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INTERIM BURST TEST RESULTS ON EBR-II
IRRADIATED FUEL PIN SECTIONS

R. L. Fish, J. J. Holmes and R. D. Leggett

April 1970

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Technology

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FFTF Fuels Department
FFTF Division

April 1970

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ABSTRACT

Burst tests were conducted at 900 °F on specimens prepared from 75% UO_2 -25% PuO_2 fuel pins clad with 0.250 in. OD $\times$ 0.016 in. wall Type 304 stainless steel (SS) (PNL-1-3 and 1-6) that were irradiated in EBR-II to ~ 8000 MWd/MTM at 8 to 9 kW ft. Argon gas was used to internally pressurize the fuel pin sections and the fuel was left intact.

Specimens taken from above the midplane but within the fuel column exhibited brittle, intergranular fracture at drastically lower stresses ($\sim 30,000$ psi) than would be expected from Type 304 stainless steel irradiated under similar conditions. The measurable strain in these specimens was less than 1%.

Fueled specimens from below the midplane and specimens from the fission gas plenum exhibited a ductile, transgranular fracture at stresses and strains normally expected.

Post-test examination of the burst test specimens included diameter measurements, optical metallography, electron microscopy, and shielded electron microprobe analysis. Fueled specimens from above the midplane contained M_{23}C_6 and sigma phase at the grain boundaries of the cladding whereas the cladding of specimens from below the fuel midplane was free of grain boundary precipitates. Fuel particles containing cesium and other fission products were observed on the inner surface of the cladding and one area, containing cesium but no fuel, was observed at a location ~ 8 microns inside the cladding.

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INTRODUCTION

The integrity of cladding for LMFBR applications has been studied in terms of fuel-cladding interactions using metallographic analyses.⁽¹⁻⁴⁾ While emphasis generally has been placed on estimating the extent and character of fuel-cladding interaction, little work has been conducted on effects of the interactions on the strength of the cladding.

In order to evaluate the effects of fuel interaction on cladding strength, burst tests were conducted on two mixed oxide fuel pins that were irradiated in the EBR-II.

SUMMARY AND CONCLUSIONS

Burst tests were conducted at 900 °F on specimens prepared from 75% UO₂-25% PuO₂ fuel pins clad with 0.250 in. OD × 0.016 in. wall Type 304 stainless steel (SS) (PNL-1-3 and 1-6) that were irradiated in EBR-II to ~8000 MWd/MTM at 8 to 9 kW ft. Argon gas was used to internally pressurize the fuel pin sections. The fuel was left intact in order that the effects of the fuel and fission products on the properties of the cladding could be determined.

Specimens taken from above the midplane but within the fuel column exhibited brittle, intergranular fracture at drastically lower stresses (~30,000 psi) than would be expected from Type 304 stainless steel irradiated under similar conditions. The measurable strain in these specimens was less than 1%.

Fueled specimens from below the midplane and specimens from the fission gas plenum exhibited a ductile, transgranular

fracture at stresses and strains normally expected for Type 304 stainless steel irradiated under these conditions, i.e., $\sim 60,000$ and $\sim 9\%$ strain.

Post-test examination of the burst test specimens included diameter measurements, optical metallography, electron microscopy, and shielded electron microprobe analysis. Fueled specimens from above the midplane contained $M_{23}C_6$ and sigma phase at the grain boundaries of the cladding whereas the cladding of specimens from below the fuel midplane was free of grain boundary precipitates. Fuel particles containing cesium and other fission products were observed on the inner surface of the cladding and one area, containing cesium but no fuel, was observed at a location ~ 8 microns inside the cladding.

From these test results and examinations it is concluded that:

- Sections of irradiated fuel pins may fail at much lower stresses and strains than would be expected from mechanical tests conducted on unfueled cladding material.
- The premature failure in certain sections of the pin is associated with grain boundary precipitation in the cladding.
- The precipitation is believed to promote chemical attack along the cladding boundaries. The corrosive specie is either a fission product or a reaction product from their interaction with the cell environment.

EXPERIMENTAL PROCEDURE

SPECIMEN DESCRIPTION

Mixed oxide (UO_2 -25% PuO_2), Type 304 SS clad (0.250-in. OD $\times$ 0.016-in. wall) fuel pins were irradiated in position 6C1 during cycles 26C through 28C (excluding 27G through 28A) and

accumulated about 8500 MWd/MTM burnup to the fuel (56.8 EFPD at ~9.0 kW/ft peak pin power) and approximately 5.0×10^{21} n/cm² (E > 0.1 MeV) peak fluence to the cladding. The irradiation temperature of the cladding ranged from 800 to 950 °F over the fueled cladding length with an average cladding radial ΔT of about 50 °F. The irradiation⁽⁵⁾ was performed in sodium-filled capsules of the MK-A-19 type (see Figure 1). Fuel and cladding compositions are given in Table 1.

TABLE 1. As-Fabricated Fuel and Cladding Compositions

Fuel: (UO₂-25 wt% PuO₂) Sintered Pellets

Element	Maximum Concentration, ppm	Element	Maximum Concentration, ppm
C	25	Mn	5
Cl	10	Na	2
F	25	Ni	10
Ag	0.1	P	50
Al	1000	Pb	20
B	0.5	Si	80
Ca	1	Sn	20
Cr	.25	Zn	100
Cu	200	V	20
Fe	160	K	20
Mg	5	Mo	20

Cladding: Mill annealed Type 304 SS
0.250 in. OD × 0.016 in. wall

Element	Concentration, %
C	0.056
Mn	1.73
P	0.038
S	0.011
Si	0.47
Ni	9.90
Cr	18.50
B	0.0005
N ₂	0.10
Ti	0.006
O ₂	0.0103
Al	0.002
Mo	0.29
Cb	0.030
Fe	balance

(EBR-II IRRADIATION)

Neg 701163-1

FIGURE 1. PNL-1 Fuel Pin and Capsule Assembly

BNWL-1323

Specimens 2-3/4 in. in length were cut by an abrasive wheel from positions above and below the fuel midplane as well as from the plenum region. The specimen location, identification, and temperature and fluence profiles are shown in Figures 2 and 3. Following the cutting operation, each specimen was prepared for pressure testing by attaching Swagelok[®] fittings to the ends of the specimen. It is important to note that fuel material within the specimen was not removed or intentionally altered in any way in order that the effects of the fuel and fission products could be determined.

BURST TESTING

Figure 4 illustrates the test apparatus used in this study. Each specimen was individually attached to a pressurization line and then sealed in a test vessel designed to totally contain the fission products that might be released during testing. The test vessel was then connected to a pressurization system and placed in a furnace at 900 or 1000 °F to thermally equilibrate. About three hours were required to stabilize the temperature. Each specimen was pressurized at 20 psi/sec until failure occurred. Failure was indicated by a drop in pressure within the specimen.

POST-TEST EXAMINATION

After failure was indicated, the test vessel was removed from the system and disassembled in a shielded alpha facility. The specimen was visually examined and photographed in several orientations. Measurements were made with micrometers in areas removed from the fracture in order to estimate the permanent strain that had occurred. Selected specimens were examined metallographically to determine the microstructural nature of fracture. Optical metallography was supplemented with extraction replication to detect possible precipitation within the cladding. Microprobe analyses were performed to evaluate the penetration of fission products into the cladding.

Neg 700746-2

FIGURE 2. PNL 1-3 Axial Fluence and Temperature Profile

Neg 700746-1

FIGURE 3. PNL 1-6 Axial Fluence and Temperature Profile

Neg 700668-4

FIGURE 4. Test Apparatus

RESULTS

Burst test results are included in Table 2. All three specimens taken above the midplane but within the fuel column failed at stresses significantly lower than the control value, while specimens from below the midplane burst at pressures either equivalent or slightly above the unirradiated control value. The fracture mode of specimens failing at reduced stresses was greatly different than for the other specimens tested (Figure 5). Stainless steel cladding burst tested at 900 to 1000 °F normally ruptures violently and produces a large amount of specimen distortion. Considerable transgranular tearing and fragmentation often occurs during a typical failure. In specimens containing fuel material above the midplane, the low stress failures were characterized by multiple pinhole failures along the length of the specimens rather than the expected explosive tearing. Once the pinholes penetrated the cladding, failure was indicated by leakage of the pressurizing gas. The values shown in Table 2 for samples exhibiting this latter type of failure represent maximum stresses. Cladding perforation possibly may have occurred at pressures substantially below those presumed to represent failure but not detected. A single small crack, for example, would leak gas slowly and would not be detected with this method of determining failure.

The microstructural features of the two failure types are shown in Figure 6. Specimens failing explosively show the characteristic necking near the fracture and transgranular mode of failure. Specimens from below the reactor midplane failed in this fashion. Samples from above the reactor midplane, however, showed practically no local deformation and failed in a completely intergranular mode. The grain boundaries

TABLE 2. PNL-1 Fuel Pin Burst Test Results

<u>SPECIMEN IDENTIFICATION</u>	<u>OPERATING TEMPERATURE</u>	<u>FLUENCE E > 0.1 MeV n/cm²</u>	<u>TEST TEMPERATURE</u>	<u>HOOP STRESS psi</u>	<u>STRAIN ΔD/D %</u>
CONTROL			900 ⁰ F	56,960	19.2
PLENUM REGION					
PNL 1-6:H ₁	~890 ⁰ F	~1.2X10 ²¹	900	59,090	18.4
ABOVE FUEL MIDPLANE					
PNL 1-6:G ₁	~920	~4.2X10 ²¹	900	22,020*	1.0
PNL 1-6:G ₂	~920	~3.0X10 ²¹	900	27,160*	0.3
PNL 1-3:G ₄	~945	~3.7X10 ²¹	900	38,900*	0.2
BELOW FUEL MIDPLANE					
PNL 1-6:C ₃	~850	~4.1X10 ²¹	900	72,150	9.6
PNL 1-3:C ₂	~860	~4.3X10 ²¹	1000	66,790	6.7

* MAXIMUM VALUES. BECAUSE OF THE NATURE OF THE FAILURE, CLADDING PERFORATION MAY HAVE OCCURRED AT SIGNIFICANTLY LOWER VALUES.

ABOVE FUEL MIDPLANE

CONTROL

PLENUM

BELOW FUEL MIDPLANE

Neg 700692-2

FIGURE 5. Fracture Mode of PNL-1 Fuel Pins

OBSERVED ABOVE FUEL MIDPLANE

ABOVE FUEL MIDPLANE

EXPECTED FAILURE MODE

ABOVE FUEL MIDPLANE

Neg 700692-3

FIGURE 6. Microstructural Differences in Failures of PNL-1 Fuel Pins

appeared to be very weak as evidenced by the loss of several whole grains within the cladding during the polishing operation.

Extraction replication revealed greatly different levels of grain boundary precipitation in specimens taken from above and below the midplane within the fueled region. Below the midplane, no precipitates were observed by the extraction technique (Figure 7). Similar results were obtained after aging identical material for 1300 hr at 900 °F. Above the midplane, heavy grain boundary precipitation was found (Figure 8). These precipitates were identified by electron diffraction as $M_{23}C_6$ and sigma phase.

Metallography on these fuel pins was performed to further characterize the precipitate formation in the cladding as it varied axially. Figures 9 and 10 illustrate the gradual increase in grain boundary precipitation in going from the lower to the upper parts of the fueled portion of these pins.

The primary difference in irradiation conditions between specimens above and below the midplane is irradiation temperature. The average irradiation temperature below the midplane was about 850 °F, while above the midplane the average temperature was around 920 °F. Aging identical material out-of-reactor at 800, 850, and 900 °F for 1370 hr (the time in-reactor) failed to produce substantial amounts of precipitation. This finding suggests either that the irradiation temperatures were much higher than those calculated or that other environmental factors present during the irradiation enhanced the grain-boundary precipitation. Possible environmental influences include:

- Irradiation enhanced diffusion⁽⁶⁾ of sufficient magnitude to allow mass transport adequate for the formation of large precipitates at 920 °F but not at 850 °F

EXTRACTION REPLICA - 304 S.S.
PNL 1-6 TUBE BURST TEST
SPECIMEN C-3

$\phi t \sim 4.1 \times 10^{21} \text{ n/cm}^2 \text{ (E > 0.1 MeV)}$
T IRRAD. $\sim 850^\circ\text{F}$
T TEST = 900°F

Neg 700692-4

FIGURE 7. Extraction Replica of Specimen from Below Fuel Midplane

Neg 700692-5

FIGURE 8. Extraction Replica of Specimen from Above Fuel Midplane

Neg 700668-2

FIGURE 9. Axial Variation in Cladding Microstructure of Fuel Pin Irradiated in EBR-II at 8.1 kW/ft to 7900 MWd/MTM (PNL 1-6)

Neg 700668-1

FIGURE 10. Axial Variation in Cladding Microstructure of Fuel Pin Irradiated in EBR-II at 8.8 kw/ft to 8200 MWd/MTM (PNL 1-3)

- Cladding stresses which assist precipitation
- Impurity pickup in the cladding from either the capsule components or the fuel.

The possibility that the actual cladding temperatures were higher than those calculated cannot be precluded, but analysis of the fuel structure⁽⁷⁾ tended to confirm the values reported in Table 1.

Use of a shielded electron microbe to identify a corrosive agent causing the intergranular failure has been inconclusive. Figure 11 illustrates the results of the examination of a fuel pin sample taken 1/2 in. below a burst test specimen which failed at a low burst pressure (specimen from above the mid-plane). The microprobe display indicates the finding of uranium, plutonium, cesium, iodine, barium, and tellurium in a discontinuous layer along the inside of the cladding.

Cesium was observed within the cladding in only one area. Three scans, AA, BB, and CC in Figure 11 were taken across this area, and the variation of concentration with arbitrary distance is summarized in subplots A, B, C, and D in Figure 11 for cesium, uranium, iodine, iron, chromium and nickel. It is significant that the cesium distribution shows an increased concentration at about 8 μ inside the cladding (at about the 32 μ position) and that little or no fuel was found at this position. This result is illustrated by the graphical plot "B" and by the higher magnification specimen current display. Plots "A" and "C" illustrate graphically the distribution of cesium in Lines AA and CC, respectively. The lower plot D, of Fe, Cr, and Ni shows the counting rate of these elements as scanned along Line BB. The counting rates are proportional to the respective concentrations. The inside edge of the cladding is judged to be at about 24 μ on the relative distance scale. The existence of the increased cesium concentration in this region of the cladding

SPECIMEN CURRENT DISPLAY, CLAD
IS AT BOTTOM OF PHOTOGRAPH,
FUEL IS OFF THE TOP.
BRIGHT BAND IS FUEL-FISSION
PRODUCT LAYER.

200 X 200 MICRONS

URANIUM*

PLUTONIUM*

CESIUM*

IODINE*

BARIUM*

TELLURIUM*

HIGHER MAGNIFICATION OF
SPECIMEN CURRENT DISPLAY
INCLUDING AREA OF BRIGHT
BAND CURLING INTO CLAD.

50 X 50 MICRONS

CESIUM*

A B C

* DISTRIBUTION (PULSES) SUPERIMPOSED ON SPECIMEN CURRENT

Neg 700668-3

FIGURE 11. Microprobe Results (PNL 1-6-F)

is thought to be significant. However, because excess cesium was found only in this area on the circumference of the cladding cross section, it is difficult to assign a major role to cesium in promoting the severe intergranular failure observed during burst testing.

DISCUSSION

The grain boundary failure at low stresses in specimens from above the midplane represents a marked departure from expected cladding behavior. Fast reactor irradiation of unfueled 304 SS under similar conditions does not lead to reduced fracture stress or premature grain boundary failure.⁽⁸⁻¹⁰⁾ Ductility is reduced somewhat but the fracture mode remains transgranular and failure proceeds by plastic instability.

The mechanism responsible for the drastic reduction in grain boundary strength observed in the present study is as yet unclear. The key difference between specimens from above and below the midplane appears to be the level of grain boundary precipitation. Since the failure mode is different at these two locations, grain boundary precipitation is probably an underlying factor. A number of possible explanations for boundary weakening in terms of boundary precipitates exist.

The irradiation induced precipitates may be much weaker, or may lack cohesion with the matrix and thereby provide an easy path for crack propagation. Similar precipitate morphologies created by thermal aging at temperatures near 1400 °F do not lead to low stress failure or drastically reduced ductility and grain boundary weakness.⁽¹¹⁾ This observation indicates that, unless the $M_{23}C_6$ and sigma phase created during irradiation are metallurgically different than that created by thermal aging, the precipitates are not directly responsible for the loss in boundary strength.

Grain boundary precipitation by changing the relative concentration of the alloy constituents near the boundary, results in reduced corrosion resistance.⁽¹²⁾ Corrosion or stress corrosion resulting from the loss in corrosion resistance could weaken the boundaries and produce the observed effects. The constituent causing the corrosion may come from several sources including hot cell environment, fission products within the cladding, or fission products contaminated by water absorption during sample preparation. With respect to the latter possibility, corrosion from the fission products may be the result of hydrolysis of such elements as cesium or rubidium which would not have occurred had the irradiated fuel been protected from moisture at all times.

The actual source of the possible corrosion is extremely important in the application of these results to nuclear technology. If the corroding agent is derived from the hot cell environment or is due to aqueous contamination of the fission products, then special care must be taken in the interpretation of postirradiation examination of irradiated fuel pins to be certain that observed effects are the results of irradiation and not handling. More important, special procedures must be taken in handling of fuel pins during postirradiation examination. Alternately, if the corrosion is attributable to uncontaminated fission products, then the cladding strength used in design must be greatly reduced from that derived from post-irradiation tensile and creep tests on nonfueled specimens.

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