat. All holograms were made using either a cw helium-neon laser or a pulsed ruby laser with maximum power outputs of $50 \times 10^{-3}$ W and 0.5 joules, respectively. The ruby laser was operated only in the free lasing mode as the "Q" switched output power was not sufficient for film exposure.

Both real time and double exposure interferometric holograms were used in vibration analysis of a membrane and an audio speaker. Real time interference holograms refer to optical reconstructions where interference occurs between the real object and a reconstructed image stored on a hologram. In double exposure interferometric holography, the interfering images are both stored on the hologram plate. The reconstructed holograms of the speaker and membrane contained fringes produced by motion at the specific area marked by the fringe. The shape and intensity gradient of the node pattern indicate both amplitude and frequency response which may be used to evaluate the material or dimension of the vibrating object. In real time interferometric holography, the dynamic properties of the vibrating object can be studied quickly as the fringe structure is presented in real time.

Double exposure and real time holography was demonstrated to show strain induced by pressure in a vessel and by mechanically loading a sample. Both mechanical and material
properties can be studied under stress. Weld defects in FFTF reactor cladding pin weld samples were located with pressure-induced stress. Areas which appeared to be weakened or annealed by the welding process were also visible. The holographically noted defect region correlated with other NDT analyses previously applied to the cladding samples. By heating a sample, both thermal expansion and conductivity of the sample material can be studied with holography. The expansion, induced by heating, was studied with both real time and double exposure holography. Thermal gradients in a honeycomb constructed panel indicated bonding discontinuities. The thermal gradient was induced by heating and cooling the panel surface between exposures of a double exposure hologram. Fringes from an overall panel warping due to the localized heat source produced a fringe grid on the panel. This enhanced some of the smaller thermal gradients. Bonding discontinuities noted with holography correlated with other thermalgraphic testing methods.

REFERENCES


9.0 ATR WATER LOOP OPERATION AND MAINTENANCE

R. S. Hope

The Advanced Test Reactor reached full power of 185 MW for Cycle 1 on December 26, 1969 and completed the first half of the cycle on February 8, 1970. Accumulated exposure was 6833 MWD or 36.94 equivalent full power days. The reactor was restarted for Cycle 1B on February 12, 1970 and has accumulated 3700 MWD at 185 MW. Cycle 2 shutdown occurred March 4, 1970. Eighteen of the 20 full power days during Cycle 1B were without a reduction in power.

No changes were made in the ATR 1D-N pressurized water loop during reactor shutdown. The loop is being operated at 2000 psig, 520 °F, 70 gal/min, pH 10.00 ± 0.2 and oxygen concentration at less than 0.05 ppm. The loop contained 168 miniature stainless steel Fermi tensile specimens and 84 Zircaloy corrosion coupons. Total accumulated exposure at a peak fast flux of $1.2 \times 10^{14}$ nvt was $5.9 \times 10^{20}$ nvt.
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