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METALLURGICAL EXAMINATION OF BORON-
CONTAINING STAINLESS STEEL CLADDING
FROM INDIAN POINT CORE A

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**METALLURGICAL EXAMINATION OF BORON-
CONTAINING STAINLESS STEEL CLADDING
FROM INDIAN POINT CORE A**

**— FINAL REPORT —
Volume 3 of 3 Volumes**

by

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ABSTRACT

The type-304 stainless steel cladding from Indian Point Core was examined metallurgically. This cladding was of special interest because it contained 250 ± 100 ppm boron as distributed poison. The evaluation included visual examination, corrosion tests in boiling HNO_3 , potentiostatic polarization tests, creep tests, and electron microscopic examination of thin metal foils and of replicas from fractured surfaces of both irradiated and nonirradiated material. The corrosion and electrochemical tests showed that irradiation greatly increased the corrosion attack at grain boundaries. The mechanical testing and electron metallography showed brittle behavior in the temperature range of 600 C.

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1. INTRODUCTION

1.1. Background

This report describes the results of a metallurgical examination of the cladding of the Indian Point Core A. The Indian Point Reactor (IPR) is operated by Consolidated Edison at Indian Point, New York. Core A, the first core of this reactor, began fullpower operation in 1962 and was discharged on October 23, 1965. The examination was conducted by The Department of Metallurgical Engineering, Ohio State University, under contract to The Babcock & Wilcox Company (B&W Contract 80625Z issued pursuant to AEC prime contract AT-(30-1)-3809). This report is the third and last volume of the final report under the prime AEC contract. Other related reports issued by B&W as accounts of work conducted in B&W facilities are as follows:

BAW-3809-1, Progress Report No. 1, March-August 1967.

BAW-3809-2, Progress Report No. 2, September-November 1967.

BAW-3809-3, Progress Report No. 3, December 1967-February 1968.

BAW-3809-4, Progress Report No. 4, March-May 1968.

BAW-3809-5, Progress Report No. 5, June-August 1968.

BAW-3809-6, Examination of Stainless Steel-Clad ThO_2 - UO_2 Fuel Rods and Zircaloy-2 Can After Operation for 442 Effective Full Power Days in the Indian Point Reactor—Final Report, Volume 1.

BAW-3809-7, Postirradiation Examination of Thoria-Urania Fuel Rods—Isotopic Analysis of the Fuel—Final Report, Volume 2.

The specific objective of this program was to evaluate the metallurgical performance of the stainless steel cladding, which contained 250 ± 100 ppm boron as a distributed poison. Presumably, the helium produced from the nuclear reaction of the boron ($n + {}^{10}\text{B} \rightarrow {}^4\text{He} + {}^7\text{Li}$) acts to produce internal bubbles. The interaction of these bubbles with the structure should be of substantial significance to cause concern over the metallurgical integrity of cladding for fast breeder reactors.

The major portion of the experimental work was conducted by Battelle Memorial Institute under subcontract to OSU. The Battelle work was conducted under their Serial No. 1285RR as authorized by OSU purchase order RF206246, dated May 18, 1968. The principal investigators were R. W. Staehle of OSU and R. A. Wulleart, J. R. Lombard, and J. J. Perrin, all of BMI.

Unique aspects of this core were the ThO_2 - UO_2 fuel and a stainless steel cladding containing distributed boron poison. The IPR Core A fuel loading was a mixture of thorium and enriched uranium. The fuel was in the form of pellets, which were sealed into type-304 stainless steel tubing to form the fuel rods. The stainless steel cladding (ASTM A 213 type-304) contained 250 ± 100 ppm natural boron as a burnable poison. The core was in the reactor for about 1130 days and at temperature for about 20,000 hours. During this time, the core accumulated about 442 EFPD of operation and achieved peak burnups of approximately 35,000 MWd/T. The cladding was exposed to fast fluxes up to 3×10^{21} nvt (>1 MeV).

Significant physical parameters of the core are as follows:

Stainless Steel Cladding

Number of bundles	120
Rods per bundle	195
Outside diameter, in.	0.320
Wall thickness, in.	0.020
Alloy	ASTM A 213 TP-304
Boron, ppm	250 ± 100
Peak fast fluence, nvt	3×10^{21}
Peak heat flux, Btu/h-ft ²	538,000
Coolant outlet temperature, F	579

Zirconium Alloy Channels

Number of channels	120
Wall thickness, in.	0.155
Alloy	Zircaloy-2
Peak fast fluence, nvt	3×10^{21}

The essential portions of this program were: visual inspection of as-received cladding, Huey tests, potentiostatic polarization tests, tensile tests, creep tests, fractographic examination of various fractured surfaces, and transmission electron microscopy. The work was conducted by BMI personnel except for (1) the polarization studies, which were made by Dr. Robert Cowan (formerly a graduate student at OSU) at the BMI West Jefferson facility, and (2) the tensile testing, which was done at B&W's laboratories in Lynchburg, Virginia.

1.2. Metallurgical Problem of Bubbles in Fuel

The problem of radiation-induced embrittlement of austenitic stainless steels at elevated temperatures has been of major concern to reactor technologists over the past decade. In general, this embrittlement is characterized by a drastic loss of ductility and minor changes in strength. Embrittlement normally occurs above 500 to 700 C for stainless steels, and, unlike irradiation-induced displacement damage, cannot be removed by annealing. The failure mechanism for irradiated material is by brittle intergranular fracture, whereas unirradiated material normally fails by ductile transgranular fracture under the same conditions.

Radiation-induced embrittlement at elevated temperatures has generally been attributed to the presence of helium. The helium is produced by (n, α) reactions of the boron impurity with thermal neutrons, or it is generated by the fast-neutron reactions with the iron, nickel, and chromium in the matrix. The helium produced by the (n, α) reactions is believed to coalesce into bubbles at grain boundaries, where it promotes intergranular fracture.

Although the mechanism of the original bubble formation is not fully understood, the formation of bubbles is believed to be inevitable, since the solubility of helium in the matrix is thought to be negligible.

The mechanism of the helium movement in metals, including bubble agglomeration, has been discussed in detail by several investigators.^{1,2} Various investigations have reported observing helium at grain boundaries, on dislocations, at precipitates, and dispersed throughout the matrix.³⁻⁷ It is generally acknowledged that about 1×10^{-5} atom fraction of helium must be produced during irradiation to cause drastic embrittlement.

Although helium from (n, α) reactions is the generally accepted source of radiation-induced embrittlement at elevated temperatures, the actual mechanism is not completely understood. There is disagreement on whether the helium bubbles cause grain-boundary fracture by matrix strengthening or by grain-boundary weakening. An unanswered question concerning helium-induced embrittlement is the failure to correlate the helium content generated to the bubble densities observed. Since the helium content that is generated cannot account for all the "voids or helium bubbles" observed, some investigators have suggested that the "voids" are actually large defect clusters (which may contain some helium) resulting from the condensation of vacancies. Obviously, further investigations will be required to gain a thorough understanding of the embrittlement phenomenon.

Since the boron content of the cladding material from IPR was so high, large amounts of helium were generated, and almost all of it originated from the boron. This offered an ideal opportunity to study the role that helium plays in elevated-temperature embrittlement and formed the basis for this investigation.

2. CONCLUSIONS AND RECOMMENDATIONS

It was observed that irradiation greatly increased the rate of grain boundary corrosion at electrochemical potentials higher than those expected during normal service. No intergranular corrosion of the outside fuel cladding surface was observed during reactor operation. The accelerated intergranular corrosion of irradiated material is considered to be produced by in situ sensitization (i.e., chromium carbide precipitation) due to radiation enhanced diffusion, which renders the grain boundary regions less resistant to corrosion.

The boron in these fuel elements accelerated the intergranular attack over that in nonborated stainless steel studied in COO-1319-30.⁸ This result may, in fact, explain the intergranular attack of the General Electric fuel elements studied in their high-power-density program (also evaluated in COO-1319-30, OSU evaluation report). It is reasonable that the higher oxygen content in the boiling water reactor environment may actually raise the electrochemical potentials barely into the range where there is a tendency for the intergranular attack described herein to occur.

A further important consequence of this corrosion work is the implication regarding chemical cleaning of irradiated cores. Any stainless steel structural material having a fluence in the range described herein might be expected to corrode catastrophically if a highly oxidizing cleaning agent were used. This matter is clearly important to all water-cooled thermal reactors in which stainless steel components can accumulate significant fluences.

The boron in the cladding seems to cause a nonrecoverable loss in elongation as observed in the normal tensile test on irradiated tubing. Creep testing of irradiated cladding showed that irradiation causes a clear loss of creep-rupture ductility. Fractographs showed ductile-brittle transitions in the range of 600 C. In irradiated specimens, the

general effect of the boron was to reduce ductility beyond that normally seen in nonborated material.

Intergranular attack and voids observed on the inside diameter of the fuel rods is attributed to a combination of slight sensitization and poor rinsing of the pickling solution during manufacture of the cladding.

3. EXPERIMENTAL

3.1. Inspection of Nonirradiated Tubing

The control material was type-304 stainless steel tubing containing 250 ppm natural boron. The tubes had an outside diameter of 0.304 inch and a wall thickness of 0.0205 inch. Two as-received control tubes, approximately 3-1/2 inches long, and two annealed control tubes (3000 h at 316 C) of the same length were used. The as-received tubes were designated as AR-1 and AR-2, and the annealed tubes as 3A-1 and 3A-2. The data shown in the figures associated with the text are representative of the effects of irradiation on the stainless steel cladding. Additional supporting data are shown in the figures of the appendix.

Metallographic mounts in the transverse direction of the control material were prepared using standard mounting and polishing techniques. The specimens were nickel-plated to preserve the edges for inspection. Photomicrographs were taken of the material in both the as-polished and the etched condition. These photomicrographs are shown in Figures 1 and 2.

Examination of the photomicrographs shows that both the annealed and the as-received control materials are quite "dirty," containing a large number of evenly dispersed precipitates. However, the grain boundaries appear to be relatively clean in most cases. The primary differences between the as-received and the annealed specimens is the larger grain size and the occurrence of more twins in the annealed material. No unusual edge effects were observable. Measurements with a Filar eyepiece at 100X showed the wall thickness to be between 0.0192 and 0.0195 inch.

3.2. Inspection of Irradiated Cladding

The shipment of irradiated specimens consisted of 10 vials containing eight tube specimens and 10 tensile specimens:

<u>Shipping vial No.</u>	<u>From pin No.</u>	<u>Specimen No.</u>	<u>Tube specimen</u>	<u>Tensile specimen</u>	<u>Burnup zone</u>
1	105-C	16	X		Avg
1	106-B	11		X	Low
2	106-A	16	X		Low
2	106-B	11		X	Low
3	106-A	15	X		Low
3	106-B	13		X	Low
4	105-B	19	X		Avg
4	106-B	13		X	Low
5	105-B	23	X		High
5	105-D	14		X	High
6	105-B ²	17	X		High
6	105-D	14		X	High
7	105-B ²	21	X		High
7	105-D	16		X	High
8	105-B ²	24	X		High
8	105-D	16		X	High
9	105-D ²	18		X	High
10	105-D ²	18		X	High

The cask was unloaded and the specimens were examined. The tubes appeared to be free of fuel particles, but had not been deburred after cutting. Most tubes contained small nicks and scratches from handling. Several received helical and circumferential mars during removal of the pins from the bundles.

The tensile specimens had not been deburred, and the radii were not cut symmetrically on either side of the gage section. One specimen was received in a bent condition and therefore was not suitable for testing. The outer surfaces of the specimens were covered with a dark scale. The inner surfaces quite often contained surface stains and occasionally small clusters of pits. Figure 3 shows typical low-magnification pictures of the irradiated tubes and tensile specimens.

Transverse metallographic mounts were made of samples selected from areas of low (specimen 105-C-16), medium (specimen 106-A-16), and high fluence (specimen 105-B-23). Specimens were mounted in epoxy and ground flat on SiC grinding paper with grit sizes ranging from 120 to 600. Initial polishing was done on 1/2-micron diamond mylor

cloth using kerosene as a carrier. The final polishing was done using Linde-A powder on micro cloth with water as the carrier. Good edge retention was obtained with the diamond polish. An etchant consisting of 20 parts glycerin: 20 HCL: 4 HNO₃ was used. These photographs are shown in Figures 4 through 8.

There was no significant difference in the microstructures of low-, medium-, and high-fluence specimens. The most pronounced difference between the irradiated and the control specimens was that the grain size of irradiated specimens was approximately 5 to 10 times smaller than that of control specimens. Also of importance is the existence of voids and cracks on the inside surfaces of the cladding tubes. This surface condition, also noted by B&W (Figure 7), did not seem to be dependent on the level of fluence or the position of the specimen in the reactor. The attack is associated with the grain boundaries and extends about 0.001 to 0.003 inch in from the tube's inner surface. In contrast, the outer surfaces of the tubes are characteristically smooth and unattacked (Figure 8).

Measurements of the wall thickness of the three irradiated specimens ranged from 0.0199 to 0.0204 inch, indicating that there was little or no overall change during operation in the reactor.

3.3. Specimen Preparation

To ensure use of the same procedures, both control and irradiated tubes were sectioned and the specimens prepared at Battelle's hot cell. Sectioning was done slowly with a fine cut-off wheel using carbon tetrachloride as a lubricant-coolant. A typical tube-sectioning diagram is shown in Figure 9.

The types of samples that were prepared are as follows:

As-Received Control Tubes

One 1/2-in. —long cylinder (metallography sample).

Two 3/4-in. —long cylinders (Huey test samples).

Two 1/4-in. —long cylinders (potentiostatic polarization samples).

Two 1-in. —long split tube halves (electron microscope samples).

Annealed Control Tubes

- One 1/2-in. —long cylinder (metallography sample).
- Two 3/4-in. —long cylinders (Huey test samples).
- Two 1/4-in. —long cylinders (potentiostatic polarization samples).
- Two 1-in. —long split tube halves (electron microscopy samples).

Low-Fluence-Irradiated Tubes

- One 1/2-in. —long cylinder (metallography sample).
- Two 3/4-in. —long cylinders (Huey tests).
- Two 1/4-in. —long cylinders (potentiostatic polarization samples).

Medium-Fluence-Irradiated Tubes

- One 1/2-in. —long cylinder (metallography sample).
- Two 3/4-in. —long cylinders (Huey tests).
- Two 1/4-in. —long cylinders (potentiostatic polarization samples).

High-Fluence-Irradiated Tubes

- One 1/2-in. —long cylinder (metallography sample).
- Two 3/4-in. —long cylinders (Huey tests).
- Two 1/4-in. —long cylinders (potentiostatic polarization samples).
- Two 1-in. —long split tube halves (electron microscopy samples).

Once the specimens had been sectioned, they were deburred using a center drill on the inside tube edge and a file on the outside tube edge. Any remaining fuel particles were removed by passing a small wire brush through the tubes.

3.4. Huey Tests

The Huey test is a standard test used to measure the susceptibility of austenitic stainless steels to intergranular attack. It is conducted in boiling HNO_3 and is not intended to indicate the performance of the 304 stainless steel cladding in other corrosive environments.

In this program, Huey tests were conducted in duplicate on five samples: two (AR-1) as received, two (3-A-1) annealed for 3000 h at

316 C, two (106-A, No. 16) low fluence, two (105-C, No. 16) average fluence, and two (105-B, No. 23) high fluence. The deburred 3/4-in.-long tube specimens were cleaned by immersion in boiling 28 wt% HNO_3 for 2 hours followed by rinsing several times in distilled water and then in acetone. The samples were then weighed on a precision balance. An identical procedure was used for the control specimens.

The test apparatus was a 50-ml Erlenmeyer flask fitted with a condenser. A hot plate was used to bring the solutions to a boil. In total, five of these units were set up—two control samples and three irradiated samples (Figure 10).

Enough 28 wt% HNO_3 * was prepared for conducting three 48-hour tests on all the samples. A total of 250 ml of solution was used in each flask. After initial cleaning and weighing, the samples were boiled for 48 hours. Samples were rinsed in distilled water and then in acetone before final weighing. After each run the Erlenmeyer flasks were cleaned in Alconox and rinsed several times with water and finally alcohol. The results of these tests are tabulated in Table 1.

The four control samples had a fairly consistent dissolution rate. The rate gradually increased for each run, probably because of an increase in exposure area resulting from the newly formed irregular surface. The irradiated specimens were almost completely dissolved during the first 48-hour period. The dissolution rate was directly related to the exposure; the high-fluence specimens were completely dissolved, only residue remained from the medium-fluence specimens, and fragile skeletons remained from the low-fluence specimens.

Since these results were unexpected, short-term Huey tests were rerun on two high-fluence specimens (105B-24 and 105B-24(N)). Specimen 105B-24 was run for 6 hours and then removed for metallographic examination; specimen 105B-24(N) was run for 21 hours before being examined. The results of these short-term tests—adjusted to a base of 48 hours—are shown in Table 1.

- - - - -
* The usual Huey testing procedure involves 65% HNO_3 . This concentration was used inadvertently in an initial test. Thereafter, it was decided that all testing would be performed at the same concentration.

Table 1. Huey Test Results

Specimen		Weight loss per equivalent 48-h period, g		
Number	Condition	1st run	2nd run	3rd run
3A-1(N)	Annealed control	0.0006	0.0011	0.0015
3A-1	Annealed control	0.0008	0.0012	0.0016
AR-1(N)	As received control	0.0007	0.0016	0.0014
AR-1	As received control	0.0009	0.0013	0.0015
106-A-16	Low-fluence irradiated	Completely dissolved		
105-C-16	Medium-fluence irradiated	Completely dissolved		
105-B-23	High-fluence irradiated	Completely dissolved		
105-B-24	High-fluence irradiated	0.0171		
105-B-24(N)	High-fluence irradiated	0.0171	0.0210	0.0283

The dissolution rates of the irradiated specimens are from 10 to 50 times greater than those of the control specimens, indicating that the susceptibility of the cladding to intergranular attack is greatly enhanced during irradiation. Figure 11 shows how the irradiated specimens were affected by exposure to the boiling HNO_3 for periods totaling 6 and 21 hours.

Metallographic mounts of the annealed and as-received Huey control test specimens were prepared using standard techniques. The specimens in these mounts (Figures 12 and 13) were exposed to the HNO_3 solution for a total of 144 hours, yet show only slight intergranular attack.

Metallographic mounts of the irradiated Huey test specimens 105B-24(N) and 105B-24 were also prepared. The techniques used in preparing mounts of the irradiated fuel cladding were again used.

Photomicrographs of the inside and outside surfaces of the cladding are shown in Figures 14 through 17 for exposure periods of 6 and 21 hours.

The increased grain boundary attack in the irradiated specimens is quite evident. Penetration up to 0.005 inch was observed. Also noticeable is the complete absence of some of the grains. The difference in the degree of attack for the 6- and 21-hour Huey test periods is evident, as is the difference in attack at the inside and outside edges. The attack at the inside edge was greater in all cases. This is probably due to (1) cracks and voids on the inside surface of the cladding tubes after removal from the reactor, and (2) the scale formed on the outside of the tubes during reactor operation.

The cladding thickness of the irradiated Huey specimens ranged between 0.0204 and 0.0210 inch, indicating no appreciable change of the cladding thickness due to dissolution. Also, the final measurements of the Huey control specimens showed no change in wall thickness.

3.5. Potentiostatic Polarization Tests

Although the Huey test provides a standard approach to evaluating the susceptibility of a stainless steel to intergranular corrosion as a result of chromium carbide precipitation, it does not provide a sufficient flexibility. The corrosion behavior can be assessed over a wide range of electrochemical potentials by measuring the current produced on the specimen by applying a controlled potential using a potentiostat. Thus, the Huey test examines only a single potential, while the potentiostat can evaluate a wide range.

In these experiments, irradiated and nonirradiated specimens were examined at two pHs. The nonirradiated specimens were studied in two conditions: as-received and as-received plus a 500-hour anneal at 260 C. The irradiated specimens were examined at three fluences: high (2.5×10^{21} nvt), average (1.6×10^{21} nvt), and low (0.5×10^{21} nvt). The irradiated samples had a very tenacious thin black film, whereas both nonirradiated samples appeared metallic. The samples were right cylinders of 0.275-inch length and 0.304-inch diameter.

Tests were conducted in two different environments. The 1N H_2SO_4 solutions were made from reagent-grade sulfuric acid and triple-distilled water, and had a pH of +0.35. The pH 8.4 solution was composed of boric acid and sodium borate.

The polarization cell shown in Figure 18a was used for all tests. The cell has separate compartments for the auxiliary electrode and the working sample electrode connected by a fritted disc. A movable Luggin probe was incorporated in the design so that the top of the probe could be adjusted to 0.5 mm from the sample. The sample was mounted on a Teflon holder as shown in Figure 18b. The part shown in the lower left-hand side of 18a was screwed snugly into the middle section. When assembled, the holder provided a leak proof seal around the edges of the specimen, and the specimen was the only metallic part of the holder exposed to the solution. A saturated calomel electrode was used as the reference electrode.

A model 66-TS-10 Wenking potentiostat and Wenking motor potentiometer were used for all potentiostatic scans. A Keithley 610-B electrometer and a Leeds and Northrup potentiometer were used for all potential measurements. A Hewlet-Packard X-Y recorder was used to record all polarization curves. The recorder output of the potentiostat enabled direct recording of a psuedo $\log i$ versus E curve. This curve was latter clamped to a potential versus current density plot by use of a calibration curve.

The cell was filled with 500 ml of test solution, and nitrogen was bubbled through for 1 hour to remove dissolved oxygen. The specimen was mounted on the holder, activated in boiling 1N H_2SO_4 solution, and then inserted into the cell. The Luggin probe was adjusted to 0.5 mm away, and the probe and the solution bridge were filled with test solution. The corrosion potential of the sample was determined by using the output of the potentiometer and nulling the electrometer. The potentiometer was then removed from the circuit. The corrosion potential was set onto the potentiostat and then put on operate. The motor potentiometer was then set at 2V/h and the scan was initiated. The polarization curve was recorded automatically on the X-Y recorder. The anodic scan was run first, and the potentiostat was then turned off. The specimen was allowed to return to the original corrosion potential and the foregoing procedure was repeated to determine the cathodic curve.

This procedure was relatively easy to carry out for the nonirradiated samples, since the experimentation could be done without the precautions attendant to radioactive specimens. Since the irradiated

samples were radioactive (1R or more at contact), the experimentation had to be performed at the Battelle hot cell facility. The electrochemical cell was set up inside the airborne radiation area. The potentiostat and recorders were set up outside the cell, and electrical leads were taken through the radiation boundary. The lead gloves required in handling the irradiated specimens made this work tedious, and it is possible that the specimens could have been damaged. However, there were no indications of damage.

The experimental data are summarized in Figures 19 through 22. It is clearly indicated that the irradiated specimens are more reactive over a wide range of potentials and at both pHs. The higher pH was selected in view of the generally slightly alkaline pH used in reactor environments. It is noteworthy that specimens having the thicker oxides (i.e., as removed from the reactor) are more reactive than those for which the oxides were removed. This may be related to the difference in defect structures of the new film on the latter compared with that on the former.

The acceleration of the reaction due to irradiation, as measured by the potentiostat, is in general accord with that shown by the Huey testing. As can be seen from Figures 19 through 22, the acceleration of the reaction rate due to irradiation covers a very wide range of potentials. Also, the data on the Huey tests and polarization of irradiated specimens are very similar to that found from examining irradiated specimens from GE (without boron). These data are summarized in AEC report COO-1319-30.

3.6. Tensile Testing

The irradiated and nonirradiated stainless steel cladding was tensile-tested at B&W's laboratories in Lynchburg, Virginia. The complete compilation of the data is given in B&W report BAW-3809-6.⁹ Some of the significant data from this report are reproduced herein, since the selection of the creep test conditions for section 3.7 is based on these tensile data. Table 2 shows the array of test conditions used. Figures 23 through 26, from B&W's work, show yield strength, ultimate tensile strength, uniform elongation, and total elongation as affected by the parameters of Table 2 (specimens were annealed for 1 hour at 1000 C). Figures 27 through 30 from B&W's work show the same tensile data for specimens that were not annealed.

Table 2. Tensile Testing of Stainless Steel Cladding

Condition	Test Temperature, C											
	70		316		400	480		564	650		705	746
	<u>BA</u>	<u>AA</u>	<u>BA</u>	<u>AA</u>	<u>BA</u>	<u>AA</u>	<u>BA</u>	<u>AA</u>	<u>BA</u>	<u>AA</u>	<u>AA</u>	<u>AA</u>
Maximum fast fluence (2.5×10^{21} nvt)	2	2	2	2	2	2	2	2	2	2	2	0
Minimum fast fluence (0.5×10^{21} nvt)	2	2	2	2	0	0	0	0	2	2	0	0
As received, control	2	2	2	2	0	0	0	0	2	2	0	0
As received and aged 3000 h at 316 C	2	2	2	2	2	2	0	2	2	2	2	2
As received and aged 6000 h at 316 C	2	2	2	2	0	0	0	0	2	2	2	0

BA = before annealing.

AA = after annealing for 1 hour at 1000 C.

In Figures 23 through 30, the trends of data as affected by annealing and test temperature are largely in accord with well-known patterns. The most important result here is the lack of recovery of uniform elongation after annealing when the tests were conducted in the range from 540 to 650 C. The detailed patterns of the trends are discussed in BAW-3809-6.

3.7. Creep Tests

Creep testing was conducted on both control and irradiated specimens, so that the effect of boron on the rupture life could be determined. In addition, since the 650 C test temperature was above the embrittlement temperature (as determined by tensile tests that B&W conducted on the cladding), these tests provided specimens for studying the role of helium on embrittlement with the electron microscope.

Three high-fluence irradiated specimens and three control specimens were tested at stresses between 11,000 and 28,000 psi. All tests were performed at 650 C in an air atmosphere. The split-tube specimens described earlier and shown in Figure 31a were annealed for 1 hour at 930 C before testing. Special Inconel grips were designed to insure load alignment and to eliminate grip slippage during the long-term tests. (See Figure 31b).

All creep tests were performed on Satec Model-D creep stands using a 3:1 lever arm ratio. The Model-D features an automatic draw-head, which relevels the lever arm after approximately 0.005 inch of specimen elongation. This leveling device is designed to minimize axial or torsional loads to the specimen during operation.

Specimens were heated with an independently controlled, three-zone, wire-wound furnace as shown in Figure 32. An unirradiated specimen with three thermocouples equally spaced along the gage section was used to profile the furnace before testing. During testing, the temperature was measured with a sheathed thermocouple contacting the center of the specimen's gage section.

Elongation measurements were made with an LVDT-type extensometer attached to the grips just beyond the ends of the specimen. Parallel extension arms were used to establish and maintain radial alignment of the specimen and the grips during loading. The

extensometer systems were calibrated before and after testing. Figure 33 shows the pull-rods, grips, specimen, and extensometer ready for insertion into the creep stand.

Figure 34 is a plot of stress versus creep rupture life. The control specimens failed after about 25% elongation, whereas the irradiated specimens failed after only approximately 3% elongation. The rupture life of the control specimens was more than 100 times greater than that of the irradiated specimens at the higher stress levels. All specimens exhibited normal failures within the gage section, but showed no signs of slippage in the grips. Examination showed that both control and irradiated specimens had additional cracks or tears within the gage section. These cracks (perpendicular to the tensile axis) began at the sharp edge of the gage section and extended inward for a distance of 0.010 to 0.050 inch. The cracks were visible to the naked eye, and at least several were detected on each specimen. The irradiated specimen loaded to 11,000 psi is still running after 2200 hours, but it has elongated to almost 3% and failure is expected at any time.

The initial elongation upon loading was characteristically less for the irradiated samples. (Figures A-27 through A-32 of the Appendix are curves showing elongation versus time for each specimen.) Although insufficient tests were run to make quantitative comparisons, the secondary creep rates of the irradiated specimens were normally greater than those of the control specimens.

3.8. Fractographic Studies

Fractographic studies were undertaken to determine the relationships between irradiation-induced defects, such as gas bubbles, and the fracture modes. In order to observe a spectrum of conditions, both irradiated and control specimens were selected from fractured tensile specimens which were tested at temperatures above (650 C) and below (480 C) the embrittlement temperature. The remainder of the specimens were taken from fractured creep specimens, both irradiated and control.

Specimens were prepared at Battelle's hot cell facility by the standard cellulose-acetate method. After carbon shadowing, they were examined in a REC-EMU-3E electron microscope.

Figures 35 and 36 are typical fractographs taken from the fractured control tensile specimen TA-14 tested at Babcock & Wilcox. This specimen, tested at 480 C (below the embrittlement temperature), exhibited the elongation expected for 304 stainless steel. Dimpling is quite evident, and the failure is described as transgranular and ductile. Some precipitates can be detected.

Figures 37 and 38 are typical fractographs from the control tensile specimen TA-9 which was tested at 650 C (above the embrittlement temperature). Dimpling and the occasional presence of precipitates are again evident. Although this failure is predominantly ductile and transgranular, there could be some argument for low-angle facets in Figure 38.

Figures 39 and 40 are taken from the fracture area of a high-fluence irradiated tensile specimen. This specimen (PA6) was tested at 480 C and failed after 7.5% total elongation. The failure mode is still ductile transgranular. Figure 39a shows an area in which normal dimpling is combined with tear dimpling. These areas were common throughout the specimen. Fractographs taken at higher magnification (Figure 40) showed no distinct evidence of voids or helium bubbles.

Figures 41 and 42 were taken from the irradiated tensile specimen PA-10, which was tested at 650 C. It showed a total elongation of only 1.1%. Figure 41 shows a distinct intergranular brittle failure. The center lower portion of Figure 42a shows evidence of acicular precipitates near a grain boundary junction. Again, there is no evidence of helium bubbles.

Figures 43 and 44 were taken from an unirradiated creep control specimen. This specimen (3-9) was tested at 650 C and had a rupture life of approximately 13 hours with 26% elongation to failure. The fractographs indicate a ductile intergranular fracture.

Figures 45 and 46 were taken from irradiated creep specimen 9-18. This specimen was tested at 650 C and had a rupture life of approximately 23 hours with 2.9% elongation to failure. The failure mode was predominately brittle intergranular, but, there were a few areas that exhibited ductile tearing (Figure 46b). The presence of helium bubbles was not detected.

Fractographs of other specimens are shown in Figures A-15 through A-26 of the Appendix.

3.9. Transmission Electron Microscopy Studies

The transmission studies were undertaken to evaluate the effects of irradiation on such characteristics as insoluble second-phase particles, grain boundaries, dislocations, and microstructural defects. Of primary importance was the detection of voids associated with helium bubbles produced after irradiation. Thin foils were prepared from the following areas:

1. Sections from as-received and annealed control tubes.
2. Sections from high-fluence-irradiated tubes.
3. Sections near the fracture area of annealed control tensile specimens tested above and below the embrittlement temperature.
4. Sections near the fracture area of high-fluence-irradiated tensile specimens tested above and below the embrittlement temperature.
5. Sections near the fracture area of irradiated and control creep specimens.

The specimens were prepared in Battelle's hot cell at West Jefferson and subsequently thinned and examined at Battelle's King Avenue laboratories. The electrolytic jet-indenting technique as developed by DuBose and Stiegler was used to thin the unirradiated specimens.¹⁰ Small, concentric dimples are formed on both sides of a specimen with a pneumatically operated electrolytic jet. The specimens are indented until approximately 0.001 to 0.005 inch of material remains between the dimples. An electrolyte of 10% HCl at 100 V was used for the indenting. The electrolytic jet was also used to section the specimens to the size required for the specimen holder in the electron microscope; this was done to minimize the possibility of damaging the specimens.

The final thinning was done in an electrolytic cell with a collimated light source on one side and a low-power microscope on the other. The electrolyte was 5% HClO_4 in glacial acetic acid at a potential of 35 V. The specimen was electrolytically thinned until the first indication of light appeared through the specimen. The edges of the hole formed in this manner were usually thin enough to be examined by transmission electron microscopy.

It was difficult to thin the irradiated specimens with the electrolytic jet because a fine spray created by the jet spread radioactive

contamination over the surrounding area in a short time. This exposed the operator to unnecessarily high radiation levels when the specimens were positioned in the jet apparatus. In an effort to alleviate this condition and at the same time obtain more consistent results, a recently developed electrolytic technique was used in which pointed electrodes form dimples in the specimens.¹¹ A pointed electrode was positioned approximately 0.01 inch from the surface of the specimen, and a dimple was quickly formed under the electrode. An electrolyte of 2% HNO₃ and 2% H₂SO₄ in ethylene glycol was used at a potential of 35 V. A highly polished dimple approximately 0.010-inch deep could be formed in about two minutes with this technique. The final thinning was done in a similar manner as with the unirradiated specimens.

Figures 47 and 48 are typical of the dislocation structure found in specimen 3A-2 (annealed for 3000 h at 316 C control). All of these figures have at least one reflection operating for diffraction contrast, but they may not show all sets of dislocations. (This is because $\vec{g} \cdot \vec{b}$ conditions may not be satisfied for all $\vec{b}$; since $\vec{g}$ is the reciprocal lattice vector of the operating reflection and $\vec{b}$ is the Burger's vector of the dislocation, when $\vec{g} \cdot \vec{b} = 0$, the dislocation will not be in contrast even though the specimen is suitably oriented for diffraction contrast.) As these figures show, all the dislocations were close to particles, and the matrix was relatively free of dislocations. The particles are probably carbides, and the dislocations may have been generated from the carbides on cooling. This would be a particularly reasonable assumption if the material had been quenched from the annealing temperature.

Figure 47b demonstrates a difficulty encountered in thinning the material for transmission electron microscopy. The figure shows the edge of the thinned portion of a sample. The two holes adjacent to the edge were undoubtedly formed by preferential attack around the carbides, and the carbides subsequently dropped from the specimen. The dark bands at the edges of the specimen and the holes are thickness contours resulting from diffraction contrast; these bands demonstrate the rather pronounced wedge shape of the specimen adjacent to holes. This indicates that holes formed by the preferential attack of the carbides enlarge very rapidly after formation and thus prevent the formation of relatively large thinned areas for transmission electron microscopy.

Figures 49 and 50 are close enough to optimum diffraction contrast conditions to reveal dislocation in specimens AR-2 (as-received control specimens). Figures 49a, 49b, and 50a show the particles that are thought to be carbides. Although a few dislocations are seen around one of the particles in Figure 50a, very few dislocations were found around the particles in this material, and the dislocation density was relatively low.

Figure 49a shows a twin and the twin boundaries with characteristic diffraction contrast fringes. Tilting experiments to obtain different operating reflections showed that the anomolous spots in this figure and in Figure 49b are due to contamination of the specimens. Figure 50b displays an interesting structure along one of the grain boundaries. Close observation reveals the usual diffraction contrast fringes along the boundary, but the boundary appears to be enveloped with an unidentified substance. Since this material appeared to have more severe etching along some of the grain boundaries, this substance could be related to grain boundary carbides. It was not abundant enough to be identified by selected area electron diffraction.

A high-fluence-irradiated tube section (specimen 105-B-23) was examined, and the results are shown in Figures 51 and 52. This specimen had an exceptionally high concentration of irradiation-induced defects. This high concentration caused considerable overlap of the diffraction contrast images obtained from the defects and prevented use of the Ashby-Brown coherency strain-contrast criterion for identification of the defects. Evidence of reciprocal lattice streaking was found in several of the electron diffraction patterns taken from this specimen, but the source of the streaking was not determined. Reciprocal lattice streaking of the type normally observed results from relaxation of the Laue conditions in one dimension; this is usually caused by thin planar defects, such as disc- or platelet-shaped precipitates, micro-twins, and stacking faults. An angular-shaped grain-boundary precipitate was also found in this specimen.

Figure 51a is from an area of the specimen containing a grain boundary. The specimen was oriented so that one of the grains adjacent to the boundary was in proper contrast to show the high concentration of irradiation-induced defects. However, no evidence of voids (helium

bubbles) can be seen either in or along the boundary. Figure 51b is from another area of the specimen containing a grain boundary; both grains are adjacent to the boundary in approximately the proper contrast to show the defects. The diffraction contrast fringes along the grain boundaries appear to be more severely distorted when both grains are in contrast than when only a single grain is in contrast. The probable cause is that the boundary is at an angle to the surface of the foil, and defects are contained between the boundary and both foil surfaces. Although no voids were observed in the grain boundaries or in the grains of this specimen, the possibility that unresolved helium bubbles may exist among the irradiation-induced defects cannot be discounted. No evidence of a denuded zone was found along any of the boundaries.

Figure 52 shows examples of the grain-boundary precipitates that were found in this sample. These precipitates, identified by selected-area-electron diffraction as $M_{23}C_6$, were usually angular shaped and appeared to grow preferentially into one of the grains. Selected-area-electron diffraction and dark-field analyses were used to determine the relative orientations of several grain-boundary precipitates and their adjacent grains. These precipitates were found to have the same orientation as one of the adjacent grains, with $[100]$ planes and $\langle 100 \rangle$ directions of both the precipitates and the grain being parallel. Although the precipitates within a single boundary were usually of the same orientation, they indiscriminantly assumed the orientation of either of the adjacent grains. The massive dark particles in the grains are foreign particles apparently picked up during the final washing of the specimens.

In an attempt to reduce the amount of defect structure and possibly permit the detection of helium bubbles, a low-fluence-irradiated specimen was thinned and examined. Typical photographs of this specimen (106-A-16) are presented in Figure 53. It is evident from these figures that even the low-fluence-irradiated specimens contain sufficient irradiation-induced defects to preclude the detection of helium bubbles.

In a further attempt to reduce the amount of defect structure, thin foils were prepared from a high-fluence-irradiated tube section (specimen 105-B-17) that had been annealed for 1 hour at 1000 C to remove

the heavy defect structure. Figures 54 through 57 are typical photographs of this section. Figure 54a shows the heavy dislocation structure found around large precipitates and the relatively light defect structure found throughout the remainder of the grain.

Figure 54b depicts an area in which a carbide precipitate has fallen out, leaving only the foil and the dislocation structure around it. Helium bubbles can be detected just outside the precipitate dislocation structure near the top of the photograph. At the very top is the hole formed in the foil during thinning. Bubbles cannot be detected around the remainder of the foil because of the large change in thickness that occurs when moving farther from the edge of the foil.

Figure 55 shows typical areas within a grain. A grain boundary junction containing several precipitates is seen in Figure 56a. Although several bubbles can be detected at the top of the photograph, tilting experiments were unable to identify any helium bubbles associated with the grain boundaries themselves. Figure 56b shows helium bubbles associated with the light dislocation structure around a precipitate; largest bubble is about 400 Å in diameter.

Figure 57a shows a fine network of helium bubbles next to some precipitates along a grain boundary. The fact that bubbles were not always detected around grain boundary precipitates was probably more a function of the foil thickness and the reflection than of the actual presence or absence of helium. Figure 57b is a higher magnification of the area in Figure 54b. The light area at the lower left of the picture is just beyond a point where a precipitate dropped out of the foil. A high concentration of small bubbles (less than 150 Å in diameter) can be seen to extend through the heavier defect structure, which encircled the precipitate. At this point (a distance of about 3μ from the precipitate) there is a low-density region of helium bubbles of large diameter (up to ≈ 500 Å). Although a two-dimensional picture of a foil can be somewhat deceiving, some of the bubbles seem to be associated with dislocations.

In order to correlate the results obtained from examining thin foils of irradiated tensile specimens, specimens were prepared from

annealed control tensile specimens. These specimens, TA-14 and TA-9, were tested at 900 and 1200 F, respectively. Both specimens displayed good elongation and possessed a dislocation cell network characteristic of plastically deformed material. Typical photographs of these specimens are shown in Figures 58 and 59.

The fractured irradiated tensile specimens were PA-6 and PA-10. Both specimens were annealed for 1 hour at 1000 C before testing at 480 and 650 C, respectively. The total elongations were 7.5% for PA-6 and 1.1% for PA-10. These specimens are shown in Figures 60 through 64.

Figures 60 and 61 show several grain boundaries, which in most cases were free of carbides. The dislocation cell network is evident in this specimen (PA-6). The helium bubbles have grown in size and are no longer associated with precipitates; rather, they are randomly dispersed throughout the matrix. Occasional bubbles can be found along the grain boundaries, but the bubbles show no high affinity for boundaries. Bubbles were found with diameters as large as 1500 Å.

Figures 62 through 64 are typical photomicrographs of specimen PA-10. The dislocation cell network is not present in this specimen (only 1.1% elongation). Again, large precipitates along the grain boundary were not detected, although small, closely spaced precipitates can be detected along grain boundaries in Figures 62b and 64a. Precipitates are also found within the grains (Figure 62a). Although helium bubbles were again found scattered throughout the grains in this specimen, they tended to lie along the grain boundaries more than in specimen PA-6. In turn, the density of the bubbles dispersed throughout the grains was not as great. The maximum diameter of any helium bubble was about 1200 Å.

Attempts to prepare thin foils from the irradiated creep specimens were unsuccessful. Problems associated with the oxide film formed during the long exposure at temperature could not be overcome in the length of time remaining in the program.

Additional transmission electron microscopy specimens are shown in Figures A-1 through A-14 of the Appendix.

4. DISCUSSION

4.1. Objectives

One objective of this investigation was to obtain additional basic information on the role of helium in the embrittlement of stainless steels irradiated at elevated temperatures. This information was not intended to substantiate or refute current embrittlement theories or to formulate new ones. Although comparisons of this nature are difficult to avoid in a discussion, the results of this study are principally meant to expand our understanding of this phenomenon. A second, though less extensively pursued objective, was to provide a better understanding of the effects of irradiation on corrosion processes. Results of interest are discussed in the following paragraphs.

4.2. Inspection of Irradiated Tubing

The most interesting observation on the irradiated cladding was the occurrence of voids and cracks along the inside surface of the cladding tubes. Considering the environment and the independence of the cracks from reactor conditions, this effect is difficult to explain as a reactor or a radiation-induced phenomenon. More likely, the cracks and voids formed before the tubing was inserted into the reactor, possibly by the pickling operation during fabrication of the tubing.

If the cooling rate from the solution heat treatment were marginal, then the inside of the tubing, which would cool more slowly than the outside, could be sensitized. Since the pickling process follows the heat treatment, some pickling solution (dilute sulfuric, nitric, or hydrochloric acid) could remain on the inside of the tube because of the increased difficulty in rinsing this surface. The remaining dilute solution could then become more concentrated as a result of the gradual evaporation of water, until conditions were favorable for intergranular corrosion. Credence in this explanation can be found by comparing

the cracks (Figures 4 through 7) with the intergranular attack resulting from the Huey tests (Figures 14 through 17).

4.3. Corrosion Studies (Huey and Polarization Tests)

As shown in report COO-1319-30, irradiation appears to accelerate the intergranular corrosion of quench-annealed stainless steel. The results of the Huey tests and the polarization tests clearly confirm this trend. The most reasonable explanation is that in situ sensitization occurs during radiation. In fact, the transmission photomicrographs, Figures 52, 58, 62, 63, and 64, show a much greater abundance of carbide precipitates at the grain boundaries than do the nonirradiated control specimens of Figures 47, 49, and 50. Although no crucial comparison of this difference between irradiated and nonirradiated specimens has been made, the photomicrographs available to us generally support the trend observed in corrosion tests; i.e., irradiated materials corrode rapidly in an intergranular manner.

4.4. Creep Tests

The drastic reduction in rupture life (a factor of 100 at higher stresses) of the irradiated type-304 borated stainless steel over that of the same unirradiated material was primarily due to a lack of ductility in the irradiated specimens. There was very little elongation during initial loading, and very little due to creep under load. In Figure 65, some data from other investigators are plotted together with data from this study. Although caution must be exercised in comparing data produced at different institutes under different test conditions, different irradiation temperatures, and different fluence levels, it can be concluded that the rupture life of the borated 304 SS is shorter in almost all instances (both irradiated and control specimens).

Although the reduction in rupture life is primarily a result of the high boron content of the cladding, it is evident that the specimens were not of the optimum design for creep tests. In cutting these specimens from the cladding, a sharp, ragged edge was produced on both sides of the gage section. These edges undoubtedly acted as stress risers and shortened the rupture life somewhat. This effect could partially explain why the rupture life of the 304 SS control specimens was shorter

than normal. This effect would be even greater for the more brittle irradiated specimens and would explain the small cracks along the sides of the fractured creep specimens as noted in the experimental section.

4.5. Fractographic Studies

At about 500 C, the mode of failure in unirradiated 304 stainless steel begins to change from transgranular to intergranular. This change is accompanied by an increase elongation. At approximately 700 C, the failure mode is predominantly intergranular due to grain boundary sliding; this results in a decrease in elongation with further increases in temperature.

The fractographs of the fractured irradiated tensile specimen tested at 480 C exhibited shear-rupture dimples as expected. The failure mode was ductile transgranular. However, the remnants of the internal voids—the shear-rupture dimples—appeared in many cases to be cluttered with localized voids, which interacted with the internal void during its growth by plastic flow (Figure 40). It is quite possible that these localized voids were nucleated by clusters of helium bubbles, which were present in abundance within the grains of the irradiated specimens. This interaction would reduce the amount of plastic flow necessary for crack initiation and ultimate failure. As was noted in the results of the tensile tests conducted at Babcock & Wilcox, there was an overall reduction in elongation, even below the embrittlement temperature, which could not be removed by annealing. Moderate damage of this type is not the usual displacement damage, and it is possible that it results from the interaction of the voids nucleated by helium bubbles within the grains.

The fractographs of the irradiated tensile specimen tested at 650 C clearly exhibited a brittle transgranular fracture. There is an occasional occurrence of grain boundary voids, probably nucleated from carbides at the grain boundary. There is no indication of helium bubbles at the grain boundaries. This is not entirely unexpected, however, since it is likely that voids are nucleated at precipitates in the grain boundary and that their growth is merely accelerated by the interaction with helium bubbles, which become void extensions. In this case, the

void created by a helium bubble would not be distinguishable from an enlarged void nucleated at a grain boundary precipitate.

The fractographs from the irradiated creep specimen tested at 650 C exhibited some unusual characteristics. Although the failure was predominantly brittle-intergranular, there is some evidence of ductile tearing in Figure 46b. Evidently, at the slower rates of straining, the transition from a transgranular to an intergranular mode of failure is retarded. It is also worth noting that the creep specimen failed after almost 3% total elongation, whereas the tensile specimen tested at the same temperature failed after only about 1% elongation. Figure 46a also shows a much higher concentration of voids at the grain boundary than found in the irradiated tensile specimen. This is due to the increase in carbide precipitation at the grain boundary resulting from the time at test temperature (23 hours at 650 C).

4.6. Transmission Electron Microscopy Studies

Evidence of helium bubbles was not detectable in the unstrained specimens until the heavy irradiation-induced defect structure was removed by annealing at 1000 C. This unavoidably resulted in some bubble growth, although it cannot be inferred that bubbles were not present or were too small to be detected before annealing. Bubbles were detected around precipitates and along dislocations, and were locally dispersed in the matrix. Helium attached to precipitates was not detected. However, in the foils observed, precipitates quite often had fallen out owing to preferential attack during thinning. The precipitates that did remain were in a thick portion of the foil or were surrounded by such a heavy defect structure that helium could not be detected. Also, helium was not detected in any of the grain boundaries observed in the unstrained irradiated material.

Figure 57b shows a typical helium formation around a precipitate that has fallen out of the matrix. A very high concentration of small helium bubbles exists throughout the defect tangle that surrounded the precipitate. However, this helium distribution ends abruptly beyond this defect structure and is replaced by a circumferential band (about 1 μ wide) containing a much lower concentration of larger bubbles. This outer band is well beyond the normal recoil distance of an alpha particle

($\approx 2\mu$), and the source of the helium cannot be explained unless there is another precipitate nearby that cannot be seen in the photograph. It is thought that changes in helium size and density are a result of the annealing treatment. Small helium bubbles in the outer band were able to coalesce and grow, whereas coalescence of the helium nearer the particle was impeded by the heavy dislocation structure.

The helium bubbles in the irradiated tensile specimens were much larger than those in the control specimens. Since the thermal history of the tensile specimens is only moderately different from that of the constrained specimen 105-B-17, almost all of this growth must result from straining. The movement of dislocations during straining sweeps small helium bubbles along and aids greatly in bubble coalescence. Once the bubbles become large enough to cause sufficient drag, they separate from the dislocation and occupy a random position within the matrix as illustrated in Figures 60 through 62. These larger bubbles usually assume a polyhedral shape, but in Figures 60a, 61b, and 62b a square shape is resolvable.

The similarities between the 480 C irradiated tensile specimen (7.5% elongation) and the 650 C irradiated tensile specimen (1.1% elongation) seem to outnumber their differences. Except for the dislocation cell structure in specimen PA-6 both specimens contain an abundance of helium bubbles of approximately the same size, and neither specimen shows a high tendency for helium to accumulate at the grain boundaries. Both specimens contain helium at the grain boundaries, but not as much as might be expected for a specimen containing this much helium (more than 10^{-4} atom fraction). Given equal amounts of helium, the fracture mode, whether transgranular or intergranular, appears to be the controlling variable when testing above and below the embrittlement temperature.

Figure 1. As-Received Control Specimen AR-1:
(a) Etched in 20 H₂O-20 HCl-10 HNO₃-
5g FeCl₃; (b) As Polished

250X

(a)

250X

(b)

Figure 2. Annealed Control Specimen 3A-1: (a) Etched in 20 H₂O-20 HCl-10 HNO₃-5g FeCl₃; (b) As Polished

250X

(a)

250X

(b)

Figure 3. Typical Photographs of Irradiated Cladding: (a) Outside Surface of Tube; (b) Outside Surface of Tensile Specimen; (c) Inside Surface of Tensile Specimen; (d) Stain on Inside Surface of Tensile Specimen

(a)

4X

(b)

4X

(c)

4X

(d)

6X

Figure 4. Inside Edge of High-Fluence
Irradiated Specimen

Etched

250X

Figure 5. Inside Edge of Medium-Fluence
Irradiated Specimen

Etched

250X

Figure 6. Inside Edge of Low-Fluence
Irradiated Specimen

As Polished

250X

Figure 7. Inside Edge of Irradiated
Cladding, Taken at B&W

Oxalic, Electrolytic

250X

Figure 8. Outside Edge of Medium-Fluence Irradiated Specimen

Figure 9. Typical Tube-Sectioning Diagram

Figure 10. Huey Test Apparatus for Control Specimens

Figure 11. Surface Attack on High-Fluence Irradiated Specimen After (a) 6 Hours and (b) 21 Hours in Huey Test Solution

(a)

6X

(b)

6X

Figure 12. As-Received Control Specimen After
144 Hours in Huey Test Solution

Etched

(a)

250X

As Polished

(b)

250X

Figure 13. Annealed Control Specimen After 144
Hours in Huey Test Solution

Etched

250X

(a)

As Polished

250X

(b)

Figure 14. Inside Edge of High-Fluence Irradiated Specimen After 6 Hours Exposure to Huey Test Solution

As Polished

250X

Figure 15. Outside Edge of High-Fluence Irradiated Specimen After 6 Hours' Exposure to Huey Test Solution

As Polished

250X

Figure 16. Inside Edge of High-Fluence Irradiated Specimen After 21 Hours' Exposure to Huey Test Solution

Etched

250X

Figure 17. Outside Edge of High-Fluence Irradiated Specimen After 21 Hours' Exposure to Huey Test Solution

Etched

250X

Figure 18a. Electrochemical Cell

Figure 18b. Sample Holders for Specimens Used in Polarization Experiments

Figure 19. Anodic Polarization Curves in H_2SO_4

Figure 20. Cathodic Polarization Curves in H_2SO_4

Figure 21. Anodic Polarization Curves in pH 8.4
Boric Acid-Sodium Borate

Figure 22. Cathodic Polarization Curves in pH 8.4
Boric Acid-Sodium Borate

Figure 23. Yield Strength After Anneal at 1000 °C

Figure 24. Ultimate Tensile Strength After Anneal at 1000 °C

Figure 25. Uniform Elongation After Anneal at 1000 °C

Figure 26. Total Elongation After Anneal at 1000 °C

Figure 27. Yield Strength Before Anneal

Figure 28. Ultimate Tensile Strength Before Anneal

Figure 29. Uniform Elongation Before Anneal

Figure 30. Total Elongation Before Anneal

Figure 31a. Split-Tube Creep Specimen

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Figure 34. Creep Rupture Curves for Irradiated and Nonirradiated Borated Type-304 SS Split Cladding Specimens

Figure 35. Typical Electron Microscope Fractographs, Specimen TA-14

3900X

(a)

3900X

(b)

Figure 36. Typical Electron Microscope Fractographs, Specimen TA-14

7250X

(a)

4700X

(b)

Figure 37. Typical Electron Microscope Fractographs, Specimen TA-9

4700X

(a)

7250X

(b)

Figure 38. Typical Electron Microscope Fractographs, Specimen TA-9

7250X

(a)

3900X

(b)

Figure 39. Electron Microscope Fractographs, Specimen PA-6

(a)

3000X

(b)

6000X

Figure 40. Electron Microscope Fractographs, Specimen PA-6

22,500X

(a)

15,000X

(b)

Figure 41. Electron Microscope Fractographs, Specimen PA-10

Figure 42. Electron Microscope Fractographs, Specimen PA-10

22,500X

(a)

15,000X

(b)

Figure 43. Electron Microscope Fractographs, Specimen 3-9

3000X

(a)

3000X

(b)

Figure 44. Electron Microscope Fractographs, Specimen 3-9

15,000X

(a)

15,000X

(b)

Figure 45. Electron Microscope Fractographs, Specimen 9-18

3000X

(a)

6000X

(b)

Figure 46. Electron Microscope Fractographs, Specimen 9-18

6000X

(a)

6000X

(b)

Figure 47. Transmission Electron Microscopy, Specimen 3A-2

15,000X

(a)

15,000X

(b)

Figure 48. Transmission Electron Microscopy, Specimen 3A-2

6000X

(a)

15,000X

(b)

Figure 49. Transmission Electron Microscopy, Specimen AR-2

(a)

15,000X

(b)

15,000X

Figure 50. Transmission Electron Microscopy, Specimen AR-2

15,000X

(a)

15,000X

(b)

Figure 51. Transmission Electron Microscopy, Specimen 105-B-23

(a)

(b)

Figure 52. Transmission Electron Microscopy, Specimen 105-B-23

7500X

(a)

12,000X

(b)

Figure 53. Transmission Electron Microscopy, Specimen 106-A-16

30,000X

(a)

15,000X

(b)

Figure 54. Transmission Electron Microscopy, Specimen 105-B-17

9000X

(a)

12,000X

(b)

Figure 55. Transmission Electron Microscopy, Specimen 105-B-17

12,000X

(a)

22,500X

(b)

Figure 56. Transmission Electron Microscopy, Specimen 105-B-17

22,500X

22,500X

(b)

Figure 57. Transmission Electron Microscopy, Specimen 105-B-17

9000X

(a)

30,000X

(b)

Figure 58. Typical Transmission Electron Micrographs of Shoulder Area

15,000X

(a) Specimen TA-14

22,500X

(b) Specimen TA-9

Figure 59. Typical Transmission Electron Micrographs Near the Fracture of Specimen TA-9

30,000X

(a)

15,000X

(b)

Figure 60. Transmission Electron Microscopy, Specimen PA-6

15,000X

(a)

15,000X

(b)

Figure 61. Transmission Electron Microscopy, Specimen PA-6

(a)

(b)

Figure 62. Transmission Electron Microscopy, Specimen PA-10

30,000X

(a)

30,000X

(b)

Figure 63. Transmission Electron Microscopy, Specimen PA-10

30,000X

(a)

30,000X

(b)

Figure 64. Transmission Electron Microscopy, Specimen PA-10

30,000X

(a)

30,000X

(b)

Figure 65. Type-304 SS Creep Data From This and Other Investigations

APPENDIX

Additional Electron Microscopy Specimens
and Curves Showing Elongation Vs Time
for Creep Specimens

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Figure A-1. Transmission Electron Microscopy, Specimen 105-B-17

9000X

(a)

30,000X

(b)

Figure A-2. Transmission Electron Microscopy, Specimen 105-B-17

9000X

(a)

22,500X

(b)

Figure A-3. Transmission Electron Microscopy, Specimen 105-B-17

12,000X

(a)

12,000X

(b)

Figure A-4. Transmission Electron Microscopy, Specimen 105-B-17

12,000X

(a)

12,000X

(b)

Figure A-5. Transmission Electron Microscopy, Specimen 105-B-17

30,000X

(a)

22,500X

(b)

Figure A-6. Transmission Electron Microscopy,
Specimen 105-B-17

22,500X

Figure A-7. Transmission Electron Microscopy, Specimen PA-6

15,000X

(a)

15,000X

(b)

Figure A-3. Transmission Electron Microscopy, Specimen PA-6

28,000X

(a)

30,000X

(b)

Figure A-9. Transmission Electron Microscopy, Specimen PA-6

30,000X

(a)

30,000X

(b)

Figure A-10. Transmission Electron Microscopy,
Specimen PA-6

30,000X

Figure A-11. Transmission Electron Microscopy of Shoulder, Specimen TA-14

22,500X

(a)

22,500X

(b)

Figure A-12. Transmission Electron Microscopy of Shoulder, Specimen TA-9

15,000X

(a)

15,000X

(b)

Figure A-13. Transmission Electron Microscopy: (a) Specimen TA-9;
(b) Shoulder View, Specimen TA-9

15,000X

(a)

15,000X

(b)

Figure A-14. Transmission Electron Microscopy, Specimen TA-9

22,500X

(a)

30,000X

(b)

Figure A-15. Electron Microscope Fractographs, Specimen PA-6

3000X

(a)

3000X

(b)

Figure A-16. Electron Microscope Fractographs, Specimen PA-6

15,000X

(a)

22,500X

(b)

Figure A-17. Electron Microscope Fractographs, Specimen PA-10

22,500X

(a)

22,500X

(b)

Figure A-18. Electron Microscope Fractographs, Specimen TA-14

3900X

(a)

3900X

(b)

Figure A-19. Electron Microscope Fractographs, Specimen TA-14

4700X

(a)

7250X

(b)

Figure A-20. Electron Microscope Fractographs, Specimen TA-14

7250X

(a)

(b)

Figure A-21. Electron Microscope Fractographs, Specimen TA-9

(a)

(b)

Figure A-22. Electron Microscope Fractographs, Specimen TA-9

7250X

(a)

7250X

(b)

Figure A-23. Electron Microscope Fractographs, Specimen TA-9

7250X

(a)

7250X

(b)

Figure A-24. Electron Microscope Fractographs, Specimen 3-9

6000X

(a)

6000X

(b)

Figure A-25. Electron Microscope Fractograph,
Specimen 3-9

15,000X

Figure A-26. Electron Microscope Fractograph,
Specimen 9-18

15,000X

Figure A-27. Elongation Vs Time for Irradiated Specimen 8-16
at 650 C and 28,000 psi Applied Stress

Figure A-28. Elongation Vs Time for Control Specimen 3-9
at 650 C and 28,000 psi Applied Stress

Figure A-29. Elongation Vs Time for Irradiated Specimen 9-18
at 650 C and 15,000 psi Applied Stress.

Figure A-30. Elongation Vs Time for Control Specimen 4-7
at 650 C and 23,000 psi Applied Stress

Figure A-31. Elongation Vs Time for Irradiated Specimen 7-16
at 650 C and 20,000 psi Applied Stress

Figure A-32. Elongation Vs Time for Control Specimen 4-4
at 650 C and 16,000 psi Applied Stress

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