ANNEALING STUDIES ON IRRADIATED
TYPE 347 STAINLESS

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TYPE 347 STAINLESS

by

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March 6, 1963

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William E. Murr and Frederic R. Shober

The effects of 1-hr anneals at 1000 and 1800 F on the mechanical properties of Type 347 stainless steel specimens taken from the ETR J-10 pressure tube after fast-neutron (>1 Mev) exposures of about 7.1×10^{21} nvt were studied. The pressure tube operated at a maximum temperature of about 750 F in a stressed condition. Testing was carried out at 75 and 750 F. Uniform elongations were increased from approximately 1 per cent in the as-irradiated material to 15 and 25 per cent, respectively, by the 1000 and 1800 F anneals. The 1000 F annealing appeared to produce a better combination of mechanical properties. The microstructure of unirradiated, as-irradiated and postirradiation-annealed specimens were very similar except for two specimens inadvertently heated above 1800 F during annealing.

INTRODUCTION

The ETR contains facilities to accommodate test loops which provide special temperature, pressure, and atmosphere conditions for irradiation testing according to specific requirements of the various reactor customers. Such a test loop was recently removed from the J-10 position of the ETR after having been in service approximately 17 months. This loop consisted of two basic capsules or tubes, an inner tube containing test specimens in pressurized water and an outer surrounding tube or jacket separated from the inner tube by a helium gas annulus. The outside of the outer tube made contact with reactor process water. This particular J-10 test loop was placed in service in the ETR in October, 1959, and was removed from the ETR on March 16, 1961. During this time the loop was exposed to approximately 222 effective full-power days (38,800 megawatt-days) at an estimated computed maximum flux (neutrons with energies >1 Mev) of 3.7×10^{14} nv. A 36-in.-long section corresponding in length to the active fuel core of the ETR was removed from both inner and outer loop sections and shipped to the Battelle Hot-Cell Facility for postirradiation examination.

The J-10 loop sections were utilized in two individually sponsored programs. The Division of Reactor Development of the AEC funded the preparation, testing, and posttest examination of 18 specimens, including six from the outer gas jacket and 12 from the inner pressure tube. These specimens were all tested in the as-irradiated condition at room temperature or at 750 F. The results of the examinations were presented in BMI-1609.⁽¹⁾ The second program, sponsored by the Westinghouse Electric Corporation, Bettis Atomic Power Laboratory, included the preparation, heat treatment, testing, and posttest examination of 18 additional specimens machined from the J-10 inner pressure tube. The details and results of the examinations performed in the program sponsored by Bettis are presented in this report. The results reported in BMI-1609 for the tests made on the inner pressure tube and the outer gas jacket are also listed in this report in order to provide a comparison of properties of as-irradiated and annealed tensile specimens.

(1) References appear at end of text.

FABRICATION HISTORY OF J-10 PRESSURE TUBE AND GAS JACKET

The pressure tube was fabricated by Thompson Products (Jet Division) from a forging produced by McInnes Steel Company. The initial billet, which was 6 in. in diameter, was heated to 2250 F and reduced by forging to 3 in. in diameter and air cooled. The forging was then rough machined, stress-relief annealed for 1 hr at 1000 F, and water quenched. The core section of the pressure tube was milled from the billet at this point. Tensile properties of specimens removed from opposite ends of the billet are given in Table 1, along with chemical analyses of the finished billet.

TABLE 1. COMPOSITION AND PROPERTIES OF J-10 GAS JACKET AND PRESSURE TUBE

	Outer Loop, or Gas Jacket(a)	Inner Loop, or Pressure Tube(b)
Composition, w/o		
Carbon	0.035	0.045
Manganese	1.59	1.60
Phosphorus	0.013	0.025
Sulfur	0.018	0.015
Silicon	0.56	0.42
Nickel	10.76	10.30
Chromium	18.66	19.85
Niobium	0.84	.81
Tantalum	0.10	--
Tensile Strength, psi	84,500 - 87,400	91,300 to 93,000
0.2 Per Cent Offset Yield Strength, psi	36,300 - 43,300	53,500 - 60,700
Elongation, per cent	53.0 - 57.0	53.0 - 61.4
Reduction in Area, per cent	73.0 - 75.0	68.0 - 72.1

(a) Composition furnished by Ladish of the billet which supplied the gas jacket. Mechanical properties were obtained on billets from opposite ends of the forging following 1000 F stress-relief heat treatment.

(b) Composition furnished by Thompson-Ramo-Wooldridge on finished component. Mechanical properties were obtained on billets from large and small ends of forging following rough machining and 1000 F stress-relief hot treatment.

The gas jacket was forged by Ladish. The forgings were heat treated for 1 hr at 1950 F and water quenched. The forgings were then reheated to 1000 F for 1 hr and water quenched. Tensile properties of the gas jacket, as determined by tests of material removed from opposite ends of the gas jacket, are given in Table 1. It can be observed from the table that the composition and fabrication conditions of both pressure tube and gas jacket were somewhat similar, although forging was accomplished at a higher temperature for the pressure tube. The tensile strength of the pressure tube was slightly higher, and the 0.2 per cent offset yield strength was appreciably higher than that of the gas jacket. Ductility, as indicated by the total elongation and reduction of area of both loop sections, was approximately equal to the preirradiation condition.

IN-PILE DESIGN PARAMETERS AND OPERATING HISTORY

A list of the pertinent design and actual operating parameters associated with the J-10 tube is given in Table 2. The list was compiled by Bettis Atomic Power Laboratory personnel from data supplied in part by the Phillips Petroleum Company ETR staff. It was observed that the actual operating conditions were somewhat less stringent than intended; however, the estimated maximum fast flux levels and total exposures were in excellent agreement with postirradiation data obtained at Battelle on samples with maximum neutron exposure. A calculated wall temperature profile versus the axial core position, as supplied by Bettis personnel, is given in Figure 1.

TABLE 2. OPERATING HISTORY OF THE J-10 PRESSURE LOOP DURING IRRADIATION
FOR 38,794 MEGAWATT-DAYS IN THE ETR FOR A TOTAL ACCUMULATED
FAST-NEUTRON (>1 MEV) EXPOSURE OF 7.15×10^{21} NVT(9)

<u>Operating Conditions</u>		
	<u>Design</u>	<u>Actual</u>
Pressure, psig	2,500	2,200
Pressure Stress, psi	14,100	12,400
Gamma Heat, w per g, peak	35	25
Water Inlet Temperature, F	600	450-500
Wall ΔT, F	268	191
Maximum Wall Temperature, F	898	~ 700
Thermal Stress, psi	67,200	48,000
Wall Thickness, in.	0.182	0.188
Yield Strength, psi	30,000 (at 70 F)	62,500 (at 70 F)
Ultimate Strength, psi	75,000 (at 70 F)	92,600 (at 70 F)
<u>Exposure</u>		
<u>Cycle</u>	<u>Megawatt-Days</u>	<u>Report</u>
22	595	IDO-16595
23	1,662	IDO-16581
24	975	IDO-16585
25	1,648	IDO-16589
26	3,063	IDO-16598
27	4,467	IDO-16603
28	4,134	IDO-16618
<u>Cycle</u>	<u>Megawatt-Days</u>	<u>Report</u>
29	2,129	IDO-16619
30	3,364	IDO-16627
31	1,358	IDO-16641
32	5,310	IDO-16645
33	5,086	IDO-16649
34	3,499	IDO-16676
35	1,505	IDO-16680

A schematic of the pressure tube and outer loop of the J-10 loop is shown in Figure 2. The section of the tube which passed through the reactor core was removed for testing. Both inner and outer tube sections arrived at Battelle in two pieces, a 19-in.-long top and 17-in.-long bottom section. The inner loop was 2.200 in. in OD by 0.188 in. thick and the outer loop was 2.450 in. in OD by 0.092 in. thick. The north axis of the loop was identified prior to arrival at Battelle by a 1/4-in.-diameter hole drilled near the top of the upper sections and near the bottom of the lower sections.

FIGURE 1. CALCULATED J-10 WALL TEMPERATURE

FIGURE 2. SCHEMATIC SECTION OF J-10 PRESSURE TUBE AND GAS JACKET

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HOT-CELL EXAMINATION

Surface Condition

Examination of the tubes was initiated shortly after they were received at the Battelle Hot-Cell Facility. The four tubes were viewed under a stereomicroscope and at this time it was noted that the surface of the inner loop, in particular, contained mottled areas indicative of selective oxidation. Temperature measurements obtained on the surface of the inner loop showed a 10 F higher temperature than ambient cell temperature, due to residual gamma heating. The temperature of the outer loop was not measured.

Sectioning of J-10 Tubes

The pressure tube of the J-10 loop was slit into six approximately equal strips about 1.15 in. in width. This was sufficient to produce 36 approximately 5.0-in.-long pieces suitable for machining into tensile specimens. Prior to cutting into specimen length, the strips were scanned to measure the intensity of the emitted gamma rays as a function of position along the length of the section. The procedure for slitting the loop sections was as follows:

- (1) The tubes were mounted in a specially designed jig and slit longitudinally with a 1/32-in.-wide Norton A60-OE7 Alumdum abrasive wheel mounted on a shaft of a modified milling cutter.
- (2) The initial cut on each tube was made through the identification hole located on the north axis. The tube was slit along the entire 17- or 19-in. length, then rotated clockwise 1.15 in. (circumferential measurement) and slitted again. Six strips approximately 1.12 in. wide were produced from both the top and bottom sections by this procedure from the pressure tube, while three strips plus a half-section were produced from the outer loop sections.
- (3) The strips were identified by indelible marker as they were removed from the tube sections in order to maintain original identity. The strips were numbered ST-2 through ST-7.

Gamma Scan of Loop Strips

The strips produced by the slitting techniques were subjected to scanning by a gamma-ray spectrometer in order to aid in selecting areas of low, intermediate, and high flux exposure for testing. The relative intensity of the gamma radiation emitted by the strips was determined from gamma-ray spectrographic traces made by adjusting the spectrometer for maximum activity response from Mn^{54} standards, and performing physical scans on strips at these energy settings. The Mn^{54} isotope, whose energy peak occurs at 0.835 Mev, was selected for scanning on the basis that it is produced in iron-bearing materials by the nuclear reaction $\text{Fe}^{54}(\text{n}, \text{p})\text{Mn}^{54}$.

Traces were made on the top and bottom sections of each of the six strips using the techniques discussed above. Plots of the relative gamma intensity, as determined from the individual inner loop strips, were prepared; and upon the basis of plots, sections that received low, intermediate, and high levels of fast-neutron flux exposure were located and marked for subsequent sectioning. The location of the test specimens relative to calculated loop temperature and analyzed fast-neutron flux is shown graphically in Figure 3. The data shown in this figure include those from specimens tested in the as-irradiated condition (supported by AEC Division of Reactor Development funds) as well as those from the Bettis series of specimens which were annealed prior to testing. The latter data constitute the bulk of this report.

Dosimetry

The total fast-neutron exposure of the J-10 tube had been calculated on the basis of data from the Phillips Petroleum Company. No attempt had been made to accumulate cycle-to-cycle data from analysis of either nickel- or iron-wire dosimeters. An estimate of the total fast flux was ascertained by utilizing the $\text{Fe}^{54}(\text{n},\text{p})\text{Mn}^{54}$ reaction and analyzing filings from individual specimens cut from the inner and outer tube. This reaction appeared worthy of consideration due to its longer half-life (300 days), an estimated cross section of 54 millibarns, and the fact that burnup due to thermal-neutron absorption was insignificant. The total fast fluxes calculated on the basis of these analyses are given in Table 3.

The technique followed for these determinations was as follows:

- (1) A known weight of stainless steel was dissolved in hydrochloric acid and diluted to a known volume. An ion-exchange column was prepared by slurring Dowex 2-X-8, 100 to 200 mesh, in a glass tube 5 cm long with an inside diameter of about 1.3 cm. In order to regulate the flow, a stopcock was placed below the column and an inlet funnel equipped with a stopcock and ground-glass joint was fitted above the column. The resin was initially activated by passing concentrated hydrochloric acid through the column at a rate of about 1 ml per min.
- (2) An aliquot of the dissolved specimen was placed on the column through the funnel. The Mn^{54} was eluted by passing 9 N HCl through the column at about 1 ml per min. An aliquot of the effluent was then radioassayed for Mn^{54} .

The counting accuracy for the Mn^{54} has been shown to be within ± 15 per cent. Although this limit of accuracy will present some scatter in values, the $\text{Fe}^{54}(\text{n},\text{p})\text{Mn}^{54}$ reaction can provide useful dosimeter data from the test samples themselves. Data from samples taken from specimens cut from the tube at approximately the same horizontal reactor core plane are shown in Figure 4. Some difference in values may have resulted from fracture location within the reference section. These graphs show, in addition to the scatter resulting from the counting accuracy, an effect related to location of the sample taken with respect to fuel location. Any radial variation would not have been detected as entire wall thickness was used as part of chemical-analysis sample.

TABLE 3. TEST RESULTS OBTAINED ON IRRADIATED TENSILE

Specimen	Approximate Temperature of Irradiation, F	Fast-Neutron (>1 Mev) Flux Exposure ^(a)		Temperature of Test, F
		Instantaneous, nv	Integrated, nvt	
				<u>Gas Jacket,</u>
LT-4-L-1	120	0.91×10^{14}	1.8×10^{21}	75
LT-4-I-2	120	0.8×10^{14}	3.5×10^{21}	75
LB-3-H	120	3.4×10^{14}	6.5×10^{21}	75
Control	--	0	0	75
LT-3-L	120	3.1×10^{14}	2.0×10^{21}	750
LT-3-I	120	2.4×10^{14}	4.5×10^{21}	750
LT-3-H	120	2.7×10^{14}	5.1×10^{21}	600
Control	--	0	0	750
				<u>Inner Loop,</u>
ST-3-L	510	1.0×10^{14}	1.9×10^{21}	75
ST-2-I	625	2.0×10^{14}	3.9×10^{21}	75
ST-2-H	735	3.7×10^{14}	7.1×10^{21}	75
ST-2-L	540	1.1×10^{14}	2.1×10^{21}	750
SB-2-I	740	2.7×10^{14}	5.1×10^{21}	750
SB-2-H	760	3.6×10^{14}	6.9×10^{21}	750
Control ^(b)	--	--	--	70
Handbook data ^(c)	--	--	--	750
				<u>Inner Loop,</u>
ST-7-L	540	0.87×10^{14}	1.7×10^{21}	75
ST-7-I	675	2.9×10^{14}	5.6×10^{21}	75
ST-7H (d)	735	1.7×10^{14}	3.3×10^{21}	75
SB-7-L	700	1.2×10^{14}	2.3×10^{21}	750
SB-7-I	760	1.9×10^{14}	3.7×10^{21}	750
SB-7-H	760	3.4×10^{14}	6.5×10^{21}	750
				<u>Inner Loop,</u>
ST-4-I	680	0.0091×10^{14}	1.7×10^{19}	75
SB-5-I	760	0.52×10^{14}	1.0×10^{21}	75
SB-4-H	760	--	--	75
ST-5-H	735	2.7×10^{14}	5.3×10^{21}	75
ST-5-L	510	2.6×10^{14}	5.0×10^{21}	750
SB-3-I	760	--	--	750
ST-5-I	670	1.2×10^{14}	2.3×10^{21}	750
ST-3-H ^(e)	735	--	--	750
SB-5-H	760	0.89×10^{14}	1.7×10^{21}	750
				<u>Inner Loop,</u>
ST-3-I ^(f)	670	0.49×10^{14}	9.00×10^{20}	75
SB-6-I	760	--	--	75
SB-3-H	760	3.4×10^{14}	6.50×10^{21}	75
ST-6-H	735	3.3×10^{14}	6.30×10^{21}	75
ST-6-L ^(g)	510	0.047×10^{14}	9.00×10^{19}	750
SB-4-I	760	--	--	750
ST-6-I	670	2.4×10^{14}	4.60×10^{21}	750
ST-4-H	735	--	--	750
SB-6-H	760	3.3×10^{14}	6.3×10^{21}	750

(a) Exposures based on Mn⁵⁴ ratio.

(b) From McInnes Company, reports on sample cut from as-machined J-10 pressure tube.

(c) Metals Handbook, Vol 1, Eighth Edition, p 503.

(d) Only 1.5 mg of sample was analyzed. Small weighing errors could have caused large changes in analysis of exposure. Sample should have duplicated ST-2-H.

SPECIMENS MACHINED FROM SECTIONS OF THE J-10 LOOP

0.2 Per Cent Offset Yield Strength, 10 ³ psi	Ultimate Tensile Strength, 10 ³ psi	Fracture Strength, 10 ³ psi	Uniform Elongation, per cent	Total Elongation, per cent	Reduction of Area, per cent
<u>Initial Test Run</u>					
100.0	103.5	191.0	>18.0	31.0	54.6
95.2	101.3	192.5	>14.8	26.0	57.0
102.5	108.0	226.0	>18.6	25.0	60.5
46.8	99.7	--	> 8.5	50.4	61.8
62.5	66.7	118.0	--	13.5	54.0
65.4	69.3	123.0	7.7	11.5	54.6
71.8	73.3	115.0	9.0	12.5	48.3
36.5	67.6	--	--	26.2	62.9
<u>Initial Test Run</u>					
132.0	135.0	215.0	1.2	4.3	53.5
143.0	145.0	231.0	1.2	6.5	54.5
143.0	149.0	250.0	2.0	10.3	58.2
103.0	103.0	124.0	0.6	4.5	42.7
115.0	115.0	138.0	0.7	1.5	40.8
120.0	120.0	175.0	0.8	3.5	52.5
52.5-60.7	91.3-93.0	--	--	53-61	58-72
40-50	70-80	--	--	45-50	--
<u>Duplicate Test Run</u>					
130.0	134.0	247.0	2.0	6.5	59.1
139.5	144.8	264.0	1.8	10.6	59.8
143.0	147.0	278.0	1.0	9.4	61.5
115.3	115.3	239.0	0.6	4.2	64.0
123.0	123.5	162.0	0.7	4.2	45.5
123.6	125.8	150.0	0.8	4.1	40.5
<u>Annealed for 1 Hr at 1000 F</u>					
77.5	99.0	190.0	>16.0	35.5	58.7
72.3	98.0	183.0	>20.0	44.3	56.2
69.4	94.5	193.5	>15.0	38.0	59.1
75.0	96.0	208.0	>14.0	41.0	63.3
47.5	64.0	101.0	>18.0	24.0	50.2
55.0	75.4	108.5	14.2	21.2	50.8
58.0	71.4	118.5	11.9	19.5	55.2
87.5	88.5	122.5	1.1	7.5	47.3
54.4	69.2	117.5	11.1	16.6	55.8
<u>Annealed for 1 Hr at 1800 F</u>					
51.4	85.5	145.0	>26.0	42.9	49.9
38.4	87.8	132.0	>24.0	49.0	37.1
55.8	86.5	130.0	>25.0	51.0	42.9
36.5	77.5	121.0	>25.0	41.2	36.9
20.8	61.0	95.0	>27.0	35.9	46.3
44.7	65.4	120.0	18.0	23.4	58.0
33.0	62.5	87.5	>21.5	26.2	42.9
43.6	61.5	96.2	14.5	20.1	53.6
27.2	62.5	172.0	>28.0	41.2	67.3

(e) Erratic thermocouple operation resulted in annealing temperature below 1000 F.

(f) Annealing temperature went to 1930 F for 2 min, then decreased to 1800 F in about 6 min.

(g) Annealing temperature went to 1950 F for 3 min, then decreased to 1800 F in about 7 min.

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FIGURE 3. TEMPERATURE AND RELATIVE FLUX PROFILE OF J-10 PRESSURE TUBE AND SPECIMEN LOCATIONS

FIGURE 4. ESTIMATED TOTAL FAST FLUX EXPOSURE WITH RESPECT TO LOCATION ON PRESSURE TUBE AS DETERMINED BY Mn^{54} ANALYSIS

Heat Treatment of Specimen Blanks

A total of eighteen irradiated specimen blanks removed from various exposure levels of the J-10 inner loop were annealed at BMI according to information received from personnel at Bettis. Those blanks receiving the annealing treatments included nine which were heated for 1 hr at 1000 F and nine which were heated for 1 hr at 1800 F. A 5-kw Lepel electronic power supply was used in conjunction with a specially designed induction core contained in a Plexiglas cover to provide the required thermal conditions. Specimen temperatures were monitored by means of a thermocouple positioned in the center of each specimen blank. An argon flow of 35 ft³ per min (in the case of the 1000 F anneal) or 45 ft³ per min (1800 F anneal) was used as a protective cover during the period of temperature annealing. A series of out-of-cell heating tests on unirradiated Type 347 stainless steel segments of similar geometry established the proper power adjustment necessary to maintain the required heating and cooling sequences. The heating and cooling sequences requested by Bettis Atomic Power Laboratory personnel were as follows:

- (1) 1000 F anneal: The specimen was heated at 200 F per min to 1000 F, held at this temperature for 1 hr, and cooled at 25 F per min to 600 F. The total elapsed time was about 1 hr 25 min.
- (2) 1800 F anneal: The specimen was heated at a rate of 200 F per min to 1000 F, at 125 F per min to 1500 F, at 30 F per min to 1800 F, held at 1800 F for 1 hr, then cooled to 1000 F at a rate of about 57 F per min and from 1000 to 600 F at 25 F per min. The total elapsed time was about 1 hr 50 min.

In most of the annealing tests it was possible to maintain the thermal conditions requested by BAPL. The proper heating conditions were rather easily maintained, although cooling rates at the higher temperature anneal gave some difficulty to achieve. In two instances, temperature overrides to about 1950 F were observed; in a third case, R-F feedback into the temperature recorder caused it to record a higher temperature than actually prevailed in the specimen. Representative temperature curves for the 1000 and 1800 F anneals are shown in Figure 5.

Machining of the Tensile-Test Specimens

The tensile-specimen geometry selected for evaluating core sections of the J-10 loop was dictated to some extent by the curvature of the loop and the amount of material available. The size of the specimens ultimately prepared for evaluation included an over-all length of 5 in.; a gage section 2.0 in. long by 0.250 in. wide, and a shoulder width of 1.12 in. This thickness of the inner loop specimens was about 0.187 in. This is not standard for sheet-type tensile specimens, but was considered of sufficient size to give good results. A drawing of the specimen design ultimately selected for testing is shown in Figure 6.

Considerable difficulty was encountered in machining of the as-irradiated stainless steel billets into tensile specimens. Initial attempts were made to machine the tensile specimens by means of a 1-in. -diameter end-mill cutter attached to a conventional milling machine. When this method proved ineffective because of the combined effects of

FIGURE 5. REPRESENTATIVE TIME-TEMPERATURE CURVES FOR SPECIMENS GIVEN 1-HR ANNEALS

FIGURE 6. CURVED TENSILE SPECIMENS MACHINED FROM J-10 LOOP

The outer jacket was 2.265 in. in OD and 2.450 in. in ID; the inner loop was 2.200 in. in OD and 1.824 in. in ID.

radiation hardening and work hardening, a newly designed cutting tool was constructed to perform the machining task. This cutting tool consisted of an air-driven, high-speed motor connected to a carbide-tipped rotary file. A series of cams was used to guide the cutter and establish the necessary specimen geometry. By means of this cutter, it was possible to remove 2 to 4 mils of metal from the workpiece per pass. Tests on unirradiated samples of Type 347 stainless steel showed that this material was not work hardened during cutting, probably due to the small size of the cutter and its very high rate of rotation. Temperature measurements obtained by thermocouples placed adjacent to the cutter showed a temperature rise of only 30 F during cutting.

The difficulty noted above was not nearly so pronounced in machining of the annealed specimens. Generally, the specimens were machined with no more difficulty than is normally encountered in machining unirradiated stainless steel. The relative ease with which the machining of the annealed billets was accomplished was indicative of the large decrease in hardness resulting from the heat treatment.

Tensile-Testing Experimental Procedure

The irradiated J-10 specimens were tested either at room temperature (about 75 F) or 750 F. Those specimens tested at the elevated temperature were heated in a 3-in. - diameter resistance furnace that enclosed the grip ends of the tester to minimize heat losses from the ends of the specimen. Test specimens were placed in the furnace, brought to 750 F, and held at that temperature 30 min prior to the application of a load to the sample. Specimen temperatures were monitored by thermocouples strapped to the center gage section of each specimen.

The specimens were tested with a Riehle Model PH-30 tensile tester, using a strain rate of 0.005 in. per in. per min from point of yield to specimen fracture. Specimen elongation was measured and recorded with a Riehle Model DC-10 compressometer whose output signal activated one leg of an x-y recorder. Load-versus-elongation curves were autographically obtained. The data obtained from these tensile tests were converted to normal stress-versus-strain data and are reported in Table 3. Also included in the table for comparison are data from a series of tests of the same material and exposures but in the as-irradiated condition. These data on as-irradiated material are discussed in BMI-1609.

The uniform elongation was determined by ascertaining and recording the elongation at maximum load from the load-versus-strain curves. The maximum load was well defined in most instances, but for tests in which it was not because of the range limitation of the compressometer, the uniform elongation is reported greater than the total elongation recorded. Total-elongation measurements were based on measurements made by fitting the fractured specimen together and measuring its total length after fracture. It was assumed that the plastic deformation occurred in the reduced gage section.

Photographs of the fracture zone from the front and the side of each specimen were made at 4X magnification. These are shown in Figures A-1 through A-24 in the Appendix. The reduction-of-area calculations were made by measuring the width and thickness of the samples at the fractures to determine the specimen area at fracture. These measurements were made from the photographs by comparing with a scale graduated into 0.01-in. divisions which had been photographed at 4X magnification along with each specimen. The fracture strength was calculated by using the load at fracture and dividing by the calculated cross-sectional area at fracture.

Test Results

A comparison of the pre- and postirradiation mechanical properties showed that the yield and ultimate strengths of the Type 347 stainless steel were greatly increased following irradiation, whereas the ductility, as reflected by decreases in reduction of area, uniform elongation, and total elongation, was decreased. The most startling results were those that revealed that the pressure-tube, irradiated in the stressed state and at about 750 F, gave values for ultimate strength and 0.2 per cent yield strength that were approximately 35 to 40 per cent greater than for the gas jacket, which was irradiated at 120 F in the unstressed condition. The total elongation showed a decrease of about 80 per cent for the pressure tube as compared to the gas jacket. Increased test temperatures seemed to increase the spread in property values between the pressure tube and the gas jacket.

The effect of postirradiation annealing at 1000 and 1800 F upon the mechanical properties of specimens machined from the J-10 loop was to reduce the ultimate and yield strength and increase the ductility. Using the as-irradiated properties of Type 347 stainless steel as a base, the 0.2 per cent yield strength was decreased about 41 to 51 per cent and 62 to 75 per cent after annealing specimens for 1 hr at 1000 F or 1 hr at 1800 F, respectively. The ultimate-strength values were also decreased by about 31 to 43 per cent and 40 to 51 per cent after 1-hr anneals of 1000 and 1800 F, respectively. There did not seem to be a correlation between the amount of recovery of tensile properties obtained by annealing with the levels of irradiation exposure the specimens had received. The largest change noted upon annealed specimens was an increase in ductility. Changes of several hundred per cent over the as-irradiated values were produced by both annealing treatments.

Both annealing temperatures were effective in producing partial recovery of the preirradiation mechanical properties of the Type 347 stainless steel, but of the two, the 1-hr anneal at 1000 F appeared to produce the best combination of mechanical properties for further service. A substantially higher yield strength is retained after the lower temperature anneal than after annealing at 1800 F.

The appearance of the fractures produced in test specimens of the J-10 pressure loop is shown in Figure 7. Included are photomacrographs of as-irradiated high- and low-exposure specimens and high- and low-exposure specimens tested after 1-hr anneals at 1000 and 1800 F. The photomacrographs show that necking in the region of fracture is reduced in specimens that received the 1-hr anneals, particularly in the specimens receiving the 1800 F anneal. This is interpreted as an indication of increased uniform elongation, rather than localized deformation in the region of fracture only.

Hardness

In addition to the use of tensile properties as a measure of irradiation effect, Rockwell hardness measurements were made on the shoulder of each tensile sample. Comparisons were made between the hardness as irradiated and the hardness after annealing. The average hardness of the as-irradiated material was about 32.0 on the Rockwell C scale. After 1 hr at 1000 F the hardness was decreased to about 8 Rockwell C. Irradiated materials annealed 1 hr at 1800 F showed a still further reduction

4X

HC10075

Low-Flux-Exposure Specimen

4X

HC10069

High-Flux-Exposure Specimen

a. Specimens Tested in the As-Irradiated Condition

4X

HC10103

Low-Flux-Exposure Specimen

4X

HC10109

High-Flux-Exposure Specimen

b. Specimens Tested After 1-Hr Anneal at 1000 F

4X

HC10114

Low-Flux-Exposure Specimen

4X

HC10060

High-Flux-Exposure Specimen

c. Specimens Tested After 1-Hr Anneal at 1800 F

FIGURE 7. APPEARANCE OF FRACTURED TENSILE SPECIMENS OF DIFFERENT TEST CONDITIONS

in hardness. The hardness was found to be in the range 38 to 55 on the Rockwell A scale. Conversion of these Rockwell A readings to the Rockwell C scale shows that the higher readings correspond to about 2 or 3 Rockwell C.

These changes in hardness are summarized in Table 4. It can also be noted that there was only a slight variation in hardness of the as-irradiated specimens representing different irradiation exposures. Although little difference was observed in the hardness of the as-irradiated specimens, the hardness values obtained after annealing indicate greater recovery effects in the specimens from regions of the pressure tube receiving a lower irradiation exposure. This was apparent for both the 1000 F and 1800 F annealing heat treatments.

TABLE 4. HARDNESS OF J-10 PRESSURE-TUBE SPECIMENS BEFORE AND AFTER HEAT TREATMENT AT 1000 AND 1800 F

Specimen ^(a)	Hardness ^(b)		
	Rockwell C	Rockwell C	Rockwell A
	Before Heat Treatment	After 1 Hr at 1000 F	After 1 Hr at 1800 F
ST-4-I	32.4	5.8	--
SB-5-I	30.6	10.6	--
SB-4-H	32.8	15.6	--
ST-5-H	34.8	11.2	--
ST-5-L	32.6	2.4	--
SB-3-I	33.4	3.6	--
ST-5-I	32.2	10.6	--
ST-3-H	34.6	14.8	--
SB-5-H	31.8	14.2	--
ST-3-I	33.8	--	53.2
SB-6-I	33.6	--	50.8
SB-3-H	31.0	--	55.0
ST-6-H	34.4	--	46.0
ST-6-L	32.8	--	42.4
SB-4-I	32.8	--	37.8
ST-6-I	31.4	--	51.2
ST-4-H	33.8	--	54.2
SB-6-H	31.2	--	48.4

(a) Letter suffix indicates amount of irradiation exposure: H = high, I = intermediate, and L = low.

(b) Average of five hardness impressions.

Metallographic Examination

Metallographic examinations were performed upon shoulder sections of several tensile specimens. The examinations included as-irradiated specimens from sections receiving low, intermediate, and high irradiation exposures as well as specimens representing exposures that were subjected to postirradiation anneals of 1 hr at 1000 or 1800 F.

Examinations were performed on the as-polished specimens, and after etching with a glyceresia swab etch or 10 per cent oxalic acid electroetch. It was noted that the structure of all of the specimens examined was similar, and it was not possible, except in two instances, to delineate, on the basis of microstructure alone, the as-irradiated specimens from specimens annealed after irradiation and/or control samples. The two exceptions were the specimens which had been unintentionally heated above 1800 F. One had a very large grained structure, apparently caused by the overheating. The second specimen had a lesser amount of grain growth, but exhibited a second phase in some of the grain boundaries. The second phase at the grain boundaries may be similar to the heavy uneven carbide reported by Bailey and Silliman in postirradiation-annealed Type 347 stainless steel.⁽²⁾

Typical microstructures of J-10 pressure-tube material before and after irradiation and after postirradiation annealing at 1000 and 1800 F are shown in Figure 8. Microstructures of the two specimens inadvertently overheated are shown in Figure 9.

CONCLUSIONS

The effects of 1-hr anneals at 1000 and 1800 F upon property changes of specimens machined from an AISI Type 347 stainless steel pressure tube from an in-pile loop have been determined. This pressure tube was removed from the ETR J-10 position in March, 1961, after having been exposed to approximately 38,800 megawatt-days of full-power operation at a maximum in-pile temperature of about 780 F. The following information relevant to the effects of these anneals on the irradiated Type 347 stainless steel was obtained:

- (1) Using as-irradiated properties of the J-10 pressure tube as a base, the 0.2 per cent offset yield strength decreased about 41 to 51 and 62 to 75 per cent, after 1-hr anneals at 1000 and 1800 F, respectively.
- (2) The ultimate strength decreased from as-irradiated values by about 31 to 43 and 40 to 51 per cent after 1-hr anneals at 1000 and 1800 F, respectively.
- (3) The ductility, as reflected by elongation, was greatly increased by annealing. Uniform elongations were increased from approximately 1 per cent in the as-irradiated material to 15 and 25 per cent by the 1000 and 1800 F anneals, respectively. Uniform elongation was noted in annealed specimens within the entire gage section, rather than localized elongation which was observed only in the area of fracture of as-irradiated specimens.
- (4) The 1-hr anneal at 1000 F appeared to produce a better combination of mechanical properties than the annealing at the higher temperature. The higher temperature anneal produced, in general, lower ultimate and yield strengths in the irradiated specimens than were present in the unirradiated controls.

100X

Etched

2970A

a. Unirradiated WAPD Photo

200X

Etched

HC10168

b. Irradiated to 6.9×10^{21} NVT

200X

Etched

HC10180

c. Irradiated to 5.3×10^{21} NVT and Annealed
1 Hr at 1000 F

200X

Etched

HC10189

d. Irradiated to 6.5×10^{21} NVT and Annealed
1 Hr at 1800 FFIGURE 8. MICROSTRUCTURES OF CONTROL AND IRRADIATED AISI TYPE 347 STAINLESS STEEL SPECIMENS
FROM THE J-10 PRESSURE TUBE

a. Specimen Given a Postirradiation Anneal for 1 hr at Some Unknown Elevated Temperature

Temperature was not known because R. F. feedback into recorder gave faulty reading. Note the large grain size.

b. Specimen Inadvertently Heated to 1960 F

Note the general rounding of grains, and the presence of grain-boundary precipitates.

FIGURE 9. MICROSTRUCTURES OF TWO SPECIMENS GIVEN POSTIRRADIATION ANNEALS IN EXCESS OF 1800 F

- (5) The microstructure of the irradiated Type 347 stainless steel was not appreciably changed by 1-hr anneals at 1000 or 1800 F. In addition, the microstructure of as-irradiated Type 347 stainless steel as determined through routine hot-cell metallographic examinations was similar to that of unirradiated control samples.

Exceptions to the above were noted in two cases of irradiated samples that were inadvertently heated above 1800 F. These samples underwent grain growth, and one contained a grain-boundary precipitate which looked similar to that present in sensitized Type 304 stainless steel.

The results of the program indicate that the recovery of the mechanical properties of irradiated Type 347 stainless steel to approach those of the unirradiated material can be accomplished by 1-hr anneals at 1000 or 1800 F. Although nearly complete recovery of preirradiation properties occurs upon annealing at these temperatures, there have been no data on the effects of subsequent irradiation upon annealed specimens.

ACKNOWLEDGMENTS

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- (2) Bailey, R. E., and Silliman, M. A., "Effects of Irradiation on the Type 347 Stainless Steel Flow Separator in the EBR-1 Core", Symposium on Radiation Effects on Materials, Vol 3, ASTM-STP-233 (1958), p 84.

WEM/FRS:mjd

APPENDIX

FRACTURE ZONES OF SPECIMENS TESTED

A-1

FIGURE A-1. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN ST-7-L AFTER TESTING AT 75 F

No heat treatment was employed before testing.

A-2

200X

Microstructure

HC10156

4X

Edge

HC10068

4X

Flat

HC10069

FIGURE A-2. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN ST-7-H AFTER TESTING AT 75 F

No heat treatment was employed before testing.

200X

Microstructure

HC10161

4X

Edge

HC10071

4X

Flat

HC10072

FIGURE A-3. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN ST-7-I AFTER TESTING AT 75 F

No heat treatment was employed before testing.

FIGURE A-4. FRACTURE ZONE OF SPECIMEN SB-7-I AFTER TESTING AT 750 F

No heat treatment was employed before testing.

4X

Edge

HC10089

4X

Flat

HC10090

FIGURE A-5. FRACTURE ZONE OF SPECIMEN SB-7-L AFTER TESTING AT 750 F

No heat treatment was employed before testing.

A-6

200X

HC10169

Microstructure

4X

HC10095

Edge

4X

HC10096

Flat

FIGURE A-6. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN SB-7-H AFTER TESTING AT 750 F

No heat treatment was employed before testing.

FIGURE A-7. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN ST-3-I AFTER TESTING AT 75 F

This specimen was annealed at 1800 F for 1 hr before testing. The temperature went to 1930 F for 2 min and then decreased to 1800 F in about 6 min during the annealing treatment.

200X

HC10190

Microstructure

4X

HC10121

Edge

4X

HC10120

Flat

FIGURE A-8. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN SB-3-H AFTER TESTING AT 75 F

This specimen was annealed at 1800 F for 1 hr before testing.

4X

HC10086

Edge

4X

HC10087

Flat

FIGURE A-9. FRACTURE ZONE OF SPECIMEN SB-6-H AFTER TESTING AT 750 F

This specimen was annealed at 1800 F for 1 hr before testing.

FIGURE A-10. FRACTURE ZONE OF SPECIMEN SB-6-I AFTER TESTING AT 75 F

This specimen was annealed at 1800 F for 1 hr before testing.

200X

HC10194

Microstructure

4X

HC10065

Edge

4X

HC10066

Flat

FIGURE A-11. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN ST-6-L AFTER TESTING AT 750 F

This specimen was annealed at 1800 F for 1 hr before testing. The temperature went to 1950 F for 3 min and then decreased to 1800 F in about 7 min during the annealing treatment.

FIGURE A-12. FRACTURE ZONE OF SPECIMEN ST-6-I AFTER TESTING AT 750 F

This specimen was annealed at 1800 F for 1 hr before testing.

FIGURE A-13. FRACTURE ZONE OF SPECIMEN ST-6-H AFTER TESTING AT 75 F

This specimen was annealed at 1800 F for 1 hr before the test.

4X

Edge

HC10098

4X

Flat

HC10099

FIGURE A-14. FRACTURE ZONE OF SPECIMEN ST-4-H AFTER TESTING AT 750 F

This specimen was annealed at 1800 F for 1 hr before testing.

FIGURE A-15. FRACTURE ZONE OF SPECIMEN SB-4-I AFTER TESTING AT 750 F

This specimen was annealed at 1800 F for 1 hr before testing.

4X

Edge

HC10117

4X

Flat

HC10118

FIGURE A-16. FRACTURE ZONE OF SPECIMEN SB-3-I AFTER TESTING AT 750 F

This specimen was annealed at 1000 F for 1 hr before testing.

200X

HC10173

Microstructure

4X

HC10050

Edge

4X

HC10051

Flat

FIGURE A-17. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN ST-5-H AFTER TESTING AT 75 F

This specimen was annealed at 1000 F for 1 hr before testing.

4X

Edge

HC10053

4X

Flat

HC10054

FIGURE A-18. FRACTURE ZONE OF SPECIMEN ST-5-1 AFTER TESTING AT 750 F

This specimen was annealed at 1000 F for 1 hr before testing.

FIGURE A-19. FRACTURE ZONE OF SPECIMEN SB-5-H AFTER TESTING AT 750 F

This specimen was annealed at 1000 F for 1 hr before testing.

FIGURE A-20. FRACTURE ZONE OF SPECIMEN SB-5-I AFTER TESTING AT 75 F

This specimen was annealed at 1000 F for 1 hr before testing.

200 X

Microstructure

HC10176

4X

Edge

HC10056

4X

Flat

HC10057

FIGURE A-21. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN ST-5-1 AFTER TESTING AT 750 F

This specimen was annealed at 1000 F for 1 hr before testing.

200X

HC10180

Microstructure

4X

HC10102

Edge

4X

HC10103

Flat

FIGURE A-22. FRACTURE ZONE AND MICROSTRUCTURE OF SPECIMEN ST-4-I AFTER TESTING AT 75 F

This specimen was annealed at 1000 F for 1 hr before testing.

FIGURE A-23. FRACTURE ZONE OF SPECIMEN SB-4-H AFTER TESTING AT 75 F

This specimen was annealed at 1000 F for 1 hr before testing.

4X

HC10111

Edge

4X

HC10112

Flat

FIGURE A-24. FRACTURE ZONE OF SPECIMEN ST-3-H AFTER TESTING AT 750 F

This specimen was annealed at 1000 F for 1 hr before the test.