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PROGRESS ON LMFBR CLADDING, STRUCTURAL,
AND COMPONENT MATERIALS STUDIES
DURING JULY, 1970, THROUGH JUNE, 1971

Annual Report, Task 32

Final Report, Task 14

Donald L. Keller

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BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Prepared for U. S. Atomic Energy Commission
Under Contract W-7405-eng-92

Report Date: July, 1971

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REPORTS RELATING TO REACTOR MATERIALS, TECHNOLOGY, AND
SAFETY ISSUED DURING THE PERIOD
JULY, 1970, THROUGH JUNE, 1971

- BMI-1884 "Progress on High-Temperature Fuels Technology During February Through April, 1970", by Donald L. Keller and Walston Chubb.
- BMI-1885 "An Evaluation of the Applicability of Existing Data to the Analytical Description of a Nuclear-Reactor Accident Quarterly Progress Report for April Through June, 1970", by Wayne A. Carbiener and Robert R. Ritzman.
- BMI-1886 "Progress on Development of Fuels and Technology for Advanced Reactors During July, 1969 Through June, 1970 Annual Report", by Donald L. Keller
- BMI-1887 "Investigation of the Initiation and Extent of Ductile Pipe Rupture Phase II Progress Report for April-June, 1970", by Robert J. Eiber, Willard A. Maxey, Arthur R. Duffy, and Thomas J. Atterbury
- BMI-1888 "ADEP-1 and ADEP-2, One- and Two-Group Kinetics Codes", by Richard S. Denning
- BMI-1889 "Progress on LMFBR Cladding, Structural, and Component Materials Studies During July, 1969 Through June, 1970 Annual Report", by Donald L. Keller
- BMI-1890 "An Evaluation of the Applicability of Existing Data to the Analytical Description of a Nuclear-Reactor Accident Quarterly Progress Report for July Through September, 1970", by Wayne A. Carbiener and Robert L. Ritzman.
- BMI-1891 "Progress on LMFBR Cladding, Structural and Component Materials Studies During July Through September, 1970 Quarterly Report", by Donald L. Keller.
- BMI-1892 "Investigation of the Initiation and Extent of Ductile Pipe Rupture Phase II Progress Report for July-September, 1970", by Robert J. Eiber, Willard A. Maxey, and Arthur R. Duffy
- BMI-1893 "Progress on Development of Fuels and Technology for Advanced Reactors During July Through September, 1970 Progress Report", by Donald L. Keller
- BMI-1894 "Development and Operation of An Apparatus for In-Pile Creep Studies of LMFBR Fuels", by Richard A. Robinson, Samuel J. Basham, and James S. Perrin.
- BMI-1895 "Compatibility of Nitride Reactor Fuels With Austenitic Stainless Steels", by John C. Hebeisen, Donald P. Moak, and William H. Pfeifer
- BMI-1896 "Progress on LMFBR Cladding, Structural, and Component Materials Studies During October Through December, 1970 Quarterly Report", by Donald L. Keller
- BMI-1897 "An Evaluation of the Applicability of Existing Data to the Analytical Description of a Nuclear-Reactor Accident Quarterly Progress Report for October Through December, 1970", by Wayne A. Carbiener, and Robert L. Ritzman
- BMI-1898 "Progress on Development of Fuels and Technology for Advanced Reactors During October Through December, 1970", by Donald L. Keller
- BMI-1899 "Effect of Irradiation on the Creep of Uranium Dioxide", by James S. Perrin and William R. D. Wilson
- BMI-1900 "A Compendium of Properties and Characteristics for Selected LMFBR Cladding Materials", by Jack G. Conner and Stanley W. Porembka
- BMI-1901 "A Survey of LMFBR Component Materials Limitations and Requirements", by Arthur A. Bauer and Jack G. Conner
- BMI-1902 "Investigation of the Initiation and Extent of Ductile Pipe Rupture Phase II Progress Report for October-December, 1970", by Robert J. Eiber, Willard A. Maxey, and Arthur R. Duffy
- BMI-1903 "Progress on LMFBR Cladding, Structural, and Component Materials Studies During January Through March, 1971 Quarterly Report", by Donald L. Keller.
- BMI-1904 "An Evaluation of the Applicability of Existing Data to the Analytical Description of a Nuclear-Reactor Accident Quarterly Progress Report for January Through March, 1971", by Wayne A. Carbiener and Robert L. Ritzman
- BMI-1905 "Progress on Development of Fuels and Technology for Advanced Reactors During January Through March, 1971 Quarterly Report", by Donald L. Keller

HIGHLIGHTS OF RESULTS DURING REPORTING PERIOD

This report summarizes the results of DRDT-supported studies at Battelle-Columbus on cladding, component, and structural materials of interest for LMFBR applications. The work under way includes a program directed toward developing mechanical-property design data for reactor materials (Task 32); this report details the results of the program during Fiscal Year 1971. Task 14, a comprehensive long-term study directed toward the elucidation of the effects of irradiation on cladding and structural alloys, ended at the conclusion of Fiscal Year 1971, and the results given herein constitute the Final Report for Task 14. Highlights of the results obtained on each task are cited below.

Development of Mechanical-Property Design Data for Reactor Materials (Task 32)

Low-cycle strain-controlled fatigue tests were conducted on both Incoloy 800 and Type 304 stainless steel. The fatigue tests on Incoloy 800 were run at 1000, 1200, and 1400 F. Specimens tested with hold times at the tensile strain limit exhibited significantly lower fatigue lives than specimens tested with no hold times. An analysis of the cyclically stable stress-relaxation curves has been developed which allows the prediction of fatigue lives at lower strain ranges from data at higher strain ranges.

Creep-rupture tests were conducted on Incoloy 800 at 1400 F to complement the fatigue data obtained on this material. A least-squares Larsen-Miller parameter analysis of the Incoloy 800 creep data available in the literature was performed. The results of the Incoloy 800 studies are being provided to the ASME Boiler and Pressure Vessel Committee for use in establishing the new code covering materials above 800 F.

The low-cycle strain-controlled fatigue tests conducted on Type 304 stainless steel specimens were run at 1000 F and 1200 F. It was found that specimens tested with tension hold times had appreciably lower fatigue lives than specimens tested with no hold times. In addition to these tests, a series of tests was run on Type 304 stainless steel specimens to determine the influence of the amount of fatigue damage at 1000 and 1200 F under more complex loading conditions.

Studies of the Effects of Irradiation on Cladding and Structural Alloys (Task 14)

Effects of irradiation, at various temperatures and fluence levels in the ETR, on the mechanical properties of a number of alloys are discussed. The alloys included Type 347 and 348 stainless steel, UMo-50, Haynes Stellite 25,

Tenelon, and nickel. Irradiations were carried out at 50, 400, and 650 C with the fast fluences ranging from 1.4×10^{21} to 3.0×10^{22} nvt. Tensile, fatigue, impact, and creep tests were performed on selected specimens. Tensile tests were performed on as-irradiated specimens as well as on specimens that had been irradiated and annealed. It was found that irradiation-produced displacement-type damage, which causes large changes in mechanical properties at low test temperatures, can be removed by annealing at elevated temperature. It was found also that changes in mechanical properties are a maximum for irradiation at 400 C, intermediate for irradiation at 50 C, and smallest for irradiation at 650 C. At elevated temperatures, (450 to 600 C, depending on material) all materials tested underwent a loss of ductility. This ductility loss could not be restored by postirradiation annealing at temperatures up to 1350 C. The influence of nickel on the elevated-temperature embrittlement was investigated. It was found that nickel was not necessary for the embrittlement but alloys containing nickel underwent the most severe losses in ductility because of high helium generation as a result of thermal-neutron reactions with nickel. Both fatigue life and stress-rupture life at 650 C were significantly reduced as a consequence of the irradiation-induced elevated-temperature embrittlement.

Tensile tests were performed on Type 347 and Type 348 stainless steel specimens that were irradiated in the EBR-II at 400 and 650 C to fluences ranging up to 3.9×10^{22} nvt ($E > 0.1$ Mev). The tensile tests were performed at temperatures ranging from 25 C to 750 C and were performed on as-irradiated specimens as well as on irradiated and annealed specimens. Tensile tests indicated that ductility was decreased and strength was increased by irradiation at 400 C for all testing temperatures. However, the irradiation-induced changes in properties were significantly less than those resulting from irradiation in a mixed thermal and fast flux spectrum in the ETR. Annealing for 1 hr at 980 C was found to restore the preirradiation tensile properties at 600 C and below.

The mechanical properties of Types 304 and 316 stainless steel were determined after the specimens had been shock strengthened by means of either a flyer plate or contact explosives. Shocking was found to significantly increase the yield strength and proportional limit, moderately increase the ultimate strength, and generally decrease the ductility, although an intermediate increase in ductility with increasing shocking pressure was noted in material shock strengthened by means of a flyer plate. The magnitude of the change in tensile properties increased with shocking pressure (80 to 320 kbar) and held for all testing temperatures. The microstructure of shock-strengthened austenitic stainless steel exhibited fine striations when examined by light microscopy, while a high dislocation density and mechanical twins were identified by transmission electron microscopy. Annealing of the shock-strengthened material for 1 hr at 900 C resulted in the disappearance of the defect structure and the changes in mechanical properties. Shock strengthening reduced the minimum creep rate of Type 316 stainless steel by a factor of about 100 and that of Type 304 stainless steel by a factor of about 1000 at 650 C for stresses ranging from 25,000 to 35,000 psi.

Impact properties of shock-strengthened Type 316 stainless steel were determined by instrumented Charpy impact tests. The material was shocked at room temperature by a pressure of 80 and 160 kbar and at 445 and 665 C by a pressure of 320 kbar. Increase in shocking pressure resulted in progressively lower values of energy required for fracture and of fracture toughness (K_{IC}). The critical crack length also decreased with shocking pressure, with the critical crack length of the shocked material being independent of testing temperature. The ratio of dynamic yield to static yield (measure of strain-rate sensitivity) was found to decrease with increasing shocking pressure; the ability of the material to strain harden was reduced and the shocked material failed in a brittle manner at temperatures as high as 650 C.

A variety of creep-rupture results have been obtained on ferritic 2-1/4 Cr-1 Mo alloy materials containing 0.012 and 0.042 w/o carbon. Larson-Miller plots of available stress-rupture data for these and commercial materials indicate a sequential reduction in rupture life with decrease in carbon content for applied stresses below about 13,000 psi for the 0.012 w/o carbon material and below about 15,000 psi for the 0.042 w/o carbon material. At higher stresses, although still below the yield strength, the low-carbon alloys apparently undergo strain hardening; resultant increases in rupture life produce a sharp break in the Larson-Miller plot for these alloys. It was found possible to describe the minimum creep rates of the 0.012 and 0.042 w/o carbon alloys, respectively, by the following equations:

$$\dot{\epsilon} = 5.76 \times 10^{14} \sigma^5 \exp(-54,960/RT),$$

$$\dot{\epsilon} = 7.45 \times 10^{13} \sigma^5 \exp(-54,960/RT),$$

for temperatures in degrees Rankine. The creep constants indicate that the creep strength of the 0.042 w/o carbon material is about 1.5 times greater than that of the 0.012 w/o carbon alloy.

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DEVELOPMENT OF MECHANICAL-PROPERTY DESIGN
DATA FOR REACTOR MATERIALS (AEC-DRDT)

PROGRAM MANAGEMENT

J. S. Perrin

The ASME has established the Boiler and Pressure Vessel Code to provide rules of safety governing the design, the fabrication, and the inspection of boilers and other pressure vessels, including vessels for nuclear plants. Incoloy Alloy 800 is of current interest for use as a pressure-vessel material for LMFBR's and other reactors. This material is presently covered under the code only up to 800 F.

It is now desired to use Alloy 800 in nuclear pressure vessels at temperatures significantly above 800 F, but these vessels cannot be built and approved until the ASME code includes higher temperatures for Alloy 800. The first phase of this work is, therefore, to obtain low-cycle strain-controlled fatigue design data on Alloy 800 in the temperature range from 800 to 1400 F for submission to the ASME Boiler and Pressure Vessel Committee. In addition, Alloy 800 creep data will be analyzed in conjunction with the fatigue data to make predictions of the behavior of Alloy 800 subjected to both fatigue and creep damage.

A second phase of this program involves generating similar code data for Type 304 stainless steel. These studies will be concerned with determining the continuous-cycling fatigue behavior at 1000 and 1200 F. In addition, the effect of hold times on the fatigue behavior will be studied at the same two temperatures.

A later phase of the work will be concerned with extending the Type 304 stainless steel studies to Type 316 stainless steel. In addition, other future phases of the program may include the generation of needed general mechanical-property data for components such as vessel cladding, piping, tubing, pressure-vessel heads, vessel nozzles, and sheets. The work will include measurements of ultimate and yield strengths, strain values, fatigue properties, fracture properties, and creep and stress-rupture properties of both base metal and welded joints.

LOW-CYCLE-FATIGUE EVALUATION OF REACTOR MATERIALS

C. E. Jaske, H. Mindlin, and J. S. Perrin

Objective of Program

To develop low-cycle-fatigue design information for several reactor materials at elevated temperatures where significant creep/fatigue interaction may occur.

Approach Being Used

Strain-controlled low-cycle-fatigue tests are being conducted to generate data for analysis and reduction to a format that can be used to present low-cycle-fatigue design information.

Experimental procedures used in this program are similar to those used previously in elevated-temperature, low-cycle-fatigue evaluations of three austenitic stainless steel alloys⁽¹⁻⁴⁾. Tests were performed under completely reversed strain cycling at a constant strain rate, $\dot{\epsilon}$. When maximum strain limits were reached, the direction of straining was either reversed or held constant for a time period and then reversed.

The basic test apparatus consists of a servocontrolled electrohydraulic system operated in closed-loop axial strain control. Axial load is applied to an hourglass-shaped specimen heated by high-frequency induction. Diametral changes in the specimen are measured with a special diametral extensometer, and load is measured with a strain-gage-fitted load cell in series with the specimen. Diametral strain and load signals are combined and converted to an equivalent axial strain using an analog computer.

Significant Accomplishments During Fiscal Year 1971

Curves of total strain range, $\Delta\epsilon_t$, versus fatigue life were developed for Type 304 stainless steel at both 1000 and 1200 F over a range of lifetimes between 10^2 and 10^6 cycles to failure. For lives less than 10^4 cycles, the fatigue strength at 1000 F was slightly above that at 1200 F. However, there was no significant difference in fatigue strength for lives greater than 10^4 cycles. These curves provide valuable information for incorporation in elevated-temperature design rules.

A useful graphical method has been found for predicting the effect of hold time at maximum tensile strain upon the constant-amplitude, full-reversed low-cycle-fatigue behavior of both Type 304 stainless steel and Alloy 800. This

method was developed and verified through a series of hold-time fatigue tests of Alloy 800 at 1000, 1200, and 1400 F. The same technique also was used on previous data⁽⁴⁾ from hold-time fatigue tests of Type 304 stainless steel at 1200 F and on similar data that were generated at 1000 F for Type 304 stainless steel.

Using constant-strain-rate incremental step tests, a new understanding of the cyclic stress-strain behavior of both Type 304 stainless steel and Alloy 800 has been developed. Both monotonic and cyclic stress-strain curves have been defined for these alloys at temperatures between 70 and 1400 F. Only one test at each temperature is required to generate these curves.

Cumulative damage of Type 304 stainless steel has been studied under variable-amplitude loading conditions at both 1000 and 1200 F. In tests with hold times at tension strain, the length of hold time was also varied. Using a linear damage rule, damage summations between 0.5 and 1.0 were obtained for the cases studied. For use in design, the rule could be conservatively applied by limiting total damage to 0.5.

Creep-rupture data from 171 tests of solution-annealed Alloy 800 were analyzed using the Larson-Miller parameter and least-squares regression. Results of the analysis were used to develop best-fit stress-rupture curves for this alloy between 1000 and 1500 F. The resulting curves correlate well with the original data. A few creep-rupture tests of Alloy 800 have been conducted at 1400 F and the results are reported.

Summary of Research Carried Out

The objective of work during Fiscal Year 1971 was to study the low-cycle fatigue behavior of Incoloy Alloy 800 and Type 304 stainless steel at temperatures in the creep range. Continuous cycling, hold time, and cyclic stress-strain fatigue studies were conducted on both Incoloy Alloy 800 and Type 304 stainless steel. A complex loading history/fatigue study of Type 304 stainless steel and creep studies of Incoloy Alloy 800 were also conducted.

A description of the materials used during the past year is given in Table A-1. Identification code letters were used in assigning numbers to each specimen tested to differentiate among the groups of materials. Typical microstructures of each material are shown in Figures A-1 and A-2. Group A of Incoloy Alloy 800 was the same material as used by Conway⁽⁵⁾, and the Type 304 stainless steel was the same material as used in previous low-cycle-fatigue studies^(1,4,6,7).

TABLE A-1. DESCRIPTION OF MATERIAL USED IN EXPERIMENTAL STUDIES

Identification Code	Solution-Annealed Incoloy Alloy 800		Type 304 Stainless Steel
	A	D	SS
Heat	HH8968A ^(a)	HH3113A ^(b)	55697 ^(c)
ASTM Grain Size	2.4	3.0	1.7
Chemical Analysis, w/o			
Carbon	0.05	0.06	0.05
Manganese	0.84	0.90	0.88
Iron	44.38	45.17	Balance
Sulfur	0.008	0.007	0.010
Silicon	0.29	0.38	0.49
Copper	0.45	0.25	0.21
Nickel	31.41	32.11	9.53
Chromium	21.61	20.35	18.29
Aluminum	0.38	0.36	--
Titanium	0.33	0.39	<0.01
Phosphorus	--	--	0.019
Molybdenum	--	--	0.18
Lead	--	--	<0.001
Calcium	--	--	0.11
Tin	--	--	0.006
Nitrogen	--	--	0.32

- (a) Hot-finished 3/4-in. -diameter bar, solution annealed at 2100 F for 1 hr, was furnished by the Huntington Alloy Products Division of The International Nickel Company, Huntington, West Virginia.
- (b) Hot-finished 1-in. -diameter bar, solution annealed at 2100 F for 1 hr, was furnished by the Huntington Alloy Products Division of The International Nickel Company, Huntington, West Virginia.
- (c) Hot-rolled 5/8-in. -diameter bar, annealed at 1959 F for 1 hr, was furnished by BNWL.

Continuous Cycling Studies. Low-cycle-fatigue tests of Type 304 stainless steel were performed at both 1000 and 1200 F. The work at 1200 F was to complement earlier studies^(5,6), and the work at 1000 F was to provide information at a temperature of current interest to the LMFBR program. To determine whether the new heat of solution-annealed Incoloy Alloy 800 (Heat HH3113A) being used for creep studies had fatigue properties similar to those of previous heats [see Reference (7) and BMI-1889], several low-cycle-fatigue tests were conducted at 1000, 1200, and 1400 F.

Experimental work was performed using the procedures and apparatus described earlier. Specimens were machined to one of the two configurations

100X

Etched

7E350

a. Group A Material

100X

Etched

2F900

b. Group B Material

FIGURE A-1. MICROSTRUCTURES OF SOLUTION-ANNEALED INCOLOY ALLOY 800 MATERIALS USED IN PROGRAM

100X

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FIGURE A-2. MICROSTRUCTURE OF TYPE 304 STAINLESS STEEL USED IN PROGRAM

shown in Figure A-3*. The test section of either type of specimen has the same hourglass shape. The only difference between the two designs is the method of gripping. After machining, the test section of each specimen was polished with successively finer grades of emery paper to produce a surface finish of 16 μ in. rms or better, with finishing marks parallel to the longitudinal axis of the specimen. No heat treatment was given to the specimens after machining.

Load was applied to each specimen axially and the computed axial strain signal was programmed to follow a constant-amplitude, fully reversed triangular waveform with a constant strain rate of $4 \times 10^{-3} \text{ sec}^{-1}$. Load was measured by a standard, strain-gage-fitted load cell in series with the specimen and diametral strain was measured with a special extensometer.

The extensometer consists of adjustable sensing arms of high-purity alumina connected to a bracket consisting of two parallel beams joined by a flexible ligament that acts as an elastic hinge. Diameter changes are magnified three times before they are measured at the other end of the extensometer by a sensitive and magnetically shielded linear variable differential transformer (LVDT). The transformer and armature of the LVDT are mounted on opposite beams and the position of the armature relative to the transformer may be adjusted after the test specimen has been brought to the desired temperature. The output signal obtained from the LVDT is proportional to the diameter change in the specimen. The extensometer is calibrated using a micrometer capable of 0.00001-in. resolution.

Specimens were heated by high-frequency induction and temperature was controlled with a standard proportional-type power controller. The geometry of the heating coil was designed to minimize temperature gradients in the test section. A specimen instrumented with several thermocouples along the central 1/2-in. portion was used to verify this design. Three Chromel-Alumel thermocouples were spot welded to the surface in the gage section of each specimen. Two of the thermocouples were 3/16 in. on one side of the minimum diameter, and the other one was the same distance to the other side of the minimum diameter. One of the first two thermocouples provided the feedback signal to the controller. The other two were used to make minor adjustments in the coil position and obtain a uniform temperature profile along the specimen.

Buttonhead-type specimens (Figure A-3a) were clamped in a loading fixture similar to that described by Slot, et al.⁽³⁾. Threaded specimens (Figure A-3b) were gripped using a fixture arrangement similar to that described by Feltner and Mitchell⁽⁸⁾. The lower end of the specimen was threaded into an adapter attached to the load cell which was in turn attached to the hydraulic actuator. The upper end of the specimen was attached to the loadframe crosshead through a Wood's metal type of liquid-solid grip⁽⁹⁾. Both the upper and lower adapters were continuously water cooled to avoid overheating of the fixtures.

*Specimens from Group A of Incoloy Alloy 800 were machined and polished by Gulf Energy and Environmental Systems, Inc., San Diego, California.

a. Buttonhead Grip Specimen

b. Threaded Grip Specimen

FIGURE A-3. DIMENSIONS OF FATIGUE SPECIMENS

In the initial assembly of either type of fixture, alignment was checked using a carefully machined specimen that was instrumented with four strain gages. The gages were oriented longitudinally and spaced at 90-deg intervals around the diameter. Final alignment adjustments were made so as to minimize bending strains under both compressive and tensile loadings. Bending strains were 1 percent or less of the average axial strain after final adjustment and several repeated installations of the specimen. During the course of the program, the alignment was periodically rechecked.

During each fatigue test, load was continuously recorded using a time-base strip chart recorder. These records were used to obtain hardening behavior and information on when cracking occurs just before total separation of the specimen. Load-strain hysteresis loops were periodically recorded during each test using an X-Y recorder. These loops were used to obtain values of total, plastic, and elastic strain range.

Results of the continuous cycling tests are summarized in Table A-2. The three values of fatigue life that are reported are defined as follows:

N_0 = number of cycles where tension load starts to decrease just before complete fracture occurs

N_5 = number of cycles where tension load decreases 5 percent below a cyclically stable value just before complete fracture occurs

N_f = number of cycles to complete fracture.

Values of plastic strain range that are listed in Table A-2 were defined as the width of the cyclically stable stress-strain hysteresis loops (usually recorded near half the number of cycles to fracture, $N_f/2$). Values for elastic strain range were defined as the difference between total and plastic strain ranges. Values for stable stress range were obtained from load-versus-time records (usually near $N_f/2$ also). Stress was calculated from the load and the original specimen minimum cross-sectional area at temperature. For Incoloy Alloy 800 at 1000 F (Specimens 1D and 2D), the stress range was never well stabilized; hence, values of stress range are reported for a specific number of cycles, as noted. Results for Incoloy Alloy 800 are compared with previous data reported by Conway⁽⁵⁾ and data reported in BMI-1889 in the plots of $\Delta\epsilon_t$ versus N_f in Figure A-4. Points for the Group D material (the triangles) fall very close to the mean curves for Group A material (the open circles and squares). In general, the results for all four heats of Incoloy Alloy 800 can be represented by a single curve at each temperature.

Similar curves of $\Delta\epsilon_t$ versus N_f for the Type 304 stainless steel are presented in Figure A-5. For comparison, data reported at 1200 F in earlier work^(6,7) are also presented. As would be expected, there is little difference between the two sets of data at 1200 F. (Compare the squares with the triangles.) Data at 1000 F fall above the data at 1200 F for fatigue lives less than 10^4 cycles

TABLE A-2. RESULTS OF CONSTANT-AMPLITUDE, CONTINUOUS CYCLING FATIGUE TESTS AT A STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

Material	Specimen Identification ^(a)	Temperature, F	Stable Strain Range, percent			Stable Stress Range, ksi	Fatigue Life, cycles			Total Time to Fracture, hr
			Total	Plastic	Elastic		N ₀	N ₅	N _f	
Incoloy Alloy 800	1D	1000	1.97	1.47	0.50	136 ^(b)	200	460	792	2.10
	2D	1000	0.49	0.18	0.31	90.5 ^(c)	30,650	31,500	37,220	25.9
	6D	1200	1.19	0.73	0.46	103	950	1,300	1,763	1.15
	7D	1200	0.48	0.12	0.36	68.9	--	--	20,488	13.7
	8D	1200	0.45	0.12	0.33	67.6	17,500	19,600	30,000	20.8
	4D	1400	2.08	1.76	0.32	86.4	243	261	376	0.98
	5D	1400	0.50	0.21	0.29	55.4	5,950	6,500	6,601	4.40
Type 304 stainless steel	SS12 ^(d)	1000	2.94	2.50	0.44	116	295	345	381	0.42
	SS1	1000	2.92	2.40	0.52	108	335	360	390	1.62
	SS13	1000	2.04	1.63	0.41	104	600	685	737	2.05
	SS3	1000	1.95	1.51	0.44	99.6	600	680	812	2.25
	SS2	1000	0.96	0.63	0.33	76.0	2,000	2,320	2,876	3.99
	SS21	1000	0.63	0.36	0.27	59.3	8,500	9,000	10,283	8.57
	SS4	1000	0.34	0.16	0.18	50.2	26,800	27,600	31,553	21.9
	SS17	1000	0.27	0.04	0.23	48.2	>825,000	>825,000	967,734	403
	SS6 ^(e)	1000	0.13	--	0.13	27.2	>1,520,257	>1,520,257	>1,520,257	>438
	SS18 ^(d)	1200	2.73	2.30	0.43	97.7	355	364	385	0.48
	SS15 ^(f)	1200	1.89	1.50	0.39	103	650	713	744	0.39
	SS14	1200	0.76	0.46	0.30	60.2	3,475	3,530	3,881	2.61
	SS5	1200	0.47	0.25	0.22	53.0	6,300	6,450	6,882	4.72
	SS10	1200	0.44	0.22	0.22	48.4	9,000	9,500	12,253	8.53
	SS16	1200	0.31	0.15	0.16	32.9	170,000	180,000	189,891	79.4

(a) All Incoloy Alloy 800 specimens and Specimens SS16, SS17, and SS21 had threaded ends. The other specimens all had buttonhead ends.

(b) At 150 cycles.

(c) At 26,000 cycles.

(d) Strain rate was $1.5 \times 10^{-2} \text{ sec}^{-1}$.

(e) Test was stopped without failure.

(f) Strain rate was $2.5 \times 10^{-2} \text{ sec}^{-1}$.

FIGURE A-4. FATIGUE LIFE CURVES FOR SOLUTION-ANNEALED INCOLOY ALLOY 800 AT A STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

FIGURE A-5. FATIGUE LIFE CURVES FOR TYPE 304 STAINLESS STEEL AT A STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

to fracture. However, for fatigue lives between 10^4 and 10^6 cycles, data at both 1000 and 1200 F seem to fall on a single curve. In this long-life region, the slope of the curve becomes very shallow. If this trend is substantiated out to 10^8 cycles to failure, it may be practical to define a fatigue strength limit for continuous cycling when less than 10^8 cycles are accumulated.

Hold-Time Studies. The effect of hold time on the low-cycle-fatigue behavior of solution-annealed Incoloy Alloy 800 was investigated at 1000, 1200, and 1400 F. This work was a direct extension of that reported previously in BMI-1889, where the effects of tension only, compression only, and combined tension and compression hold times were investigated for 10-min-long hold times at total strain ranges of about 2 to 3 percent. Since tension hold times proved to be the most detrimental, work during this year has been concentrated upon determining the effect of hold times up to 300 min long with total strain ranges as low as 0.5 percent. In addition, the effect of tension hold time on the low-cycle-fatigue behavior of Type 304 stainless steel at 1000 F was investigated.

Details of the experimental procedures for these hold-time studies were the same as those described for the continuous cycling tests, with the exception of program waveform for each strain cycle. For all tests with tension hold times, the waveform shown in Figure A-6a was used. For the test with compression hold times (Specimen 32A), the waveform of Figure A-6b was used. The length of hold time, Δt , was maintained constant throughout each test.

Results of all the hold-time fatigue tests are summarized in Table A-3. Specimen 32A was tested with a compression hold time to complete a matrix of six tests with compression hold times. Results of the other five tests were reported previously (see Table A-2 of BMI-1889). The fatigue life of Specimen 32A was about the same as that of Specimen 25A, which was also tested at 1000 F, but with both tension and compression hold times. That is, the fatigue life was about 25 percent that expected for continuous cycling. This reduction in life is just about the same as was obtained for 10-min hold times in tension only at 1000 F.

The effect of hold time on fatigue life of the solution-annealed Incoloy Alloy 800 is illustrated in Figure A-7. Data for no-hold-time strain cycling were obtained by Conway⁽⁵⁾ in earlier studies. Tests with compression hold times had fatigue lives about the same or slightly less than the no-hold-time tests. In contrast, tests with only tension hold times had significantly lower fatigue lives.

For constant lengths of tension hold time, the data are well approximated by a series of straight lines emanating from the point where the extrapolated no-hold-time curves intercept the one-cycle-to-failure line. The slopes of these lines become steeper as length of hold time increases; and at constant hold time, the ratio of hold-time cyclic life to no-hold-time cyclic life decreases as the strain ranges decreases.

A similar effect of hold time on the fatigue life of Type 304 stainless steel is shown in Figure A-8. The hold-time data at 1200 F were obtained in previous

a. Tension Hold Time With No Compression Hold Time

b. Compression Hold Time With No Tension Hold Time

FIGURE A-6. SCHEMATIC OF PROGRAM WAVEFORMS FOR LOW-CYCLE-FATIGUE TESTS WITH HOLD TIMES

TABLE A-3. RESULTS OF HOLD-TIME FATIGUE TESTS AT A STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

Material	Specimen Identification ^(a)	Temperature, F	Length of Hold Time ^(b) , min	Stable Strain Range, percent			Stable Stress Range, ksi	Fatigue Life, cycles			Total Time to Fracture, hr
				Total	Plastic	Elastic		N ₀	N ₅	N _f	
Incoloy Alloy 800	41A	1000	10	0.53	0.19	0.34	90.0	5200	5400	5985	1001
	42A ^(c)	1000	10	0.52	0.18	0.34	88.0	>1403	>1403	>1403	>237
	29A	1000	60	2.38	1.82	0.56	150	192	205	220	221
	45A	1000	60	2.39	1.80	0.59	143	180	202	253	255
	36A	1000	300	2.38	1.90	0.48	152	61	66	72	360
	32A	1000	10 ^(b)	2.25	1.59	0.66	141	450	470	508	86.5
	37A	1200	10	0.54	0.29	0.25	85.8	950	980	>998 ^(d)	>170 ^(d)
	40A	1200	10	0.54	0.34	0.20	83.7	800	850	983	167
	31A	1200	60	2.39	1.98	0.41	128	44	49	56	56.2
	35A	1200	60	2.43	2.01	0.42	130	46	50	58	58.2
	43A	1200	60	0.53	0.38	0.15	72.7	285	310	340	341
	38A	1200	300	2.37	1.96	0.41	140	35	40	47	235
	30A	1400	10	0.55	0.43	0.12	57.1	650	710	871	146
	33A	1400	10	0.55	0.43	0.12	54.4	580	620	800	134
	47A	1400	60	1.76	1.55	0.21	75.5	90	105	132	131
	44A	1400	60	0.56	0.44	0.12	54.7	300	315	399	399
	46A ^(d)	1400	300	1.75	1.58	0.17	77.5	>53	>53	>53	>265
Type 304 stainless steel	SS7	1000	10	2.22	1.80	0.42	109	230	275	366	60.4
	SS22	1000	10	1.04	0.65	0.39	79.4	750	890	1431	222
	SS9	1000	30	2.20	1.80	0.40	108	150	175	223	111
	SS8	1000	60	2.21	1.82	0.39	106	130	157	184	183
	SS11 ^(e)	1000	300	2.13	1.75	0.38	101	>90	>90	>90	>444

(a) All specimens except Specimen SS22 had buttonhead ends; Specimen SS22 had threaded ends.

(b) All hold times were in tension except for Specimen 32A, which had a compression hold time.

(c) Test was suspended because of longer than anticipated fatigue life.

(d) Function generator malfunctioned and test had to be stopped.

(e) Stability problem in control loop caused specimen to fail prematurely.

a At 1000 F

b At 1200 F

c At 1400 F

FIGURE A-7. THE EFFECT OF HOLD TIME ON THE LOW-CYCLE-FATIGUE BEHAVIOR OF SOLUTION-ANNEALED INCOLOY ALLOY 800 AT A STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

a. At 1000 F

b At 1200 F

FIGURE A-8. THE EFFECT OF TENSION HOLD TIME ON LOW-CYCLE-FATIGUE BEHAVIOR OF TYPE 304 STAINLESS STEEL AT AN AXIAL STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

studies^(4, 7) of this material. The same method of approximating the hold-time data with a fan-shaped series of straight lines that was described above for Incoloy Alloy 800 also works well for Type 304 stainless steel.

The technique of approximating tension hold-time fatigue data that is illustrated in Figures A-7 and A-8 can be used to predict fatigue lives for low strain ranges from data at high strain ranges. Because economic considerations limit the number and length of tests that can be conducted at long hold times, such a prediction method is a useful tool for extrapolating relatively short-time test results. This prediction technique, based upon the present data, should be limited to hold times of less than 300 min and strain ranges greater than about 0.25 percent until more data become available.

The reduction in fatigue life approaches a saturation level as the length of tension hold time increases. This saturation effect is more noticeable at the higher temperature than at the lower ones. It is most predominant for the stainless steel at 1200 F (Figure A-8b) where no significant difference in cyclic fatigue life is noted for tension hold times between 30 and 600 min. At 1000 F (Figure A-8a), this saturation level appears to be at hold times greater than 60 min long and it appears to be approached more slowly than at 1200 F. For Alloy 800, a saturation level is approached fastest at 1400 F and slowest at 1000 F, with 1200 F behavior intermediate, but closer to the 1400 F behavior.

The difference in saturation effect as a function of temperature is related to the stress relaxation behavior of the material. Cyclically stable stress relaxation curves that were recorded during the hold-time fatigue tests of Incoloy Alloy 800 have been analyzed and reduced to the data format of Table A-4. These data are similar to those reported by Conway^(4, 7) for Type 304 stainless steel at 1200 F. Terms used in Table A-4 are defined in Figure A-9 for the case where both tension and compression hold times are present. (For cases where only one type of hold time is present, some of these terms are redundant.) Data were taken from the load-versus-time curves near $N_f/2$ for each test.

In general, the tensile stresses relax to lower values more quickly at higher temperatures. At 1400 F, they relax to lower values faster than at 1200 F. At 1000 F, relatively little stress relaxation takes place in 10-min hold-time tests and a small amount takes place in 60- and 300-min hold-time tests. When the stresses relax to very low values, very little creep damage is done during the time at low stress compared to the creep damage done during the early portion of the hold time. Once the stress has time to relax to such a low value, little creep damage is done and little further reduction in fatigue life occurs as the length of hold time increases.

Cyclic Stress-Strain Behavior. For engineering design under low-cycle-fatigue conditions, it is important to consider the cyclic hardening and/or softening behavior of metals. The cyclic stress-strain curve is a useful tool for consideration of cyclic hardening and softening in comparison to the

TABLE A-4 CYCLICALLY STABLE STRESS-RELAXATION DATA FOR LOW-CYCLE FATIGUE OF SOLUTION-ANNEALED INCOLOY
ALLOY 800 AT AN AXIAL STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

Specimen	Tension	Compression	Stress	Relaxed	Tensile Stress		Compressive		Relaxed	Relaxed	Mean	Time to
	Hold	Hold Time,	Range	Stress	ksi		ksi		Tensile	Compressive	Relaxed	Fracture
	Time, min	min	ksi	Range, ksi	Maximum	Minimum	Maximum	Minimum	Stress ksi	Stress ksi	Tensile Stress ksi	at Tensile Stress, hr
Tested at 1000 F												
11A	10	0	109	105	57.2	53.2	-52.2	-52.2	4.0	0	55.2	81.3
12A	10	0	104	99.5	54.0	49.0	-50.0	-50.0	5.0	0	51.5	81.8
41A	10	0	90.0	90.0	45.0	45.0	-45.0	-45.0	0	0	45.0	998
42A	10	0	88.0	88.0	44.0	44.0	-44.0	-44.0	0	0	44.0	>233
29A	60	0	150	140	78.6	69.0	-71.0	-71.0	9.6	0	73.8	220
45A	60	0	143	128	71.4	56.0	-71.6	-71.6	15.4	0	63.7	252
36A	300	0	152	138	78.4	64.0	-73.6	-73.6	14.4	0	71.2	360
25A	10	10	140	131	72.0	66.8	-67.6	-64.0	5.2	3.6	69.4	94.2
32A	0	10	141	136	72.4	72.4	-68.4	-63.4	0	5.0	72.4	0
Tested at 1200 F												
9A	10	0	86.8	72.3	44.7	29.6	-42.2	-42.2	15.1	0	37.1	25.7
10A	10	0	80.1	64.2	41.0	24.6	-39.1	-39.1	16.4	0	32.8	32.2
37A	10	0	86.0	74.7	43.2	31.9	-42.8	-42.8	11.4	0	37.6	166
40A	10	0	82.9	72.9	41.8	31.9	-41.0	-41.0	10.0	0	36.9	164
31A	60	0	128	98.4	67.7	38.2	-60.2	-60.2	29.5	0	53.0	56.0
35A	60	0	130	95.6	67.1	32.3	-63.3	-63.3	34.9	0	49.7	58.0
43A	60	0	72.7	58.8	36.3	22.3	-36.5	-36.5	13.9	0	29.3	340
38A	300	0	140	102	69.7	31.9	-69.7	-69.7	37.8	0	50.8	235
23A	10	10	109	55.8	56.0	27.9	-52.8	-27.9	28.1	24.9	41.9	61.7
24A	0	10	118	89.6	57.8	57.8	-59.8	-31.9	0	27.9	57.8	0
Tested at 1400 F												
13A	10	0	88.5	55.6	45.7	13.9	-42.7	-42.7	31.8	0	29.8	20.3
7A	10	0	64.3	44.2	31.6	12.0	-32.6	-32.6	19.6	0	21.8	23.5
8A	10	0	61.6	41.7	30.5	11.2	-31.0	-31.0	19.3	0	20.9	36.5
30A	10	0	57.3	39.4	28.2	10.3	-29.0	-29.0	17.9	0	19.3	145
33A	10	0	54.5	37.8	26.8	10.1	-27.6	-27.6	16.7	0	18.5	133
47A	60	0	75.5	46.1	37.4	8.0	-38.2	-38.2	29.4	0	22.7	132
44A	60	0	54.7	35.8	28.8	9.9	-25.8	-25.8	18.9	0	19.4	399
46A	300	0	77.5	48.5	39.0	9.9	-38.6	-38.6	29.0	0	24.4	>265
21A	10	10	73.7	25.9	31.9	7.0	-41.8	-18.9	24.9	22.9	19.4	54.8
22A	0	10	87.1	56.4	44.0	44.0	-43.2	-12.5	0	30.7	44.0	0

SR=Stress Range, psi
 SRR=Relaxed Stress Range, psi
 STMAX=Maximum Tensile Stress, psi
 STMIN= Minimum Tensile Stress, psi
 SCMAX=Maximum Compressive Stress, psi
 SCMIN=Minimum Compressive Stress, psi
 STR=Relaxed Tensile Stress, psi
 SCR=Relaxed Compressive Stress, psi

$$\text{Mean Relaxed Tensile Stress} = (STMAX + STMIN)/2$$

FIGURE A-9. DEFINITION OF TERMS USED IN REPORTING STRESS RELAXATION DATA FOR HOLD-TIME LOW-CYCLE-FATIGUE TESTS

monotonic stress-strain curve obtained from an ordinary tensile test. Since both Incoloy Alloy 800 and Type 304 stainless steel tend to cyclically harden from their original annealed conditions, the cyclic stress-strain curve will fall above the monotonic stress-strain curve; the relative difference between these two curves gives an indication of the amount of hardening.

The cyclic stress-strain curve is usually defined⁽¹⁰⁾ as the curve formed by connecting the tips of stable hysteresis loops that have been developed during constant-amplitude low-cycle-fatigue tests of several specimens at different strain ranges. Alternative procedures of defining the cyclic stress-strain curve have been investigated by Landgraf, et al.⁽¹¹⁾. It was found that an incremental step test of a single specimen provided a good method of experimentally determining the cyclic stress-strain curve for a variety of alloys tested at room temperature. To determine whether this experimental procedure can be used for Incoloy 800 and Type 304 stainless steel at elevated temperatures, the incremental step tests summarized in Table A-5 were conducted. Except for the strain program waveform, all experimental procedures were the same as those described previously. The program waveform was generated using a digital block programmer with sequential input instructions from punched paper tape.

TABLE A-5. RESULTS OF INCREMENTAL STEP TESTS AT A
STRAIN RATE OF 4×10^{-3} SEC⁻¹

Material	Specimen Identification (a)	Temperature, F	Maximum Total Strain Range, percent	Number of Blocks to Failure
Incoloy Alloy 800	13D	70	3.04	32-5/40
	12D	800	2.99	39-35/40
	9D	1000	3.05	25
	10D	1200	3.04	16-37/40
	11D	1400	3.00	7-2/40 ^(b)
Type 304 stainless steel	SS45	70	2.95	33-4/40
	SS44	800	2.95	31-33/40
	SS26	1000	3.00	28-6/40
	SS48	1000	1.00	229-2/40
	SS25	1200	3.02	18-36/40
	SS31	1200	3.05	19-36/40

(a) All specimens had threaded ends.

(b) Specimen failed at thermocouple.

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FIGURE A-10. STRAIN-TIME WAVEFORM FOR AN INCREMENTAL STEP TEST

An example of the program waveform used for the incremental step tests is shown in Figure A-10. The strain was first increased from zero to half of the maximum total strain range to obtain a monotonic stress-strain curve. It was then reversed and cycled for 20 cycles in equal increments of decreasing strain range. The first block of strain cycling was completed by increasing the strain range in a corresponding manner for another 20 cycles. These blocks of strain cycling were then repeated until the specimen failed. Figure A-10 shows 2-1/2 blocks of such cycling. For these tests, a maximum total strain range of 3 percent was selected on the basis of the experience of Landgraf, et al. (11).

Stress-strain hysteresis loops were recorded during the initial half block of each test and periodically thereafter. A typical set of hysteresis loops for the first half block is shown in Figure A-11a. Note the large amount of cyclic hardening that occurs as successive hysteresis loops have higher stress ranges even though the strain range is decreasing. This type of behavior was typical of both Type 304 stainless steel and Alloy 800 at the temperatures used in this study. Similar recordings were made until the hardening behavior stabilized (i. e., the stress range became about the same from cycle to cycle). By drawing a line through the tips of the hysteresis loops for a stabilized record, such as that shown in Figure A-11b, it was possible to define a cyclic stress-strain curve. Once the material stabilized, series of hysteresis loops were essentially the same for both increasing and decreasing increments within the half block of cycling.

The monotonic and cyclic stress-strain curves obtained from the incremental step tests are presented in Figures A-12 and A-13. At all the temperatures where tests were conducted, both alloys exhibit a significant amount of cyclic hardening behavior, as indicated by the distance that the cyclic curves fall above the monotonic ones. To obtain a quantitative measure of the degree of cyclic hardening, a cyclic offset (0.2 percent) yield strength analogous to the conventional tensile yield strength has been obtained from each of the cyclic stress-strain curves. These cyclic and monotonic yield strengths are compared in Table A-6. The cyclic yield strength of Alloy 800 has maximum value at 1000 F, but it is relatively constant from 70 to 1200 F and drops by almost a factor of two between 1200 and 1400 F. In contrast, the cyclic yield strength of the Type 304 stainless steel decreases with increasing temperature between 70 and 1200 F. The ratio of cyclic to monotonic yield strength provides a measure of the degree of cyclic hardening. Alloy 800 shows a maximum amount of cyclic hardening at 1200 F, and Type 304 stainless steel shows a maximum amount of cyclic hardening at 1000 F.

Except at room temperature, where no data are available, stable cyclic stress-strain data from constant-amplitude, continuous cycling tests are plotted for comparison with the curves from the incremental step tests. In general, the cyclic curves from the incremental step tests provide a much better approximation of these plotted points than do the monotonic curves. The plotted points fall very close to the cyclic curves of the higher strain amplitudes, but tend to fall below the curve at lower strain amplitudes. This trend indicates the material has a "memory" for the cycles applied at high strain levels in the incremental

a. Block 1 - Decreasing Increments

b. Block 21 - Decreasing Increments

FIGURE A-11. STRESS-STRAIN HYSTERESIS LOOP FOR AN INCREMENTAL STEP TEST OF TYPE 304 STAINLESS STEEL AT 1000 F (Specimen SS26)

Constant Amplitude, Continuous Cycling

- Group A, Heat HH8968A (GEMP-732)
- Group A, Heat HH8968A
- Group B, Heat HH0901A } (BMI-1889)
- Group C, Heat HH3310A }
- △ Group D, Heat HH3113A (present study)

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FIGURE A-12. COMPARISON OF MONOTONIC AND CYCLIC STRESS-STRAIN CURVES FOR INCREMENTAL STEP TESTS OF INCOLOY ALLOY 800 AT A STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

FIGURE A-13. COMPARISON OF MONOTONIC AND CYCLIC STRESS-STRAIN CURVES FOR INCREMENTAL STEP TESTS OF TYPE 304 STAINLESS STEEL AT A STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

step tests; thus, the subsequent cycles at low strain levels are influenced by the prior history of cyclic hardening. To substantiate this hypothesis, incremental step tests are being conducted at a lower maximum total strain range of 1 percent. One of these tests (Specimen SS48 in Table A-5) has been completed for Type 304 stainless steel at 1000 F. Under these conditions, the cyclic stress-strain curve falls very close to constant amplitude points at low strain ranges and significantly below the cyclic stress-strain curve for 3 percent maximum total strain range (see Figure A-13c).

TABLE A-6. COMPARISON OF MONOTONIC AND CYCLIC YIELD STRENGTHS

Material	Temperature, F	Maximum Total Strain Range of Incremental Step Test, percent	0.2 Percent Offset Tensile Yield Strength, ksi	0.2 Percent Offset Cyclic Yield Strength, ksi	Ratio of Cyclic to Monotonic Yield Strength
Incoloy Alloy 800	70	3.04	24.9	56.6	2.27
	800	2.99	15.9	55.5	3.49
	1000	3.05	15.0	61.3	4.08
	1200	3.04	12.2	53.5	4.38
	1400	3.00	12.5	32.5	2.60
Type 304 stainless steel	70	2.95	31.4	66.3	2.11
	800	2.95	17.5	48.3	2.76
	1000	3.00	14.6	43.5	2.98
	1000	1.00	14.6	34.3	2.35
	1200	3.05	14.2	36.6	2.58

Under actual service conditions, materials are usually subjected to complex variable-amplitude loading histories rather than to constant-amplitude cycles. Therefore, the cyclic stress-strain response as measured by the incremental step tests is probably more indicative of the material behavior under service conditions than is the cyclic stress-strain response under constant-amplitude cycling. Because of the cyclic hardening influence of cycles at higher strain ranges, subsequent lower strain ranges will have higher stress amplitudes than predicted by constant-amplitude data. Since creep damage interacts with fatigue damage to produce failure at high temperatures, these higher stress amplitudes can adversely influence the fatigue life. Following this line of reasoning, lower fatigue lives than predicted from the constant-amplitude results would be predicted in such circumstances.

Fatigue Damage Under Complex Loading History. To obtain information about elevated-temperature fatigue damage under complex loading histories, a series of two-level block-loading fatigue tests has been conducted at 1000 and 1200 F. Tests were conducted both with continuous cycling and with hold times at the maximum tensile strain amplitude. Except for the complex program waveform, the experimental procedures were the same as used previously. As for the incremental step tests, a digital block programmer was used to generate the command waveforms. When constant hold times were employed, an auxiliary

timer was added to the system. For the test with variable lengths of hold time, a similar paper-tape instructed digital programmer with a digital clock time base was used to generate the command waveform.

Examples of the command waveforms for continuous cycling tests are shown in Figure A-14. A predetermined number of cycles were applied to the specimen at each of two levels in one block of loading. Each cycle was started in the compressive direction of loading. The blocks of loading were repeated until the specimen failed. In some instances, the high level of loading was first in the block as shown in Figure A-14, and in other cases the low level of loading was first. The test with variable hold times had a waveform like that shown in Figure A-6a and the value of Δt was changed from cycle to cycle.

Results of the continuous-cycling block-loading fatigue tests are summarized in Table A-7. Most of the tests were conducted with strain levels of about 1/2 and 2 percent. A few were also performed with strain levels of about 1 and 4 percent. A simple Miner type of linear damage rule was used to calculate a damage summation for each test:

$$\text{Damage summation} = \frac{n_1}{N_{f_1}} + \frac{n_2}{N_{f_2}} \quad , \quad (1)$$

where n_1 and n_2 are the numbers of cycles subjected to the specimen at Strain Levels 1 and 2, respectively, and N_{f_1} and N_{f_2} are the average fatigue lives at the same strain levels for constant-amplitude cycling. If the variable-amplitude strain cycling has no influence on fatigue life, these summations should be close to unity. However, most of them are slightly below unity, indicating a small detrimental influence of the block loading.

For these tests, there is no significant difference between the order of loading within a block. That is, it makes no difference whether the high or low strain level is applied first. An example of the hysteresis loops recorded during these tests will help to explain this observation. During the first few blocks of loading, the material cyclically hardens, as shown in Figure A-15a. The stress range quickly stabilizes, and most of the hysteresis loops for these tests look like those shown in Figure A-15b. Even though the initial blocks may have different hardening behavior with different orders of loading and numbers of cycles per block, the overall stable stress-strain behavior is very similar throughout all of the tests. The order of loading would thus be expected to have a significant effect only when there are relatively few blocks to failure, a condition that would probably not be likely in normal service.

At 1200 F, there is no evidence of an effect of the number of cycles per block. In contrast, at 1000 F the tests with a greater number of cycles per block (Specimens SS28 and SS29) give lower fatigue lives than those with fewer cycles per block (Specimens SS35, SS37, and SS42). A possible explanation of this behavior is the influence of mean stress on fatigue life. As shown in Figure A-15b, the cycles at the low strain range have a compressive mean

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a. 10 High- and 100 Low-Level Cycles per Block

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b. 1 High- and 10 Low-Level Cycles per Block

FIGURE A-14. STRAIN-TIME WAVEFORM FOR TWO-LEVEL BLOCK LOADING

TABLE A-7. RESULTS OF TWO-LEVEL BLOCK-LOADING FATIGUE TESTS OF TYPE 304 STAINLESS STEEL AT A STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

Specimen Identification(a)	Temperature, F	Total Strain Range, percent		Number of Cycles per Block		Stable Stress Range, ksi		Stable Mean Stress, ksi		Number of Blocks to Failure	Damage Summation
		Level 1	Level 2	Level 1	Level 2	Level 1	Level 2	Level 1	Level 2		
SS28	1000	2.03	0.49	10	100	99.8	72.9	-1.1	-4.7	32-96/110	0.66
SS29	1000	0.46	2.02	100	10	74.2	103	-5.0	0	32-105/110	0.62
SS35	1000	2.03	0.48	1	10	102	72.0	0	-6.6	472	0.93
SS37	1000	0.46	1.99	10	1	71.0	99.2	-8.0	-1.3	494-10/11	0.91
SS42	1000	0.45	1.97	10	1	76.1	97.9	-5.7	-1.0	432-6/11	0.77
SS38	1000	0.99	4.02	10	1	95.5	113	-3.2	-0.9	114	0.91
SS39	1000	0.97	4.00	10	1	95.5	112	-2.6	-0.5	115-7/11	0.89
SS40	1000	0.97	4.00	10	1	96.6	113	-2.0	-0.2	136-10/11	1.05
SS33	1200	2.03	0.47	10	100	80.8	58.8	0	-2.0	33-1/110	0.83
SS30	1200	0.50	2.01	100	10	57.2	78.2	-2.0	-1.0	34-104/110	0.89
SS34	1200	2.02	0.49	1	10	78.7	64.7	-0.4	-3.0	306-5/11	0.78
SS36	1200	0.47	1.98	10	1	62.3	76.8	-3.9	-0.8	399	0.97
SS43	1200	0.47	1.99	10	1	63.3	74.7	-2.5	-0.6	328-1/11	0.80
SS32	1200	4.34	1.27	10	100	100	76.3	0	0	6-16/110	0.97

(a) All specimens had threaded ends.

a. Cyclic Hardening During Block 1

b. Cycling Stable Loops During Block 16

FIGURE A-15. STRESS-STRAIN HYSTERESIS LOOPS FOR TWO-LEVEL BLOCK LOADING OF TYPE 304 STAINLESS STEEL AT 1000 F (Specimen SS28)

stress because the low-level cycles were always applied immediately following the tensile peak of a high-level cycle. Stress-time records from these tests (see Figure A-16) show that the mean stress relaxes more quickly at 1200 F than at 1000 F. At 1200 F, the mean stress during the low-level cycles relaxes to about the same value in both 10 and 100 cycles. However, at 1000 F the mean stress relaxes more during 100 low-level cycles than during 10 low-level cycles. Since the compressive mean stress would tend to have a beneficial influence on fatigue life, less damage is done in the tests with 1 high- and 10 low-level cycles per block than in the tests with 10 high- and 100 low-level cycles per block. If this hypothesis is valid, similar tests with tensile mean stress should show the opposite effect of number of cycles per block at 1000 F and little effect of number of cycles per block at 1200 F. Tensile mean stress could be introduced in the low-level cycles by applying them immediately following the compressive peak of a high-level cycle.

It is interesting to compare the stable stress-strain values of Table A-7 with the cyclic stress-strain curves shown earlier (see Figure A-17). The stable values fall closest to the cyclic stress-strain curves from the incremental step tests. Thus, this cyclic curve is probably more representative of the stress-strain behavior under more realistic variable-amplitude loading.

Results of the block-loading tests with hold times are summarized in Table A-8. As noted, programming difficulties were encountered during the first three tests. These occurred when the paper tape reading device misread a punched code and provided the digital programmer with an erroneous instruction. For Specimen SS46, the error caused the continuous cycling to be applied until the specimen failed; hence, this test was repeated. With Specimens SS47 and SS49, extraneous hold times were introduced during one cycle. The tensile hold time on Specimen SS47 was included in the damage computation and the compressive hold time on Specimen SS49 was ignored.

Damage calculations were again made using Miner's rule, with the estimated failure lives for constant-amplitude loading with hold times being taken from the appropriate curves for each length of hold time and strain range (see Figure A-8). All of the damage summations are less than unity; thus, an interactive influence of variable strain and length of hold time is indicated. The continuous cycling following hold-time cycling (Specimen SS46) appears to be the most severe case. However, this sort of loading is not usually expected in service and the result can probably be excluded from most practical design considerations.

Most of the damage summations for two-level block-loading tests are between 0.5 and 1.0. Considering the normal amount of scatter in fatigue results, this variation by a factor of two is certainly not excessive. Although these test results are very limited and cover only a few conditions, they do give a useful indication of the types of damage summations to be expected under complex loading conditions that are more indicative of actual service. If a linear damage rule is used to account for cumulative low-cycle-fatigue damage both with and without hold times, a damage limit of 1.0 would reduce the safety factor of 20 on cycles

B-1068

a. At 1000 F (Specimen SS28)

B-1069

b. At 1200 F (Specimen SS33)

FIGURE A-16. STRESS-TIME RECORDS FOR TWO-LEVEL BLOCK LOADING OF TYPE 304 STAINLESS STEEL

a At 1000 F

b At 1200 F

A-1058

FIGURE A-17. COMPARISON OF CYCLIC STRESS-STRAIN BEHAVIOR FROM TWO-LEVEL BLOCK-LOADING TESTS WITH CYCLIC STRESS-STRAIN CURVES

TABLE A-8. RESULTS OF TWO-LEVEL BLOCK-LOADING FATIGUE TESTS OF
TYPE 304 STAINLESS STEEL WITH HOLD TIMES

Specimen Identifica- tion ^(a)	Tempera- ture, F	Total Strain Range, percent		Number of Cycles per Block		Length of Hold Time, min		Number of Blocks to Failure	Damage Summation
		Level 1	Level 2	Level 1	Level 2	Level 1	Level 2		
SS46	1200	2.02	0.49	1	10	10	10	>20-9/11 ^(b)	0.38
SS47 ^(c)	1200	2.00	0.48	1	10	10	10	43-1/11	0.48
SS49 ^(d)	1000	1.99	0.48	1	10	10	10	156	0.71
SS50	1200	1.78	1.78	1	1	10	30	68	0.70

(a) Specimens SS46, SS47, and SS49 had threaded ends, and Specimen SS50 had button ends.

(b) Programmer malfunction at 20-9/11 blocks of loading caused specimen to be cycled continuously at a total strain range of 1.14 percent and a mean strain of 0.36 percent for 244 more cycles, where failure occurred.

(c) Programmer error caused hold time of Cycle 74 to be 537 min long.

(d) Programmer error caused a compression hold time ~ 600 min long on Cycle 382.

presently used in Section III of the ASME Boiler and Pressure Vessel Code by a factor of two (or to no less than 10 on cycles) at the most. Considering all the other assumptions and unknowns involved in design analysis, this is not a large reduction factor. A simple and conservative method of maintaining at least the 20 factor of safety would be to limit the damage summation to 0.5.

Creep Studies. For correlation of hold-time fatigue behavior with creep-rupture behavior of solution-annealed Alloy 800, a survey and analysis of available stress-rupture data for this alloy were made and a few creep tests were conducted on material from Group D of Incoloy Alloy 800.

Stress-rupture information on solution-annealed Alloy 800 has been obtained from four sources⁽¹²⁻¹⁵⁾. A total of 107 data points at temperatures from 1000 to 1500 F and for rupture lives between 1 and 12,000 hr have been obtained for material with a normal carbon content (usually about 0.04 w/o). For material with a controlled carbon content (0.05 to 0.10 w/o), another 64 data points are available at temperatures from 1200 to 1500 F and for rupture lives between 10 and 15,000 hr.

A least-squares regression approach described by Conway⁽¹⁶⁾ was used to perform a Larson-Miller parameter analysis of these data. The Larson-Miller parameter, P , is defined as follows:

$$P = T(C + \log_{10} t_r) \quad , \quad (2)$$

where

T = temperature, R

C = Larson-Miller constant

t_r = rupture life, hr.

If the following terms are defined:

S = stress, ksi

$Y = \log_{10} t_r$

$X_1 = 1/T$

$X_2 = \log_{10} S,$

then it is possible to express P in terms of a third-degree polynomial:

$$P = (Y + C)/X_1 = B_1 + B_2 X_2 + B_3 X_2^2 + B_4 X_2^3 \quad , \quad (3)$$

where B_1 , B_2 , B_3 , and B_4 are constants. Equation (3) can be rearranged to give the following expression:

$$Y = -C + B_1 X_1 + B_2 X_1 X_2 + B_3 X_1 X_2^2 + B_4 X_1 X_2^3 \quad (4)$$

Using a simple computer program, a regression analysis of the stress-rupture data was performed to obtain best-fit values of the constants C , B_1 , B_2 , B_3 , and B_4 . This method of analysis was used for three sample populations: (1) the normal and controlled carbon materials combined, (2) the normal material, and (3) the controlled carbon material. The best-fit constants that were calculated are listed in Table A-9.

TABLE A-9. BEST-FIT CONSTANTS OBTAINED FROM REGRESSION ANALYSIS OF STRESS-RUPTURE DATA ON SOLUTION-ANNEALED ALLOY 800

Type of Material	Sample Population	Best-Fit Value for Indicated Constant				
		C	B_1	B_2	B_3	B_4
Normal and controlled carbon combined	171	16.0	62,400	-53,400	35,700	-9,660
Normal	107	16.6	61,600	-48,400	30,800	-8,180
Controlled carbon	64	17.7	66,200	-54,200	36,400	-9,960

Plots of stress versus Larson-Miller parameter for each sample population are shown in Figure A-18, and stress-rupture plots for each sample population are shown in Figure A-19. Each plotted point represents the result of a single stress-rupture test, and the solid lines show the best-fit curves defined by Equation (3) and the constants listed in Table A-9. The solid lines in Figure A-19 are for the particular value of constant temperature indicated.

In Figure A-18, the best-fit curves fit the points well. There is a little more scatter in the data for the combined materials (Figure A-18) than in the data for either the normal material or the controlled carbon material separately (Figures A-18b and A-18c). The best-fit stress-rupture curves fit the corresponding data fairly well for the separate populations (Figures A-19b and A-19c). The data for controlled carbon material fall slightly above the combined-population best-fit stress-rupture curves (Figure A-19a).

The best-fit stress-rupture curves for all three sample populations are compared in Figure A-20. The normal-material curves fall very close to the combined-material curves. The curves for the controlled carbon material fall above the combined-material curves. However, considering the amount of scatter in all of the stress-rupture data, this difference is not too large. For engineering purposes, it is probably sufficient to use the combined-material stress-rupture curves.

a. Normal and Controlled Carbon Materials Combined

b. Normal Material

c. Controlled Carbon Material

FIGURE A-18. STRESS RUPTURE OF VARIOUS SAMPLE POPULATIONS OF SOLUTION-ANNEALED ALLOY 800 IN TERMS OF LARSON-MILLER PARAMETER

a. Normal and Controlled Carbon Materials Combined

b. Normal Material

c. Controlled Carbon Material

FIGURE A-19. STRESS-RUPTURE CURVES FOR VARIOUS SAMPLE POPULATIONS OF SOLUTION-ANNEALED ALLOY 800

FIGURE A-20. COMPARISON OF BEST-FIT STRESS-RUPTURE CURVES FOR SOLUTION-ANNEALED ALLOY 800 WITH NORMAL AND CONTROLLED CARBON CONTENTS

Creep-rupture tests of material from Group D of Incoloy Alloy 800 were conducted in accordance with ASTM Designation E139. Specimens were machined to the configuration shown in Figure A-21. Specimens were dead-weight loaded and heated using a shell-type furnace in conjunction with a controller. Temperature was measured with Chromel-Alumel thermocouples attached to the gage length of the specimen. Deformation over the 2.3-in. gage length was measured optically using a platinum-strip extensometer attached to the shoulders of the specimen. The extensometer consists of two platinum strips of metal, one of which slides within the folded edges of the other. The strips are polished to provide good reflecting surface, and a series of very fine marks is scribed on them. Two cross marks, one on each strip, are chosen as reference marks and the distance between them is measured periodically with a filar microscope. The change in the distance between these two reference marks is a measure of the change in length, or creep, of the specimen. This extensometer is capable of 0.00005-in. resolution.

Data from the creep-rupture tests are summarized in Table A-10. In this table are listed the temperature, stress, rupture time, elongation, reduction in area, and minimum creep rate for each of the specimens. All tests were run at 1400 F with the exception of one test (Specimen I-2) run at 1200 F for comparison. Figure A-22 is a plot of rupture time as a function of stress showing all 1400 F points. The stress range at 1400 F was 8,800 to 17,000 psi, which resulted in rupture lives ranging from 1306.3 to 11.6 hr. These stress-rupture data will be combined and analyzed with short-term elevated-temperature tensile tests now being conducted.

Plans for Future Work

Experimental work on Incoloy Alloy 800 and Type 304 stainless steel will be concluded during the first quarter of Fiscal Year 1972. To expand the understanding of cyclic stress-strain response developed already, several incremental step tests of Incoloy Alloy 800 and Type 304 stainless steel will be conducted at maximum total strain ranges of 1 percent with strain rates of 4×10^{-3} and 4×10^{-5} sec^{-1} . Also, several constant-amplitude low-cycle-fatigue tests will be conducted on these materials at room temperature. A two-level block-loading test with hold times (comparable to that of Specimen SS50 at 1200 F in Table A-8) will be completed on Specimen SS51 of Type 304 stainless steel at 1000 F. Creep-rupture tests of Incoloy Alloy 800 that are now in progress will be completed.

Sixty feet of 5/8-in. -diameter bar from Heat B65808 of Type 316 stainless steel has been received from WADCO. To check out the characterization of this material, hardness, grain size, and typical microstructure will be determined. A simple check of the chemical analysis will also be made. Before fatigue studies of the material are initiated, tensile properties will be determined at 70, 800, 900, 1050, and 1200 F at a strain rate of 4×10^{-3} sec^{-1} . Cyclic stress-strain behavior of this alloy will also be investigated at the same temperatures using incremental step tests.

TABLE A-10. SUMMARY OF CREEP-RUPTURE DATA FOR SOLUTION-ANNEALED INCOLOY
ALLOY 800 (HEAT HH3113A)

Specimen	Temperature, F	Stress, psi	Rupture Time, hr	Elongation, percent	Reduction of Area, percent	Minimum Creep Rate, percent per hr
I-2	1200	35,000	66.6	30.8	34.9	0.015
I-1	1400	13,000	106.7	79.0	66.0	0.33
I-3	1400	17,000	11.6	115.5	75.5	3.8
I-4	1400	10,000	1115.5	52.5	50.9	0.013
I-6	1400	11,500	147.2	63.9	74.2	0.25
I-7	1400	11,000	662.6	39.1	54.6	0.024
I-8	1400	8,800	1306.3	73.1	67.1	0.025
I-9	1400	15,000	23.1	61.0	76.2	1.64
I-10	1400	11,500	215.0	57.5	67.5	0.13

FIGURE A-21. DIMENSIONS OF CREEP-RUPTURE SPECIMEN

FIGURE A-22. STRESS-RUPTURE CURVE FOR INCOLOY ALLOY 800 AT 1400 F

The effect of strain rate and hold time upon the low-cycle-fatigue behavior of Type 316 stainless steel will be investigated at temperatures between 800 and 1200 F. Continuous cycling tests will be conducted at 800, 900, 1050, and 1200 F. Hold-time tests will be conducted at both 1050 and 1200 F. Finally, a series of tests with complex loading histories will be conducted at both 1050 and 1200 F. The objective of these tests will be to help develop cumulative damage relationships for predicting the life of Type 316 stainless steel under combined creep/fatigue conditions.

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STUDIES OF THE EFFECTS OF IRRADIATION ON CLADDING AND STRUCTURAL ALLOYS (AEC-DRDT)

This program (Task 14) has been carried out for the AEC Division of Reactor Development and Technology in support of the LMFBR Program. The influence of a LMFBR environment on reactor cladding and structural materials has been evaluated. A major problem in the application of cladding and structural materials in an irradiation environment is the embrittlement that results from this exposure. At low temperatures, atoms displaced in the crystal lattice as a result of collisions with fast neutrons produce individual defects and clusters of defects that are responsible for embrittlement. At elevated temperatures, bubbles formed from helium produced by (n, α) reactions with the constituents of stainless steel, and voids, formed by fast-neutron-produced vacancy condensation, are responsible for embrittlement. The extent and mechanism of embrittlement are dependent on flux spectrum as well as temperature.

The program was broken down into three efforts, two involving the irradiation behavior of AISI Type 347 and Type 316 stainless steel and the other the creep-rupture behavior of low-carbon ferritic 2-1/4 Cr-1 Mo steels. The results of the three efforts comprising Task 14 are detailed in the pages that follow. This concludes the successful operation of Task 14, and this report constitutes the Final Report for the task.

CORRELATION OF RADIATION EFFECTS ON MECHANICAL PROPERTIES AS CAUSED BY A PREDOMINANTLY FAST FLUX AND A MIXED FAST AND THERMAL FLUX

M. Kangilaski and A. A. Bauer

This effort, aimed at evaluating the effect of flux spectrum on the elevated-temperature embrittlement of stainless steels, grew out of an irradiation surveillance program of Type 347 stainless steel in the ETR. The high fast fluences (>1 Mev) reached by specimens in this program (3×10^{22} nvt) were in the range of fluences of interest for a LMFBR. Consequently, some of this same material was irradiated in the EBR-II. Because of the low temperatures of irradiation for the original specimens exposed in the ETR, additional irradiations were performed in the ETR at temperatures corresponding to those in the EBR-II irradiation. A correlation between the mechanisms of irradiation damage and the extent of embrittlement and change in mechanical properties has been attempted for specimens exposed to the fast-neutron spectrum of the EBR-II and those subjected to the more thermalized neutron spectrum of the ETR. The results of this effort are detailed in the two reports that follow, one dealing with the ETR irradiations and the other with the EBR-II irradiations.

Mechanical Properties of Potential Cladding
Materials Irradiated in the ETR

Introduction

This section summarizes irradiation and test results obtained with cladding and structural materials irradiated in the Engineering Testing Reactor (ETR). These irradiations can be divided into three broad categories.

- (1) Type 347 stainless steel specimens were irradiated at 50 C to fast fluences ($E > 1$ Mev) ranging from 5.5×10^{21} nvt to 3×10^{22} nvt. These irradiations originated in a surveillance program to monitor irradiation-induced mechanical property changes in pressure tubes that made up the ETR high-pressure loops. Later, the emphasis was shifted to studying the phenomenon of irradiation-induced elevated-temperature embrittlement.
- (2) Specimens were irradiated at 50 C to fast fluences of 1.5×10^{21} nvt to specifically study the phenomenon of elevated-temperature embrittlement. The materials which were irradiated and tested included: pure nickel (99.999 per cent), Nickel 200, Haynes Stellite 25, UMC0-50, and Tenelon.
- (3) Specimens of Type 347 and Type 348 stainless steel were irradiated at 400 and 650 C to study the effect of irradiation temperature on the irradiation-induced elevated-temperature embrittlement. The specimens were irradiated to fast fluences ranging from 3.3×10^{21} nvt to 4.6×10^{21} nvt.

A large number of high-pressure test loops located in the Engineering Test Reactor (ETR) are made of AISI Type 347 stainless steel. Since the removal of these loops is expensive and time consuming, it is desirable to operate the loops as long as possible. When this program was initiated (1958), the maximum safe operating life of these pressure tubes was not known. To determine this, it was decided to irradiate Type 347 stainless steel to fast fluences up to 30×10^{21} nvt (> 1 Mev)*. The specimens were to be tested at fast fluences of 5, 10, 15, 20, 25, and 30×10^{21} nvt to provide changes in mechanical properties as a function of fast fluence. The mechanical property tests included tensile, creep, fatigue, and impact tests. An objective of the program was to progressively test irradiated specimens prior to the accumulation of similar fast fluences by the pressure tubes so that the changes in mechanical properties of the pressure tubes could be anticipated. Specimens were to be tested at room temperature and at the operating temperature of the pressure tubes, 316 C. Since removal of the irradiated pressure tubes is expensive and time consuming, serious consideration was given to the possibility of in-place annealing of pressure tubes to remove or alleviate detrimental irradiation-induced changes in mechanical properties, and thus to prolong

* Throughout the report the fast fluence numbers refer to neutrons with energies greater than 1 Mev.

the useful life of the pressure tubes. To measure the effect of annealing, irradiated specimens at each fast fluence level were to be annealed for 1 hr at 980 C before tensile testing at room temperature and 316 C.

Only the highlights of the early irradiation and test results will be given where the emphasis was on mechanical properties at room temperature and 316 C. Results of these early tests were given in previous reports^(1,2).

A review of literature concerning the phenomenon of irradiation-induced embrittlement at elevated temperatures indicated that nickel-base alloys were embrittled to a greater degree than austenitic stainless steels for equivalent irradiation conditions⁽³⁻⁷⁾. It was also apparent that the degree of embrittlement increased with increasing nickel content, while the ductility of the unirradiated material was independent of nickel content. These trends indicated that nickel was responsible for the elevated-temperature embrittlement. This could result if a deleterious transmutation product originated from the nickel or if nickel was sensitive to the presence of some transmutation product. To establish whether the presence of nickel was necessary for elevated-temperature embrittlement, various alloys were irradiated. All of the alloys chosen for this study had a face-centered-cubic crystal structure. The alloys chosen included: (1) two cobalt alloys, one of which contained no nickel (UMCo-50) and Haynes Stellite 25 which contained 10 percent nickel, (2) a manganese-stabilized austenitic stainless steel (Tenelon) containing no nickel, (3) Nickel 200, which is 99 percent pure, and (4) high-purity nickel (99.999 percent). It was theorized that if the presence of nickel was necessary for embrittlement, Haynes Stellite 25, Nickel 200, and pure nickel would be embrittled by irradiation while Tenelon and UMCo-50 would not.

With the emphasis of the cladding program being shifted toward LMFBR applications, because of the higher operating temperatures (400 to 700 C) for LMFBR's, it became paramount to establish the effect of irradiation temperature on properties. Since at the time of change in emphasis almost all data on irradiation-induced property changes in stainless steel were obtained by irradiations in thermal reactors, it was also desirable to compare the irradiation-induced property changes of stainless steel irradiated in a fast reactor (predominantly fast neutrons) as opposed to a thermal reactor (mixture of thermal and fast neutrons) after exposure to large fast fluences. To achieve these objectives, specimens of Type 347 stainless steel, identical to those which had been irradiated in the ETR at 50 C to fast fluences of up to 3×10^{22} nvt, were irradiated in both the ETR and the EBR-II at 400 and 650 C. Since only a limited number of the original Type 347 stainless steel specimens were available, additional specimens of Type 348 stainless steel were included in the EBR-II irradiation experiment. The Type 348 stainless steel rod, from which specimens were machined, was obtained from the stockpile of structural materials kept at Battelle-Northwest for studies of radiation effects on structural materials. The Type 347 stainless steel can be considered as a Type 348 stainless steel since it meets the chemical specifications for Type 348 stainless steel (less than 0.10 w/o tantalum). From these irradiations it was hoped to obtain correlations of tensile property changes for different neutron spectrums and different irradiation temperatures. The tensile test results of the EBR-II irradiations and their comparisons to ETR irradiations are reported in the next section.

Experimental Procedures

Material Characterization. Two different types of tensile specimens were utilized. One of the specimen types had a gage length of 1 in. while the other had a gage length of 2 in. Dimensions of the tensile specimens are shown in Figure B-1. Compositions of the materials from which tensile specimens were machined are given in Table B-1. All of the specimens were in the as-annealed condition, their microstructures are shown in Figures B-2 through B-5.

In the low-temperature irradiations of Type 347 stainless steel only the short-type tensile specimens were used. For the low-temperature irradiations of UMo-50, Haynes Stellite 25, Tenelon, and Nickel 200 only the long tensile specimens were used. For the elevated-temperature irradiations, the Type 347 stainless steel tensile specimens were of the short type while the Type 348 stainless steel tensile specimens were of the long type.

Drawings of specimens for fatigue and impact tests are shown in Figure B-6. Only Type 347 stainless steel specimens were prepared for this purpose for low-temperature irradiation exposure.

Capsule Design. The low-temperature irradiation capsules were of three types. One type of capsule was designed to contain six Charpy V-notch bars and 12 subsize Izod specimens. Another type contained subsize tensile specimens and cyclic-strain fatigue specimens, while the third type of low-temperature capsule was designed to contain tensile specimens for the study of embrittlement.

In each capsule containing the subsize Izod specimens and Charpy V-notch bars, the specimens were arranged in three tiers spaced over the 8-in. capsule length. Each tier contained two Charpy and four Izod specimens held in place by stainless steel clip holders.

In each capsule containing the subsize tensile specimens and cyclic-strain fatigue specimens, the specimens were arranged in tiers, with each tier containing two subsize tensile specimens and two cyclic-strain fatigue specimens. Figure B-7 is a view of a typical capsule showing the arrangement of tensile and cyclic-strain fatigue specimens. All of the specimens were encapsulated in open-end capsules of 1100 aluminum.

The capsules were open at each end to allow reactor cooling water to flow past all test specimens in order to remove the gamma heat produced in these specimens during irradiation. The reactor cooling water remained at approximately 50 C during full-power reactor operation. Considering the large flow of water past the specimens, it is assumed that the specimens were maintained at about that temperature during irradiation. No special provisions were made to instrument the capsules or monitor the temperature during irradiation. The capsules were positioned in X-baskets fitted into a specially designed jig, cut from a single 3 by 3-in. block of 3003 aluminum. This core-filler piece extended the length of the active core and accommodated the capsules in four quadrants.

a. Short Specimen

b. Long Specimen

FIGURE B-1. DESIGN OF TENSILE SPECIMENS THAT WERE IRRADIATED AND TESTED

TABLE B-1. ANALYSIS OF VARIOUS MATERIALS USED FOR IRRADIATION STUDIES(a)

Element	Composition of Indicated Materials, w/o					
	Type 347 Stainless	Type 348 Stainless	Tenelon	Nickel 200	UMCo-50	Haynes Stellite 25
Iron	Bal	Bal	Bal	0.1	23.9	2.0
Chromium	17.91	17.5	18.6	NA	26.6	19.9
Nickel	10.41	9.67	0.32	Bal	0.30	9.86
Manganese	1.72	1.67	15.1	0.5	0.64	1.36
Niobium	0.67	0.58	NA	NA	NA	NA
Silicon	0.65	0.51	0.4	0.2	0.85	0.47
Copper	0.28	0.06	NA	NA	NA	NA
Molybdenum	0.20(b)	NA	NA	NA	NA	NA
Titanium	0.10	NA	NA	NA	NA	NA
Cobalt	0.10	0.18	0.09	0.3	Bal	Bal
Carbon	0.054	0.04	0.10	0.08	0.07	0.08
Tantalum	0.05	0.03	NA	NA	NA	NA
Tungsten	0.04	NA	NA	NA	NA	14.7
Tin	0.04	NA	NA	NA	NA	NA
Zinc	0.03	NA	NA	NA	NA	NA
Vanadium	0.03	NA	NA	NA	NA	NA
Lead	0.03	NA	NA	NA	NA	NA
Nitrogen	0.029	0.037	0.46	0.052	0.001	0.001
Phosphorus	0.024	0.014	0.025	0.003	0.003	0.013
Sulfur	0.016	0.009	0.003	0.004	0.016	0.008
Gallium	0.006	NA	NA	NA	NA	NA
Arsenic	0.006	NA	NA	NA	NA	NA
Antimony	0.005	NA	NA	NA	NA	NA
Boron	0.00006	0.0008	0.0002	0.0020	0.0001	0.0002

(a) Chemical analysis unless otherwise specified. NA = not analyzed.

(b) Emission spectrographic analysis.

250X

5A396

a. Type 347 Stainless Steel (12- μ Grain Size)

250X

OC585

b. Type 348 Stainless Steel (10- μ Grain Size)

FIGURE B-2. MICROSTRUCTURE OF STAINLESS STEELS

100X

1B200

a. Haynes Stellite 25 (130- μ Grain Size)

100X

1B193

b. UMo-50 (60- μ Grain Size)

FIGURE B-3. MICROSTRUCTURE OF COBALT ALLOYS

2

50X

7B331

a. Nickel (99.999 Percent Pure; 25 to 600- μ Grain Size)

250X

7B339

b. Nickel 200 (15- μ Grain Size)

FIGURE B-4. MICROSTRUCTURE OF NICKEL SPECIMENS

A slot 0.375 in. in diameter was cut in each of the four faces and in the center of the aluminum core-filler piece for the insertion of dosimeter wires which monitored the fast flux during irradiation. The dosimeter wires were inserted and exposed for periods compatible with the type of dosimeter wire being used. These wires were removed periodically, and the fast and thermal fluences were determined for these positions.

FIGURE B-5. MICROSTRUCTURE OF TENELON
(50- μ GRAIN SIZE)

The prolonged irradiation caused the aluminum capsule bodies to deteriorate. Five of the capsules were returned to the Battelle-Columbus Hot-Cell Facility, reencapsulated in stainless steel capsules of virtually the same design but without the sleeve, and returned to the ETR for irradiation. A reencapsulated assembly is shown in Figure B-8. Examination of the capsule bodies indicated that the failures were caused by a combination of corrosion and erosion of the embrittled 1100 aluminum.

The design of the low-temperature capsules for the study of embrittlement is shown in Figure B-9. Specimens of Tenelon, UMo-50, Haynes Stellite 25, Nickel 200, and pure nickel were irradiated in these capsules. Each of the capsules contained two tiers of nine tensile specimens arranged as shown. Specimen holders, fabricated from Type 304 stainless steel, kept the specimens aligned and allowed each to be cooled by the reactor process water. The assembly was held together with three 0.170-in.-diameter stainless steel rods that were welded to the top and bottom specimen holders. In addition, a lifting lug was welded to the top specimen holder to facilitate handling. The diameter of the specimen holder was 1.125 in., and the standard ETR ribbed X-basket was used.

a. Charpy V-Notch Impact Specimen

b. Cyclic-Strain Fatigue Specimen

FIGURE B-6. DRAWINGS OF SPECIMENS USED FOR FATIGUE AND IMPACT TESTS

N59677

FIGURE B-7. IRRADIATION-CAPSULE COMPONENTS AND TEST SPECIMENS BEFORE IRRADIATION

2632

FIGURE B-8. REENCAPSULATED STAINLESS STEEL
IRRADIATION ASSEMBLY

35963

FIGURE B-9. ASSEMBLED LOW-TEMPERATURE IRRADIATION CAPSULE FOR THE STUDY OF EMBRITTLEMENT

Fifty-four specimens were placed in an elevated-temperature capsule, which was then irradiated in the ETR. Neutrons and gamma heating were utilized to raise the specimen temperatures. The design temperature was maintained by capsule design to adjust the heat transfer from the specimens. To assure uniformity of specimen temperatures, the specimens were surrounded by NaK, which is a molten eutectic alloy with high thermal conductivity. A double-walled, single-gas-annulus capsule was used. Figure B-10 shows a photograph of the capsule components, while Figure B-11 provides a schematic drawing of the capsule design. Six specimens were placed in each tier and the capsule contained nine tiers. Each tier was strapped around a 0.25-in.-diameter center rod of Type 304 stainless steel with a 0.020-in.-diameter wire of Type 304 stainless steel placed at the grip ends of the specimens. The specimens were held in the desired vertical position by "spiders", Heliarc welded to the bottom and top of the 0.25-in.-diameter rod. Also positioned in the assembly with the 0.020-in.-diameter wires were six thermocouple protection tubes which extended from the spider at the base up into the capsule header. The specimen assembly was suspended from the heater with six 0.062-in.-diameter rods which were Heliarc welded into the top spider and capsule header.

The 0.062-in.-diameter thermocouple protection tubes were brazed into the first bulkhead seal in the capsule header. This whole assembly, specimen assembly and header, was fitted into the inner shell. The header was brazed to the inner shell. A 0.093-in.-diameter hole was drilled in the Type 316 stainless steel bottom end plug so that the NaK could be loaded, and the end plug was Heliarc welded to the inner shell. A total of 97 cm³ of NaK was loaded into the inner shell. The hole in the bottom end cap was welded under a pressure of 4 psia after the NaK was loaded.

Fifteen 0.040-in.-OD stainless steel-sheathed, Chromel-Alumel thermocouples were used for monitoring the specimen temperatures. One of each of six of these thermocouples was placed opposite the midplane of Tiers 1, 3, 4, 5, 6, and 8 in the thermocouple protection tubes inside the inner shell. One of each of the remaining nine thermocouples was placed opposite the midplane of each tier of specimens in grooves milled on the outside of the inner shell.

The gas annulus between the inner and outer shells at operating conditions ranged from 0.003 to 0.0012 in. This variation in gas annulus size was required by the variation in heating rate along the vertical length of the capsule in order to achieve the desired temperature ranges for the specimens. The outer shell was a 1.125-in.-OD tube with a 0.065-in. wall thickness. The outer shell was brazed to the header and the end cap was Heliarc welded to the base of the shell. All welds and brazes were helium leak checked and also radiographed.

Desired specimen temperatures were obtained by regulating the heat removed from the specimens. Figure B-12 illustrates the typical temperature profiles through the capsule. A mixture of helium and nitrogen gas was continuously passed through the gap between the inner and outer stainless steel shells. When the relative helium and nitrogen contents of the gas were varied, the conductivity across the gap could be varied because of the large difference in thermal conductivity

40744

FIGURE B-10. CAPSULE ASSEMBLY FOR ELEVATED-TEMPERATURE IRRADIATIONS

FIGURE B-11. SCHEMATIC DRAWING OF THE ELEVATED-TEMPERATURE IRRADIATION CAPSULE

FIGURE B-12. TEMPERATURE PROFILE OF THE CAPSULE DURING IRRADIATION

between helium and nitrogen. Temperature of the specimens was calculated from the known heat generation rate and the temperature gradient between the internal and external thermocouples. Temperature readings were taken from the thermocouples twice a day when the reactor was in operation. Table B-2 gives the range of temperatures of the specimens during irradiation, while Table B-3 provides the location of individual specimens.

TABLE B-2. TEMPERATURE HISTORY FOR SPECIMENS IRRADIATED IN ETR ELEVATED-TEMPERATURE CAPSULE

ETR Cycle	Number of Irradiation Days	Average Temperature for Specimens in Indicated Tiers (Top to Bottom), C								
		1	2	3	4	5	6	7	8	9
96	33.60	425	570	675	650	690	675	605	430	420
97	30.35	400	535	645	615	660	650	580	430	415
98	29.50	405	545	650	625	665	665	580	425	410
99	32.45	420	565	655	615	670	670	590	425	415
100	33.00	385	525	630	595	645	610	565	420	390
101	33.80	390	530	615	580	630	625	550	400	385
102	28.80	385	530	620	590	640	625	560	400	390

TABLE B-3. IDENTIFICATION AND LOCATION OF SPECIMENS IRRADIATED IN ETR ELEVATED-TEMPERATURE CAPSULE

Tier Identification	Type of Specimen	Stainless Steel Material	Specimen Identification
1	Tensile	Type 348	19, 20, 21, 22, 23, 24
2	Tubular	Type 348	43, 44, 45, 46, 47, 48
3	Tensile	Type 347	1, 2, 3, 16, 17, 18
4	Tensile	Type 348	25, 26, 27, 28, 29, 30
5	Tensile	Type 348	31, 32, 33, 34, 35, 36
6	Tensile	Type 347	4, 5, 6, 7, 8, 9
7	Tubular	Type 348	49, 50, 51, 52, 53, 54
8	Tensile	Type 347	10, 11, 12, 13, 14, 15
9	Fatigue	Type 347	37, 38, 39, 40, 41, 42

Irradiation and Dosimetry. The irradiation of the Type 347 stainless steel specimens at low temperature was started in Position F-10 of the ETR at the start of Cycle 4. All were arranged symmetrically around the vertical midposition of the core-filler piece and located around the horizontal midplane of the reactor so that the maximum number of specimens were located in the regions of highest flux. These capsules were subsequently moved to different locations as the irradiation progressed.

The method of monitoring fast fluence was changed several times during the 10-year course of the program as better methods of measurement became available. The reactions used for dosimetry and the cross sections of the reactions are given in Table B-4. At first, the fast-neutron measurements were based on the $\text{Ni}^{58}(\text{n}, \text{p})\text{Co}^{58}$ reaction with corrections being made for Co^{58} burnout by the $(\text{n}, \gamma)\text{Co}^{59}$ reaction. Since these corrections involved numerous calculations, and at the time there was some uncertainty as to Co^{58} burnout, it seemed advisable to find a more convenient method. Consequently, the $\text{Fe}^{54}(\text{n}, \text{p})\text{Mn}^{54}$ reaction was chosen because of the low Mn^{54} capture cross section, the long Mn^{54} half-life of about 310 days, and the adequate Fe^{54} cross section. However, to determine the amount of Mn^{54} present, it was necessary to separate the manganese chemically before it could be counted by gamma spectrometry. To eliminate chemical analysis, it was decided to use the $\text{Ti}^{46}(\text{n}, \text{p})\text{Sc}^{46}$ reaction as a measure of fast fluence in order to obtain the dosimetry analysis more quickly and at less cost. Since the cross section of Co^{58} became well known and automation could be used in making the numerous calculations, it was later decided to switch back to using $\text{Ni}^{58}(\text{n}_\text{f}, \text{p})\text{Co}^{58}$ as the fast-flux monitor.

TABLE B-4. NUCLEAR REACTIONS WHICH WERE USED IN DOSIMETRY ANALYSES FOR FAST-FLUX DETERMINATIONS

Reaction(a)	Cross Section, millibarns	Half-Life of Nuclide Formed
$\text{Ni}^{58}(\text{n}_\text{f}, \text{p})\text{Co}^{58}$	95	71.3 days
$\text{Fe}^{54}(\text{n}_\text{f}, \text{p})\text{Mn}^{54}$	68	310 days
$\text{Ti}^{46}(\text{n}_\text{f}, \text{p})\text{Sc}^{46}$	9.5	84 days
$\text{Co}^{58}(\text{n}_\text{t}, \gamma)\text{Co}^{59}$	1.65×10^6	Stable
$\text{Co}^{59}(\text{n}_\text{t}, \gamma)\text{Co}^{60}$	3.7×10^4	5.27 yr
$\text{Co}^{59}(\text{n}_\text{et}, \gamma)\text{Co}^{60}$	7.0×10^4	5.27 yr

(a) n_f = fast neutron
 n_t = thermal neutron
 n_et = epithermal neutron.

The flux wires were placed in the corners and the center of the reactor position. These wires were removed after each cycle and small sections were cut out at 3 to 6-in. intervals. These small sections were analyzed and a flux profile for each location was determined. From this flux profile the neutron fluence received by each individual specimen during the specific cycle was determined. These fluence values were continuously added to obtain total fluence values for the life of the irradiation. The fluence values for the different capsules are enumerated in Table B-5.

TABLE B-5. IRRADIATION HISTORY OF ETR CAPSULES

Identification Capsule	Material	Fast Fluence ^(a) , 10 ²¹ nvt		ETR Cycle Number	
		Min- mum	Maxi- num	Inserted	Removed
24-2	Type 347 stainless	30.1	31.4	4	97
24-4	Type 347 stainless	20.5	24.6	4	80
24-6	Type 347 stainless	5.5	7.4	4	42
24-8	Type 347 stainless	16.0	16.0	4	62
24-10	Type 347 stainless	26.4	28.1	4	97
24-12	Type 347 stainless	21.3	21.3	4	75
24-14	Type 347 stainless	5.6	5.8	4	28
24-18	Type 347 stainless	9.3	11.0	18	42
24-20	Type 347 stainless	21.3	24.6	18	91
24-23	Tenelon, nickel	1.4	1.4	88	91
24-24	UMCo-50, Haynes Stellite 25	1.5	1.6	89	92
24-27	Types 347 and 348 stainless	3.3	4.6	96	102

(a) Determined on the basis of the $\text{Ni}^{58}(\text{n}, \text{p})\text{Co}^{58}$ reaction during ETR Cycles 4 through 37; the $\text{Ni}^{58}(\text{n}, \text{p})\text{Co}^{58}$ and $\text{Fe}^{54}(\text{n}, \text{p})\text{Mn}^{54}$ reactions during Cycles 38 through 61; the $\text{Fe}^{54}(\text{n}, \text{p})\text{Mn}^{54}$, $\text{Ti}^{46}(\text{n}, \text{p})\text{Sc}^{46}$, and $\text{Ni}^{58}(\text{n}, \text{p})\text{Co}^{58}$ reactions during Cycles 62 through 70; the $\text{Ti}^{46}(\text{n}, \text{p})\text{Sc}^{46}$ reaction from Cycles 70 through 83; and the $\text{Ni}^{58}(\text{n}, \text{p})\text{Co}^{58}$ reaction during Cycles 84 through 102.

Mechanical Testing. The impact, tensile, and fatigue specimens were removed from their irradiation capsules, and inspected and measured in the Battelle-Columbus Hot-Cell Facility. The impact tests were made on a Riehle impact machine in accordance with ASTM Designation E23. The machine had been calibrated against standardized specimens from the U. S. Army Materials Research Agency.

Initially, tensile tests were made with a hydraulically loaded Riehle tensile machine. During later stages of the study, specimens were tested with an Instron tensile tester. Tensile testing was generally performed by using a strain rate of 0.005 per min except in tests where the strain rate was purposely varied. Specimens which were tested at elevated temperatures were held at temperature for 30 min prior to testing. The elongation values were taken from curves of load versus time on the assumption that, once plastic yielding started, all crosshead travel was accommodated in the gage section. The uniform elongation was taken

to be the strain when the load necessary for further deformation started to decrease. Values for reduction in area were obtained from measurements taken on photographs of fractured ends of tensile specimens. Those tensile specimens given a postirradiation heat treatment were held for 1 hr at their annealing temperature in either vacuum or argon.

The strain-cycle fatigue tests were made on equipment similar to that used by Baldwin, Sokol, and Coffin(8) and Coffin and Tavernelli(9) for their fatigue studies of several unirradiated materials. The strain-cycle fatigue test differs from the stress-cycle fatigue test in that for the former, the strain amplitude is maintained constant throughout the test, while for the latter, the stress amplitude is maintained constant. The stress-cycle test establishes the endurance limit, which is the largest cyclic stress that can be applied for an infinite number of times without producing failure. The strain-cycle fatigue test gives the number of cycles to failure for a given constant strain amplitude.

The fatigue equipment transmits a cyclic (equal tension and compression) longitudinal strain to the test specimen. This is accomplished by oscillating the lower specimen grip and weigh bar through a constant vertical amplitude. This motion is transmitted by the specimen to the upper specimen grip, which is an adjustable sliding head. The upper and lower specimen grips and attached specimen are actually a long piston, the stroke of which can be adjusted by the threaded mechanical stops. A scotch yoke, which consists of an eccentric cam shaft, follower, and main block, converts a rotary motion into a linear, vertical oscillatory motion of fixed amplitude which drives the weigh bar. The weigh bar is a machined steel bar with attached SR-4 resistance-type strain gages. These gages are calibrated over the elastic-strain range of the steel bar, providing a gage output of load versus strain. The calibration curve is used to determine the load and corresponding stress on the specimen during testing. A spring-loaded clip-on type of extensometer, with attached SR-4 strain gages, is used to measure specimen strain. The output signals from the strain gages on the weigh bar and extensometer are fed into individual channels of a Sanborn recorder, thus providing a permanent record of the total strain and corresponding load on a common time axis.

The test procedure followed was to load the specimen in the specimen grips, secure the grips and specimen in the load train, mount the extensometer on the specimen, adjust stops to limit the load-train travel to produce the desired total strain, and cycle at a rate of about 660 cycles per hr. A series of unirradiated specimens was tested at selected total strain ranges from 0.5 to about 2.0 percent. The base curve established for unirradiated materials enables a similar curve to be characterized with fewer irradiated specimens. This was quite desirable since there were only six specimens available from each of the fast-fluence levels of 5.5×10^{21} , 11×10^{21} , and 16×10^{21} nvt. A continuous record of the total strain per cycle and corresponding load per cycle was kept for each test, from which the following information could be obtained:

- (1) Total number of cycles to failure
- (2) Total number of cycles to crack initiation

- (3) Total strain per cycle (elastic plus plastic strain in tension and compression)
- (4) Total plastic strain (tension plus compression)
- (5) Total load and stress range.

An Instron testing machine was used for the elevated-temperature fatigue tests. In these test the specimens were never in compression since the stress was varied from a minimum of 2000 psi to the maximum stress in the given test. During testing the curve of load versus crosshead travel was traced out. Tests were conducted in flowing helium with the crosshead travel being 0.05 in./min. This resulted in a frequency of about 50 cycles per hr.

Experimental Results

Impact Tests. Results from tests on both the subsize Izod specimens and Charpy V-notch specimens of Type 347 stainless steel irradiated to a fast fluence of 5.7×10^{21} nvt were reported previously⁽¹⁾. Results of the Charpy V-notch tests are given in Figure B-13. It can be seen that the impact properties of the irradiated material at 316 C are about the same as they were at room temperature. Figure B-14 shows that the appearance of the fracture is 100 percent fibrous at both test temperatures. This is characteristic of a ductile failure. The most pronounced effects of irradiation are the predominantly brittle behavior at -196 C (liquid-nitrogen temperature) and the reduced values of absorbed energy obtained at all test temperatures. A somewhat less pronounced effect is the smaller lateral contraction values noted for the irradiated material when compared with those for the unirradiated material, indicating that the irradiated material is less ductile.

Tests were performed on unirradiated and irradiated (5.7×10^{21} nvt) Izod specimens at room temperature and liquid-nitrogen temperature (-196 C). Results of these tests are given in Table B-6. Neither the unirradiated nor the irradiated specimens broke during testing; however, irradiation tends to increase the energy absorbed during testing. It was observed that tears were produced in the notches of the irradiated specimens while not in the unirradiated specimens.

TABLE B-6. IMPACT PROPERTIES OF UNIRRADIATED AND IRRADIATED TYPE 347 STAINLESS STEEL

Testing Temperature, C	Impact Energy, ft-lb	
	Unirradiated	Irradiated
-196	12.9(a)	16.0(c)
20	10.1(b)	12.9(c)

(a) Average of five tests.

(b) Average of four tests.

(c) Average of three tests.

FIGURE B-13. THE EFFECT OF IRRADIATION ON THE IMPACT PROPERTIES OF TYPE 347 STAINLESS STEEL

The irradiation-induced decrease in fracture energy as measured in Charpy V-notch tests and the increase in energy absorbed as measured in tests of Izod specimens may at first appear contradictory. However, the fracture energy is approximated by the area under the stress-strain curve. In the case of the Charpy V-notch tests, because the yield strength is increased while the strain at fracture is decreased as a result of irradiation, the energy at fracture is decreased. For the Izod specimens, however, where no fractures occurred, the increase in yield strength leads to an increase in the energy absorbed during testing. The relative energy areas for Charpy and Izod specimens are schematically compared in Figure B-15.

Tensile Tests on Stainless Steel Irradiated at 50 C. A large number of Type 347 stainless steel specimens were tested in tension during this program. At first only the room-temperature and 316 C tensile properties of the irradiated material were of interest. However, later the main effort of the study was shifted to studying the tensile properties at 500 C and above. Results of the tensile tests on these specimens are tabulated in Table B-7. These test results are discussed in terms of a number of variables, detailed below.

TABLE B-7. TENSILE PROPERTIES OF TYPE 347 STAINLESS STEEL
BEFORE AND AFTER IRRADIATION AT 50 C

Test Temperature, C	Fast Fluence, 10 ²¹ nvt	0.2 Percent		Uniform Elonga- tion, percent	Total Elonga- tion, percent	Reduction in Area, percent
		Offset Yield Strength, 1000 psi	Ultimate Strength, 1000 psi			
RT	0	37.0	90.0	52.0	64.0	75
RT	5.5	88.7	111.5	22.1	32.7	70
RT	5.5	104	112	22.7	34.5	68.5
RT	11.0	103.5	112	24.1	30.9	70
RT	11.0	109	113	24.0	30.9	69
RT	16.0	112	112	26.0	33.8	79
RT	16.00	114	114	25.0	33.2	69.8
RT	22.0	113	113	24.3	34.5	67.2
RT	30.5	104	115	32.7	45.6	--
315	0	26.8	64.2	31.9	36.0	--
315	5.5	80	84	8.5	16.4	65
315	5.5	81.8	83	8.2	14.5	65
315	11.0	86.8	89	7.8	13.2	65
315	11.0	85.3	87.5	7.3	11.9	65
315	16.0	86.2	86.2	8.0	13.7	58.6
315	16.0	83.6	84.3	8.0	14.0	60.2
315	22	86	86	5.0	10.2	56.8
315	30.5	78.7	83.4	9.0	15.7	--
500	0	24.0	47.5	22.4	26.6	--
525	22	51.5	65.6	16.0	21.2	--
550	22	46.0	58.0	7.3	8.8	--
575	22	44.2	50.5	1.9	2.0	--
600	0	23.0	40.5	17.8	22.5	--
600	22	34.6	38.4	0.7	0.7	--
650	22	36.6	39.0	0.8	0.9	--
750	0	22.0	33.0	16.0	26.0	--
750	22	35.0	35.0	0.5	0.5	--
750	30.5	(a)	28.7	0.3	0.3	--

(a) Specimen broke before 0.2 percent offset yield strength was achieved.

~3.5X Unirradiated N91402

~4X Irradiated HC5460

a. Tested at -196 C

~3.5X Unirradiated N91401

~4X Irradiated HC5359

b. Tested at 20 C

4X Unirradiated 2A157

~4X Irradiated HC14736

c. Tested at 316 C

FIGURE B-14. FRACTURE SURFACES OF UNIRRADIATED AND IRRADIATED CHARPY V-NOTCH SPECIMENS TESTED AT -196 C, 20 C, AND 316 C

Irradiated specimens received a fast fluence of 5.7×10^{21} nvt.

FIGURE B-15. EFFECT OF IRRADIATION ON ENERGY ABSORPTION FOR IZOD AND CHARPY SPECIMENS

(1) Fast Fluence. Irradiation was found to increase both the yield and ultimate strengths and decrease the elongation. Figure B-16 shows the increases in room-temperature 0.2 percent offset yield strength and ultimate strength. The yield strength is observed to approach the ultimate tensile strength in value. Also, both the uniform and total elongations are decreased by irradiation (Figure B-17). The increase in strength appears to saturate after a fast fluence of about 1.6×10^{22} nvt while the ductility reaches saturation after a fast fluence of 5.5×10^{21} nvt. However, after a fast fluence of 3.1×10^{22} nvt it appears that the irradiation-induced increase in yield strength had decreased somewhat compared to the change due to a fast fluence of 2.2×10^{22} nvt. Similarly, the ductility, after a fast fluence of 3.1×10^{22} nvt, is somewhat higher than after a fast fluence of 2.2×10^{22} nvt. This irradiation-induced recovery in tensile properties appears to apply to the tensile properties at both room temperature and 316 C.

(2) Testing Temperature. Figure B-18 compares the strength of irradiated (2.1×10^{22} nvt) and unirradiated stainless steel as a function of testing temperature. At low testing temperatures irradiation increases both the yield and ultimate strengths, with the yield and ultimate strengths becoming identical. This strength increase is attributed to a displacement type of radiation damage which is annealed out at higher temperatures. It has been found that 1 hr at 500 C results in removal of irradiation-induced strength increases at room temperature⁽¹⁰⁾. Consequently, it would be expected that some of the displacement-type irradiation damage would be annealed out during the 30-min hold time at testing temperature before testing at these higher temperatures. At test temperatures above about 300 C the yield strength of the irradiated stainless steel begins to drop below its ultimate strength. The ultimate tensile strength of the irradiated stainless steel actually becomes identical with that of the unirradiated stainless steel at about 600 C. However, some of the irradiation-induced increase in yield strength persists up to 750 C.

Figure B-19 compares the elongation of the unirradiated and irradiated stainless steel as a function of testing temperature. At the lower testing temperatures the displacement type of radiation damage causes reductions in both uniform and necking elongation, with the uniform elongation being reduced to a greater degree. As the testing temperature is increased the displacement type of radiation damage is annealed out and at about 500 C the ductility of the irradiated specimens approaches that of the unirradiated specimens. However, above 500 C the ductility is drastically reduced. This reduction in ductility does not recover at higher testing temperatures, with the total elongation being only 0.5 percent at 750 C. At temperatures of 575 C and above, little necking takes place in the specimen and consequently uniform and total elongation become identical.

(3) Postirradiation Annealing. Selected irradiated specimens were annealed for 1 hr at 980 C before being tested in tension. The 980 C annealing temperature was chosen because considerable amounts of data were available for this annealing temperature and the temperature is sufficiently high to remove all displacement-type radiation damage. Table B-8 enumerates the tensile properties of the irradiated and annealed Type 347 stainless steel specimens for comparison with the data in Table B-7. These results show that annealing restores the preirradiation

FIGURE B-16. INFLUENCE OF FAST FLUENCE ON STRENGTH OF TYPE 347 STAINLESS STEEL

FIGURE B-17. INFLUENCE OF FAST FLUENCE ON DUCTILITY OF TYPE 347 STAINLESS STEEL

FIGURE B-18. STRENGTH OF UNIRRADIATED AND IRRADIATED (2.1×10^{22} nvt) TYPE 347 STAINLESS STEEL AT DIFFERENT TESTING TEMPERATURES

FIGURE B-19. ELONGATION OF UNIRRADIATED AND IRRADIATED TYPE 347 STAINLESS STEEL AT DIFFERENT TESTING TEMPERATURES

TABLE B-8. TENSILE PROPERTIES OF IRRADIATED TYPE 347 STAINLESS STEEL SPECIMENS ANNEALED FOR 1 HR AT 980 C

Test Temperature, C	Fast Fluence 10^{21} nvt	0.2 Percent Offset Yield Strength, 1000 psi	Ultimate Strength, 1000 psi	Uniform Elongation, percent	Total Elongation, percent	Reduction in Area, percent
RT	5.5	33.0	86.6	--	55.5	75.0
RT	11.0	33.8	87.0	--	61.0	77.0
RT	16	32.3	84.8	50.9	56.6	66.3
315	5.5	28.2	64.3	--	30.0	77.0
315	11.0	27.4	65.0	--	31.7	75.0
315	16.0	20.2	58.2	27.8	32.6	55.5
315	22	33.0	61.0	26.8	32.6	--
500	22.0	28.0	54.0	20.0	24.2	--
560	22.0	23.2	38.0	8.6	9.6	--
600	22.0	22.3	33.1	5.2	5.8	--
750	30.0	14.5	16.6	0.7	0.8	--

TABLE B-9. EFFECT OF 1-HR ANNEAL AT 1350 C ON TENSILE PROPERTIES OF TYPE 347 STAINLESS STEEL AT 750 C

Fast Fluence 10^{22} nvt	0.2 Percent Offset Yield Strength, 1000 psi	Ultimate Strength, 1000 psi	Elongation, percent	
			Uniform	Total
0	22.0	33.0	16	26
2.2(a)	35.0	35.0	0.5	0.5
2.2(b)	19.0	20.2	0.5	0.5

(a) As irradiated.

(b) Annealed.

ductility and strength for testing temperatures up to 500 C. At temperatures above 500 C the elongation is only slightly improved. The yield strength is restored to the unirradiated level but the ultimate strength is somewhat reduced.

One of the irradiated (2.2×10^{22} nvt) specimens was annealed for 1 hr at 1350 C, and then tested in tension at 750 C. Table B-9 illustrates that the high-temperature anneal reduced the strength below that of the unirradiated material at 750 C. The ductility of the irradiated specimen was not improved by the anneal. Therefore, it is apparent that the irradiation-induced elevated-temperature embrittlement cannot be removed by annealing even at temperatures close to the melting point of the material.

(4) Influence of Holding Time. The embrittlement phenomenon in irradiated stainless steel is generally believed to be caused by grain-boundary precipitation of some type. Consequently, it is necessary for the embrittling species to reach the grain boundary to cause embrittlement. If helium is responsible for the extreme embrittlement, then it must migrate to the grain boundaries to cause the embrittlement. Similarly, any solid transmutation product which is produced homogeneously in the stainless steel also would have to diffuse to the grain boundaries before it could cause embrittlement.

To establish whether diffusion plays a role in the embrittlement phenomenon, specimens were held at test temperature for various times before being tested. Rapid heating times were obtained by using a 4000-watt radiant heating furnace and preheating the grips before the specimens were loaded. It was established that by appropriate preheating of the grips it was possible to bring the test specimen to thermal equilibrium in about 2 to 4 min. Results of tensile tests on rapidly heated irradiated specimens are given in Table B-10. These test results indicate that the degree of embrittlement at 650 C is not significantly influenced by holding times at test temperatures of 240 sec or more.

TABLE B-10. ELONGATION OF IRRADIATED TYPE 347 STAINLESS STEEL AT 650 C AS A FUNCTION OF TIME AT TEMPERATURE BEFORE TESTING^(a)

Hold Time Before Testing, sec	Total Elongation, percent
~6000	0.9
800	2.3
240	1.6

(a) Specimens irradiated to a fast fluence of 2.2×10^{22} nvt.

250X

3C310

a. Unirradiated (22.4 Percent Uniform Elongation)

250X

HC21017

b. Irradiated to a Fast Fluence of 2.1×10^{22} nvt
(20.0 Percent Uniform Elongation)

FIGURE B-20. COMPARISON OF THE FRACTURES OF UNIRRADIATED AND
IRRADIATED TYPE 347 STAINLESS STEEL SPECIMENS
TESTED AT 500 C

250X

3C365

a. Unirradiated (17.8 Percent Uniform Elongation)

250X

HC20996

b. Irradiated to a Fast Fluence of 2.1×10^{22} nvt
(3.2 Percent Uniform Elongation)

FIGURE B-21. COMPARISON OF THE FRACTURES OF UNIRRADIATED AND
IRRADIATED TYPE 304 STAINLESS STEEL SPECIMENS
TESTED AT 600 C

3800X

E2018C

a. Unirradiated (16 Percent Uniform Elongation)

3800X

E2019E

b. Irradiated to a Fast Fluence of 2.1×10^{22} nvt
(0.5 Percent Uniform Elongation)

FIGURE B-22. FRACTURE SURFACES OF UNIRRADIATED AND IRRADIATED
TYPE 347 STAINLESS STEEL SPECIMENS TESTED AT 750 C

Metallography. Metallographic examinations were performed near the fracture areas of the tested unirradiated and irradiated specimens that had been annealed for 1 hr at 980 C. Figure B-20 shows the fracture areas of the specimens that were tested at 500 C. It can be seen that the fracture for both the irradiated and unirradiated material is similar and has taken place transgranularly. The fracture areas of the specimens tested at 600 C are shown in Figure B-21. Here the fracture is intergranular in both cases. However, the unirradiated specimen had a uniform elongation of 17.8 percent while the irradiated specimen had a uniform elongation of only 3.2 percent before fracture. Significantly, the irradiated specimens exhibit a complete freedom from wedge-type cracks and no evidence of grain deformation.

Fractography. Replicas were taken from the fracture surfaces of the tested specimens. The replicas were prepared for electron microscopy by the two-stage cellulose acetate-carbon technique and were shadowed with platinum-carbon from an angle of 45 deg. Figure B-22 illustrates the replicas which were taken from the fracture surfaces of specimens that were tested at 750 C. The replica from the unirradiated specimen shows elongated dimples which are characteristic of transgranular fracture. The replica from the irradiated specimen shows a rough surface and distinct grain-boundary facets. Figure B-23 is an electron fractograph of the replica from the irradiated specimen which was annealed at 980 C for 1 hr and then tested at 500 C. It shows that the irradiated and annealed specimen failed in a ductile transgranular manner when tested at 500 C.

8750X

J16565

FIGURE B-23. FRACTURE SURFACE OF IRRADIATED AND ANNEALED TYPE 347 STAINLESS STEEL

The specimen was irradiated to a fast fluence of 2.1×10^{22} nvt, annealed for 1 hr at 980 C, and tested at 500 C. The uniform elongation was 20.0 percent.

Tensile Tests on Other Materials Irradiated at 50 C. Tensile properties of Tenelon, UMCo-50, Haynes Stellite 25, and nickel were determined at temperatures ranging from room temperature to 650 C after irradiation. Before tensile testing, the irradiated specimens were annealed at elevated temperature to remove the displacement type of radiation damage. This was done so that the strength increases and ductility decreases due to defect clusters would not mask out the tensile property changes associated with the elevated temperature embrittlement phenomenon. The Nickel 200 and Tenelon specimens were annealed for 1 hr at 700 C while the UMCo-50 and Haynes Stellite 25 specimens were annealed for 1 hr at 800 C. One of the irradiated pure nickel specimens was tested in the as-irradiated condition while the other one was annealed for 1 hr at 600 C before testing. The unirradiated control specimens were given identical anneals before testing. These annealing temperatures are considerably lower than the annealing temperatures employed before irradiation. It is therefore believed that the post irradiation anneal does not influence the condition of the irradiated material except for the removal of defect clusters.

These tensile test results were then compared with tensile properties of unirradiated alloys that were tested under similar conditions. Comparative values for ultimate tensile strength, 0.2 percent offset yield strength, total elongation, and uniform elongation for the different alloys are given in Table B-11. Irradiation followed by annealing appears to cause a slight reduction in yield strength. However, this small difference may reflect the stiffness of different Instron testing machines that were used for testing the irradiated and control specimens, since no extensometers were used on the specimens. The tensile properties which were significantly affected by irradiation were the ultimate tensile strength and elongation. Uniform elongation, rather than total elongation, is utilized in these comparisons because a large number of the fractures for both unirradiated and irradiated specimens were intergranular and therefore the uniform elongation and total elongation were about the same for most of the tested specimens.

UMCo-50. The ultimate strength and uniform elongation of the UMCo-50 are compared in Figure B-24. At testing temperatures below 450 C the ultimate tensile strengths of the unirradiated and irradiated specimens are about the same. At higher testing temperatures the ultimate tensile strength of the irradiated specimens is lower and the difference increases with increasing testing temperature. The uniform elongation of the irradiated and unirradiated specimens is about the same for testing temperatures below 450 C. At temperatures above 450 C the uniform elongation of irradiated UMCo-50 decreases with increasing testing temperatures. Figure B-25 compares the stress-strain diagrams of unirradiated and irradiated and annealed UMCo-50 at 550 C. This diagram shows that the work-hardening characteristics of the irradiated and annealed specimen are similar to those of the unirradiated control specimen and that the reduced ultimate strength is a direct result of decreased ductility for the irradiated material.

TABLE B-11. TENSILE PROPERTIES OF IRRADIATED AND UNIRRADIATED TENELON, NICKEL, UMCo-50, AND HAYNES STELLITE 25

Material	Test Temperature, C	0.2 Percent Yield Strength, 1000 psi		Ultimate Tensile Strength, 1000 psi		Uniform Elongation, percent		Total Elongation, percent	
		Unirradiated	Irradiated	Unirradiated	Irradiated	Unirradiated	Irradiated	Unirradiated	Irradiated
Tenelon ^(a)	(b)	77.8	67.4	130.0	125.0	54.0	56.0	63.5	61.9
	315	44.5	35.0	95.5	86.5	41.5	34.5	45.5	38.0
	450	35.3	26.5	85.5	55.3	37.0	11.0	49.0	11.2
	550	30.3	26.0	77.5	48.5	32.5	6.4	33.0	6.5
	650	29.8	25.3	55.8	31.9	21.4	1.7	23.7	1.8
Nickel 200 ^(a)	(b)	21.0	18.8	74.3	73.0	39.0	23.8	42.0	26.7
	315	14.4	14.8	73.3	47.2	32.5	23.6	35.4	34.2
	400	10.8	12.4	50.6	31.6	32.0	12.4	44.6	16.4
	500	8.7	11.8	33.4	19.7	27.0	5.4	36.0	6.7
Pure nickel	500 ^(c)	3.6	8.7	22.1	13.0	19.8	1.5	22.0	2.0
	500 ^(d)	--	8.7	--	19.0	--	9.2	--	11.0
UMCo-50 ^(e)	(b)	47.5	--	126.0	127.0	35.0	27.4	35.0	27.4
	315	33.6	29.7	121.0	112.1	61	56.7	61	56.7
	450	28.9	26.0	82.5	75.1	23	29.7	24.5	30.7
	550	25.0	24.6	72.0	49.1	25.9	11.5	27.0	13.9
	650	23.9	--	61.5	33.6	29.5	8.0	29.5	8.0
Haynes Stellite 25 ^(e)	(b)	66.3	56.2	139	133	68	55.6	68	55.6
	315	39.7	36.0	118.5	117	86	60.2	86	60.2
	450	36.1	29.9	96.3	70.3	40	22.2	42	22.8
	550	33.9	28.5	82.3	53.5	28	8.6	30	8.6
	650	32.3	29.8	69.3	40.5	14	4.5	14.8	4.5

(a) All irradiated Nickel 200 and Tenelon specimens were annealed for 1 hr at 700 C before testing.

(b) Ambient testing temperature. In the case of the unirradiated specimens this was 25 C, but due to heating by irradiated specimens it was 45 C for Tenelon and Nickel 200 and 70 C for the cobalt alloys.

(c) As irradiated.

(d) Irradiated specimen was annealed for 1 hr at 600 C.

(e) All irradiated UMC0-50 and Haynes Stellite specimens were annealed for 1 hr at 800 C before testing.

FIGURE B-24. ULTIMATE STRENGTH AND UNIFORM ELONGATION OF UNIRRADIATED AND IRRADIATED UMC0-50 AS A FUNCTION OF TESTING TEMPERATURE

FIGURE B-25. STRESS-STRAIN DIAGRAM OF UNIRRADIATED AND IRRADIATED UMC0-50 TESTED AT 550 C

Haynes Stellite 25. The tensile properties of unirradiated Haynes Stellite 25 compared with those of the irradiated material in Figure B-26. The ultimate tensile strength of both irradiated and control specimens is about the same provided the testing temperature is 315 C or less. At higher testing temperatures the ultimate strength is decreased by irradiation and the magnitude of the strength loss increases with increasing testing temperature. The uniform elongation is reduced by irradiation to about the same degree at all testing temperatures. Figure B-27 compares the stress-strain curves for unirradiated and irradiated and annealed Haynes Stellite 25 specimens that were tested at 650 C. Comparison of these stress-strain diagrams indicates that, although ductilities and ultimate strengths are widely different, the strain-hardening characteristics of the irradiated and annealed specimen are very similar to those of the control specimen.

Tenelon. Tensile properties of Tenelon before and after irradiation are given in Figure B-28. The ultimate tensile strength of Tenelon is reduced by irradiation for all testing temperatures. However, the reduction of strength due to irradiation is minor at temperatures of 315 C and below but increases with increasing testing temperature above 315 C. Below 315 C the uniform elongation does not appear to be influenced by irradiation. However, above 315 C, the uniform elongation of Tenelon is significantly reduced by irradiation.

Nickel 200. The effect of irradiation on the ultimate strength and uniform elongation of Nickel 200 is illustrated in Figure B-29. The ultimate tensile strength of Nickel 200 is reduced by irradiation at all testing temperatures except at room temperature. Uniform elongation is also reduced by irradiation at all testing temperatures. However, the magnitude of the irradiation-induced ductility loss becomes especially significant at testing temperatures above 315 C.

Pure Nickel. Tensile tests were performed on pure nickel at 500 C in the as-irradiated condition and after annealing for 1 hr at 600 C. Stress-strain curves for these tests as well as for an unirradiated control specimen are shown in Figure B-30. Radiation causes a significant increase in yield strength while the ultimate strength and elongation are reduced. Annealing for 1 hr at 600 C did not change the yield strength significantly while considerable recovery in ultimate strength and elongation occurred. The large irradiation-induced increase in yield strength, which persists even after annealing at 600 C, is rather surprising. One possible explanation of this behavior is that helium atoms, produced in large numbers by thermal- and fast-neutron reactions with nickel, and copper, produced by thermal-neutron reactions with nickel, are responsible for the yield strength increase as a result of lattice strengthening.

Metallography. Metallographic examinations of the fracture areas were performed for both the irradiated and unirradiated test specimens. These fracture areas are shown in Figures B-31 through B-34, where the microstructures of the irradiated and unirradiated specimens are compared for the same testing

FIGURE B-26. ULTIMATE STRENGTH AND UNIFORM ELONGATION OF UNIRRADIATED AND IRRADIATED HAYNES STELLITE 25 AS A FUNCTION OF TESTING TEMPERATURE

FIGURE B-27. STRESS-STRAIN CURVES FOR UNIRRADIATED AND IRRADIATED HAYNES STELLITE 25 TESTED AT 650 C

FIGURE B-28. ULTIMATE TENSILE STRENGTH AND UNIFORM ELONGATION OF UNIRRADIATED AND IRRADIATED TENELON AS A FUNCTION OF TESTING TEMPERATURE

FIGURE B-29. ULTIMATE TENSILE STRENGTH AND UNIFORM ELONGATION OF UNIRRADIATED AND IRRADIATED NICKEL 200 AS A FUNCTION OF TESTING TEMPERATURE

FIGURE B-30. STRESS-STRAIN CURVES FOR UNIRRADIATED, IRRADIATED, AND IRRADIATED AND ANNEALED PURE NICKEL TESTED AT 500 C

100X

8D464

a. Unirradiated (21.4 Percent Uniform Elongation)

100X

HC21926

b. Irradiated (1.7 Percent Uniform Elongation)

FIGURE B-31. FRACTURE AREA OF TENELON TESTED IN TENSION AT 650 C
BEFORE AND AFTER IRRADIATION TO 1.5×10^{21} NVT

100X

2D820

a. Unirradiated (25.9 Percent Uniform Elongation)

100X

HC21922

b. Irradiated (11.5 Percent Uniform Elongation)

FIGURE B-32. FRACTURE AREA OF UMC0-50 TESTED IN TENSION AT 550 C
BEFORE AND AFTER IRRADIATION TO 1.4×10^{21} NVT

100X

8D459

a. Unirradiated (14 Percent Uniform Elongation)

100X

HC21916

b. Irradiated (4.4 Percent Uniform Elongation)

FIGURE B-33. FRACTURE AREAS OF HAYNES STELLITE 25 TESTED IN TENSION AT 650 C
BEFORE AND AFTER IRRADIATION TO 1.4×10^{21} NVT

100X

2D810

a. Unirradiated (27 Percent Uniform Elongation)

100X

HC21939

b. Irradiated (5.4 Percent Uniform Elongation)

FIGURE B-34. FRACTURE AREAS OF NICKEL 200 TESTED IN TENSION AT 500 C
BEFORE AND AFTER IRRADIATION TO 1.5×10^{21} NVT

temperature. For comparison, a temperature was chosen at which the uniform elongation had been significantly reduced by irradiation. The unirradiated specimens of Tenelon, UMC0-50, and Haynes Stellite 25 show considerable elongation of the grains, with the fracture mode being a mixture of transgranular and intergranular fracture. Unirradiated Nickel 200 was found to fail completely by transgranular fracture at 500 C.

Irradiation appears to promote the tendency toward intergranular fracture in UMC0-50, Tenelon, and Haynes Stellite 25. This is characterized by reduced elongation of the grains and with an increase in the percentage fracture path which follows the grain boundaries. Although fracture in the irradiated specimens occurs at the grain boundaries there are only a minimal number of grain-boundary cracks in areas away from the fracture. This implies that the first grain-boundary crack formed propagates and causes fracture of the whole specimen. In the case of Nickel 200, the fracture mode itself is changed from complete transgranular to complete intergranular, as shown in Figure B-34.

Tensile Tests on Stainless Steel Irradiated at 400 and 650 C. Tensile properties of the Type 347 and Type 348 stainless steel specimens were determined for both the as-irradiated condition and the irradiated and annealed condition.

As-Irradiated Specimens. Tensile tests were performed on irradiated Type 347 and Type 348 stainless steel specimens at temperatures ranging from room temperature to 750 C. These specimens had been irradiated at either 400 or 650 C. The tensile properties of irradiated Type 347 stainless steel specimens are given in Table B-12 (see Table B-7 for unirradiated specimen properties), while Table B-13 gives the tensile properties of Type 348 stainless steel specimens. The yield strength, uniform elongation, total elongation, reduction in area, and ultimate strength of the irradiated specimens of Type 347 and Type 348 stainless steel, as a function of testing temperature, are illustrated in Figures B-35 through B-39. In these figures the tensile properties of material irradiated at 50 C are included for comparison.

The tensile properties of irradiated Type 347 and Type 348 stainless steel show similar trends. Irradiation at 400 C causes larger increases in yield strength than irradiation at 650 C. The ultimate strength is also increased to a greater degree by irradiation at 400 C than at 650 C. As the testing temperature is increased the yield strength and ultimate strength of the irradiated material approach those of the unirradiated material and at the higher testing temperatures the strength of the irradiated material is in some cases less than that of the unirradiated stainless steel.

Both the uniform elongation and total elongation of the stainless steel are reduced by irradiation at all testing temperatures. Irradiation at 400 C causes equal or larger reductions in ductility than irradiation at 650 C for all testing temperatures. Of special note is the rather large reduction in both uniform and total elongation when testing is done above about 500 C. Values for reduction in

FIGURE B-35. YIELD STRENGTH OF TYPES 347 AND 348 STAINLESS STEEL

FIGURE B-36. UNIFORM ELONGATION OF TYPES 347 AND 348 STAINLESS STEEL

FIGURE B-37. TOTAL ELONGATION OF TYPES 347 AND 348 STAINLESS STEEL

FIGURE B-38. REDUCTION IN AREA OF TYPES 347 AND 348 STAINLESS STEEL

FIGURE B-39. ULTIMATE STRENGTH OF TYPES 347 AND 348 STAINLESS STEEL

TABLE B-12 TENSILE PROPERTIES OF TYPE 347 STAINLESS STEEL
IRRADIATED AT ELEVATED TEMPERATURES

Test Temperature, C	0.2 Percent Offset Yield Strength, 1000 psi	Ultimate Strength, 1000 psi	Uniform Elongation, percent	Total Elongation, percent	Reduction in Area, percent
<u>Irradiated at 400 C</u>					
25	109.0	131.0	12.3	20.6	89
315	95.6	104.0	4.8	9.5	47
750	18.0	25.4	2.2	2.3	0.1
<u>Irradiated at 650 C</u>					
25	39.1	95.5	47.5	56.7	88
315	31.4	63.5	24.0	30.6	65
750	18.7	21.2	3.1	3.7	5.0
<u>Irradiated at 650 C and Annealed 1 Hr at 980 C</u>					
25	32.5	92.2	57.5	69.9	91
315	22.7	64.5	31.2	37.0	53
750	14.5	18.2	2.5	3.6	6.0

TABLE B-13 TENSILE PROPERTIES OF UNIRRADIATED AND IRRADIATED
TYPE 348 STAINLESS STEEL

Test Temperature, C	0.2 Percent Offset Yield Strength, 1000 psi	Ultimate Strength, 1000 psi	Uniform Elongation, percent	Total Elongation, percent	Reduction in Area, percent
<u>Unirradiated</u>					
25	36.2	93.0	58	61.4	82
315	27.8	62.8	26.7	29.4	74
440	23.2	60.2	22.5	25	70
500	23.8	53.2	22.7	25.2	69
600	23.4	49.1	21.5	30.3	64
750	19.0	27.3	4	35	55
<u>Irradiated at 400 C</u>					
315	88.1	93.1	3.5	6.6	60
500	70.6	80.9	2.7	4.9	17
600	44.9	45.8	1.4	3.2	13
<u>Irradiated at 650 C</u>					
25	38.0	--	50	--	--
315	33.7	63.7	21.0	24.4	69
425	27.6	55.1	19.7	24.6	46
500	26.2	48.9	14.8	15.7	15
600	26.8	38.7	6.5	6.9	6.9
<u>Irradiated at 650 C and Annealed 1 Hr at 980 C</u>					
500	25.2	58.5	24.5	32.2	48
600	23.4	42.1	13.1	14.6	29
750	15.1	17.6	4.6	6.3	14

area are slightly reduced by irradiation when specimens are tested at 315 C. However, at higher testing temperatures the values for reduction in area are drastically reduced by irradiation. Typical stress-strain curves for both the unirradiated and irradiated specimens are illustrated in Figures B-40 and B-41.

Irradiated and Annealed Specimens. Selected specimens were annealed after irradiation for 1 hr at 980 C to see whether the anneal would restore the pre-irradiation tensile properties. Annealing of the irradiated specimens for 1 hr at 980 C resulted in restoration of the preirradiation tensile properties if the testing was performed at about 500 C or below. At testing temperatures above 500 C the strength properties were reduced but the annealed, irradiated specimens still exhibited significant losses in ductility although some of the ductility was restored by the anneal. Thus, the elevated-temperature embrittlement cannot be removed by postirradiation annealing.

Metallographic Examination. Metallographic examination was carried out on selected unirradiated and irradiated specimens that were tested in tension. Of special interest in this examination was the appearance of the fracture area. The irradiation and testing conditions of the specimens that were examined are given in Table B-14. Figures B-42 through B-44 compare the fracture area of an unirradiated specimen of Type 348 stainless steel that was tested at 500 C with irradiated specimens that were also tested at 500 C.

TABLE B-14. IRRADIATION AND TESTING CONDITIONS OF SPECIMENS THAT WERE EXAMINED BY LIGHT MICROSCOPY AND FRACTOGRAPHY

Stainless Steel Material	Irradiation Temperature, C	Testing Conditions
Type 347	650	Annealed 1 hr at 980 C, tested at 750 C
Type 347	650	Tested at 750 C
Type 347	400	Tested at 315 C
Type 348	400	Tested at 500 C
Type 348	650	Tested at 500 C
Type 348	650	Annealed 1 hr at 980 C, tested at 500 C

FIGURE B-40. ENGINEERING STRESS-STRAIN CURVES FOR TYPE 348 STAINLESS STEEL TESTED IN TENSION AT 315 C

FIGURE B-41. ENGINEERING STRESS-STRAIN CURVES FOR TYPE 348 STAINLESS STEEL TESTED IN TENSION AT 600 C

250X

5C926

FIGURE B-42. FRACTURE APPEARANCE OF UNIRRADIATED TYPE 348 STAINLESS STEEL TESTED IN TENSION AT 500 C (27 PERCENT TOTAL ELONGATION)

Figure B-42 shows that the unirradiated specimen which was tested at 500 C fractured transgranularly. This specimen underwent an elongation of 27 percent; this is evident considering the large amount of elongation of the grains. Figure B-43 shows the fracture area of the specimen that was irradiated at 400 C and tested in tension at 500 C. This specimen exhibited an intergranular fracture. There is very little elongation of the grains and voids are visible at grain boundaries. Figure B-44 shows the fracture area of the specimen that was irradiated at 650 C, annealed for 1 hr at 980 C, and then tested at 500 C. The fracture appears to be completely transgranular and is comparable to that of the unirradiated specimen shown in Figure B-42. Since the specimen had a total elongation of 32 percent, which is actually more than that of the unirradiated specimen, this is consistent with the fracture behavior.

Figures B-45 and B-46 show the fracture areas of unirradiated and irradiated Type 347 stainless steel specimens that were tested in tension at 315 C. The unirradiated specimen failed by transgranular fracture with considerable elongation of the grains. Although irradiation at 400 C reduced the total elongation from 36 to 9.5 percent, the irradiated specimen still fractured transgranularly.

Figures B-47 and B-48 compare the fracture areas of unirradiated and irradiated Type 347 stainless steel specimens that were tested in tension at 750 C. The unirradiated specimen exhibits a large number of voids, with the fracture being apparently intergranular. The irradiated specimen also exhibits an

250X

HC23669

FIGURE B-43. FRACTURE APPEARANCE OF TYPE 348 STAINLESS STEEL IRRADIATED AT 400 C TO A FAST FLUENCE OF 5×10^{21} NVT AND TESTED IN TENSION AT 500 C (4.9 PERCENT TOTAL ELONGATION)

250X

HC23665

FIGURE B-44. FRACTURE APPEARANCE OF TYPE 348 STAINLESS STEEL IRRADIATED AT 650 C TO A FAST FLUENCE OF 5×10^{21} NVT, ANNEALED FOR 1 HR AT 980 C, AND TESTED IN TENSION AT 500 C (32 PERCENT TOTAL ELONGATION)

100X

4F930

FIGURE B-45. FRACTURE APPEARANCE OF UNIRRADIATED TYPE 347 STAINLESS STEEL TESTED IN TENSION AT 315 C (36 PERCENT TOTAL ELONGATION)

100X

HC25118

FIGURE B-46. FRACTURE APPEARANCE OF TYPE 347 STAINLESS STEEL IRRADIATED AT 400 C AND TESTED IN TENSION AT 315 C (9.5 PERCENT TOTAL ELONGATION)

250X

3C675

FIGURE B-47. FRACTURE APPEARANCE OF UNIRRADIATED TYPE 347 STAINLESS STEEL TESTED IN TENSION AT 750 C (26 PERCENT TOTAL ELONGATION)

250X

HC23679

FIGURE B-48. FRACTURE APPEARANCE OF TYPE 347 STAINLESS STEEL IRRADIATED AT 650 C TO A FAST FLUENCE OF 5×10^{21} NVT AND TESTED IN TENSION AT 750 C (3.7 PERCENT TOTAL ELONGATION)

intergranular fracture, but without elongation of the grains. There were also a large number of cracks near the fracture surface and at the side of the specimen. There is a considerable difference in the overall behavior and appearance of the two materials. Grain-boundary voids apparently form by shear in the unirradiated material and these elongate as the grains deform plastically. In the irradiated material, on the other hand, cracks nucleate at the grain boundaries and grow with little associated grain deformation.

Fractography. Replicas were obtained from the fracture surfaces of all the irradiated specimens that are enumerated in Table B-14, except for the specimen tested at 315 C. For comparison, replicas were also obtained from unirradiated stainless steel specimens which were tested in tension at 500 and 750 C. A typical fracture surface of the unirradiated specimens was illustrated earlier in Figure B-22a. The unirradiated specimens fractured transgranularly with considerable plastic flow, as indicated by presence of the dimples.

Both of the irradiated specimens that were tested at 750 C showed completely intergranular fracture. This is evident from the grain-boundary facets shown in Figures B-49 and B-50. The specimen that was irradiated at 650 C and tested at 750 C shows the possible presence of bubbles at the fracture which occurred at the grain boundaries. In the case of the specimen that was irradiated at 650 C, annealed for 1 hr at 980 C, and tested at 750 C there do not appear to be any bubbles. However, this specimen exhibits a dimpled structure at the grain boundaries. It is hypothesized that these dimples may have formed from helium bubbles that were enlarged by the annealing at 980 C and then assumed the appearance of dimples when they were locally deformed.

The specimen that was irradiated at 400 C and tested at 500 C shows complete intergranular fracture (see Figure B-51a). The grain boundaries are decorated by a large number of small bubbles. Figure B-51b shows these grain-boundary bubbles at higher magnification.

The specimen that was irradiated at 650 C and tested at 500 C exhibits a mixture of intergranular fracture (Figure B-52a) and transgranular fracture (Figure B-52b). The transgranular fracture appears very similar to that exhibited by the unirradiated specimen.

Stress Rupture Tests. Stress rupture tests were performed on specimens of Type 347 stainless steel that were irradiated to a fast fluence of 2.2×10^{22} nvt at 50 C. These tests were performed at 650 C with the stresses ranging from 17,500 psi to 28,500 psi. Results of these tests are summarized in Figure B-53. It is apparent that irradiation reduces the fracture life by a factor of 8 to 10. Figure B-54 compares the creep curves for the unirradiated and irradiated specimens at a stress of 28,500 psi. The irradiated specimen underwent an initial strain on loading, but no further elongation was generally detected. This specimen underwent an elongation of less than 0.2 percent before fracture occurred as compared to about 10 to 25 percent elongation for the unirradiated specimen.

7500X

857

a. General View of Fracture

15,000X

852

b. Close-Up of Intergranular Fracture

FIGURE B-49. APPEARANCE OF FRACTURE SURFACE FOR TYPE 347 STAINLESS STEEL SPECIMEN WHICH WAS IRRADIATED AT 650 C AND TESTED IN TENSION AT 750 C (3.7 PERCENT TOTAL ELONGATION)

7500X

859

a. General View of Intergranular Fracture

5,000X

862

b. Dimpled Structure at Grain Boundaries

FIGURE B-50. APPEARANCE OF FRACTURE SURFACE FOR TYPE 347 STAINLESS STEEL SPECIMEN WHICH WAS IRRADIATED AT 650 C, ANNEALED FOR 1 HR AT 980 C, AND TESTED IN TENSION AT 750 C (3.6 PERCENT TOTAL ELONGATION)

7500X

832

a. Intergranular Fracture

50,000X

833

b. Grain-Boundary Bubbles at Higher Magnification

FIGURE B-51. APPEARANCE OF FRACTURE SURFACE FOR TYPE 348 STAINLESS STEEL SPECIMEN WHICH WAS IRRADIATED AT 400 C AND TESTED AT 500 C (4.9 PERCENT TOTAL ELONGATION)

15,000X

880

a. Intergranular Fracture

7500X

878

b. Transgranular Fracture

FIGURE B-52. APPEARANCE OF FRACTURE SURFACE FOR TYPE 348 STAINLESS STEEL WHICH WAS IRRADIATED AT 650 C AND TESTED AT 500 C (15.7 PERCENT TOTAL ELONGATION)

FIGURE B-53. EFFECT OF IRRADIATION ON THE STRESS RUPTURE PROPERTIES OF TYPE 347 STAINLESS STEEL TESTED AT 650 C

FIGURE B-54. CREEP CURVES FOR UNIRRADIATED AND IRRADIATED TYPE 347 STAINLESS STEEL TESTED AT 650 C AND A STRESS OF 28,500 psi

Figure B-55 compares the fracture appearance of the unirradiated and irradiated specimens. The unirradiated specimen underwent an elongation of 23.3 percent and shows a large amount of void formation at grain boundaries. In the case of the irradiated material, the elongation at failure was less than 0.2 percent and there were no cracks at the grain boundary away from the fracture.

Fatigue Tests. Fatigue tests were carried out at both room temperature and 650 C on the Type 347 stainless steel specimens irradiated at 50 C.

Room-Temperature Tests. The room-temperature tests were performed on specimens that were irradiated to fast fluences of 5.5×10^{21} , 11×10^{21} , and 16×10^{21} nvt(2). Results of the fatigue tests are summarized in Table B-15 and plotted in Figure B-56. For the material that was irradiated to 5.5×10^{21} nvt, the fatigue life is decreased for strains above about 1.9 percent and improved for strains below that. Irradiation to a fast fluence of 1.1×10^{22} nvt reduces the fatigue life slightly for all strains, while irradiation to 1.6×10^{22} nvt reduces the fatigue life by a factor of about 10.

The improved fatigue life for material irradiated to 5.5×10^{21} nvt is attributed to an increase in proportional limit. Thus for equivalent total strains, an irradiated material undergoes less plastic strain than an unirradiated material. For equivalent plastic strains, the irradiated specimens show significantly lower fatigue life than the unirradiated specimens (Figure B-57). Thus at higher fluences the irradiation-induced ductility decrease becomes important and the fatigue life is reduced by irradiation.

Elevated-Temperature Tests. Cyclic-stress fatigue tests were also performed on Type 347 stainless steel that was irradiated at 50 C to fast fluences of 2.2×10^{21} nvt. Table B-16 compares the fatigue life at 650 C of unirradiated and irradiated Type 347 stainless steel. It can be seen that irradiation results in about a five-fold decrease in fatigue life at 650 C. It was not possible to exactly determine the elongation at fracture for the unirradiated specimens, but a post-irradiation length measurement indicated that the elongations were less than 1 percent. Measurements on the unirradiated specimens showed that the specimen that failed after 7485 cycles had undergone an elongation of 19 percent in the gage section. Therefore, it is believed that the irradiation-induced loss in fatigue life is due to the embrittlement which is exhibited by austenitic stainless steels at elevated temperatures. Metallographic examinations of fractured fatigued specimens support these findings in that the unirradiated specimen exhibits a large number of grain-boundary voids resulting from grain-boundary shear and grain deformation, while the irradiated specimen does not show either cracks or voids at the grain boundaries.

250X

2D967

a. Unirradiated Specimen

Under a stress of 35,000 psi, the specimen had a rupture life of 31.5 hr. Elongation was 23.3 percent.

250X

HQ896

b. Irradiated at 50 C to 2.2×10^{22} nvt

Under a stress of 28,500 psi, the specimen had a rupture life of 20.8 hr. Elongation was less than 0.2 percent.

FIGURE B-55. APPEARANCE OF FRACTURE OF TYPE 347 STAINLESS STEEL SPECIMENS TESTED IN STRESS RUPTURE AT 650 C

TABLE B-15. ROOM-TEMPERATURE CYCLIC-STRAIN FATIGUE PROPERTIES OF IRRADIATED AND UNIRRADIATED TYPE 347 STAINLESS STEEL

Total Fast Fluence, 10^{21} nvt	Strain Range, percent		Strain-Range Ratio (ϵ_p / ϵ_t)	Number of Cycles to Failure		Stress Range ($\Delta\sigma$) At Crack Initiation, psi
	Plastic (ϵ_p)	Total (ϵ_t)		At Crack Initiation	At Separation	
0	0.17	0.57	0.30	97,000	98,210	63,300
0	0.23	0.67	0.34	34,000	36,735	72,000
0	0.58	1.17	0.50	6,400	6,886	81,300
0	1.08	1.84	0.59	1,650	1,841	84,200
5.5	0.04	0.54 ^(a)	0.07	56,000	59,192	95,600
5.5	0.30	0.97 ^(b)	0.31	11,000	11,685	104,200
5.5	0.46	1.29	0.36	4,700	5,142	125,000
5.5	0.62	1.51	0.41	1,300	1,824	141,500
5.5	1.04	1.99	0.52	950	969	152,000
11	0.02	0.49	0.04	809,000 ^(c)	--	81,500 ^(c)
11	0.13	0.86	0.15	56,000	56,950	107,800
11	0.23	1.10	0.21	14,500	16,316	128,500
11	0.61	1.72	0.35	2,300	2,427	154,500
11	0.82	1.98	0.41	1,350	1,406	150,500
16	0.35	1.40	0.25	5,000	5,289	160,000
16	0.59	1.92	0.31	1,700	1,892	192,000

(a) Total strain varied from 0.54 to 0.71 percent.

(b) Total strain varied from 0.97 to 1.10 percent.

(c) Specimen did not fail.

TABLE B-16. CYCLIC-FATIGUE PROPERTIES OF IRRADIATED AND UNIRRADIATED TYPE 347 STAINLESS STEEL TESTED AT 650 C^(a)

Stress Range, 100 psi		Number of Cycles to Failure	
Minimum	Maximum	Irradiated ^(b)	Unirradiated
2.0	22.0	2,368	15,324 ^(c)
2.0	27.0	1,496	7,485

(a) The specimens were cycled at 50 cycles per hr using a crosshead speed of 0.02 in. per min.

(b) Specimens were irradiated to a fast fluence of 2.2×10^{22} nvt.

(c) Specimen did not fail; the test was discontinued.

FIGURE B-56. FATIGUE LIFE OF UNIRRADIATED AND IRRADIATED TYPE 347 STAINLESS STEEL

FIGURE B-57. THE EFFECT OF PLASTIC STRAIN PER CYCLE ON THE FATIGUE LIFE OF IRRADIATED AND UNIRRADIATED TYPE 347 STAINLESS STEEL TESTED AT 20 C

Transmutations. The cause of the irradiation-induced elevated-temperature embrittlement was originally attributed to helium production by neutron reaction with the impurity boron^(11, 12); i. e., $B^{10} (n, \alpha) Li^7$. However the Type 347 stainless steel irradiated in the ETR to fast fluences of 2.1×10^{22} nvt contained only 0.5 ppm of boron, yet exhibited more severe embrittlement than any other stainless steel⁽¹³⁾. Consequently it was considered that some other transmutation product, aside from the helium originating from boron, was responsible for the extreme embrittlement that was observed. The possibilities that were considered included:

- (1) Solid transmutation products originating from the various elements originally present in the Type 347 stainless steel
- (2) Production of hydrogen from (n, p) reactions with alloy constituents
- (3) Sources of helium other than by transmutation from boron and from known (n, α) reactions between alloy constituents and fast neutrons.

Solid Transmutations. Because the capture cross sections for thermal neutrons are considerably higher and the cross sections for fast neutrons are not well known, only the possible transmutations caused by thermal neutrons were considered. So that the transmutations produced by thermal neutrons might be predicted for a long irradiation period, a machine code was developed to calculate the concentrations of a few pertinent chains. Since many of the thermal absorption cross sections for different isotopes were unknown, it was necessary to perform calculations to determine how significant the unknown cross sections were. For each chain the following input information was required: length of chain, flux, time, initial concentrations, cross sections, isotopic abundance, and decay constants.

Where cross sections were unknown, they were assigned some value which was as large as practical. On subsequent runs, the cross section was decreased and the difference in the final result noted. In this manner the significance of unknown cross sections was determined. It was found that, in most cases, the results of the reactions, where the cross sections were unknown, were not affected to a large extent by the uncertainty in cross-section value. An irradiation time of 10^8 sec and an instantaneous thermal flux of 4×10^{14} nv were assumed. These values were chosen because they represented the average instantaneous thermal flux to which the specimens were exposed. For simplicity, the 2-week shutdowns between cycles and the other extended shutdowns were neglected.

It was believed that if significant compositional changes took place, they would occur by neutron capture followed by either beta decay, proton decay, alpha decay, or electron capture. If gamma decay occurred, no changes in mass or atomic numbers would occur and the chemical composition would not be changed. When beta decay occurs the atomic number would be increased which the mass number would remain the same. In case of proton and alpha decay both the mass

TABLE B-18. ROUTES OF EXPECTED TRANSMUTATIONS^(a)
ORIGINATING FROM STABLE Ni⁶² AND Ni⁶⁴

(a) Code: x - unknown thermal-neutron capture cross section

■ - stable isotope.

Since the capture cross sections were not known for Ni⁶³ and Cu⁶⁴ various cross sections were assumed. It was found that varying the Cu⁶⁴ cross section from 0 to 6 barns did not affect the amount of zinc produced. Varying the Ni⁶³ cross section from 0 to 12 barns also did not significantly change the amount of zinc resulting from nickel transmutation, as shown in Table B-19.

TABLE B-19. DEPENDENCE OF ZINC PRODUCTION FROM NICKEL ON THE THERMAL-NEUTRON CAPTURE CROSS SECTION OF Ni⁶³

Isotope	Zinc Fraction for Indicated Capture Cross Section for Ni ⁶³ , atoms per atom of total nickel				
	0 Barns	3 Barns	6 Barns	9 Barns	12 Barns
Zn ⁶⁴	1.29 x 10 ⁻⁶	1.25 x 10 ⁻⁶	1.21 x 10 ⁻⁶	1.17 x 10 ⁻⁶	1.14 x 10 ⁻⁶
Zn ⁶⁵	3.52 x 10 ⁻⁹	3.43 x 10 ⁻⁹	3.34 x 10 ⁻⁹	3.26 x 10 ⁻⁹	3.18 x 10 ⁻⁹
Zn ⁶⁶	2.76 x 10 ⁻⁵	2.81 x 10 ⁻⁵	2.86 x 10 ⁻⁵	2.90 x 10 ⁻⁵	2.94 x 10 ⁻⁵
Zn ⁶⁷	7.42 x 10 ⁻⁷	7.50 x 10 ⁻⁷	7.58 x 10 ⁻⁷	7.66 x 10 ⁻⁷	7.73 x 10 ⁻⁷
Zn Total	2.96 x 10 ⁻⁵	3.01 x 10 ⁻⁵	3.06 x 10 ⁻⁵	3.10 x 10 ⁻⁵	3.13 x 10 ⁻⁵

In general, it appears that although very little nickel transmutes to zinc, a considerable fraction of copper is converted to zinc and some gallium is produced from copper, as shown in Table B-20. The gallium production is small because all gallium isotopes with a mass number less than 69 are unstable and decay by β^+ emission back to zinc isotopes.

TABLE B-20. ZINC AND GALLIUM PRODUCTION FROM COPPER BY NEUTRON-INDUCED TRANSMUTATIONS

Isotope	Zinc or Gallium Fraction ^(a) , atoms per atom of total copper
Zn ⁶⁴	1.13×10^{-2}
Zn ⁶⁵	4.50×10^{-5}
Zn ⁶⁶	2.61×10^{-2}
Zn ⁶⁷	1.03×10^{-3}
Zn ⁶⁸	4.18×10^{-5}
Zn ^{69m}	1.39×10^{-9}
Zn ⁶⁹	8.75×10^{-11}
<u>Zn Total</u>	<u>3.84×10^{-2}</u>
Ga ⁶⁹	4.60×10^{-8}

(a) The results are independent of Cu⁶⁷ cross sections ≤ 6 barns.

Because of the large capture cross section of manganese for thermal neutrons, calculations were made to see how much of the manganese would be burned out. By using a capture cross section of 13.3 barn and a thermal fluence of 4×10^{22} nvt, it was calculated that 40 percent of the manganese would be depleted. This manganese would be converted to iron by the $\text{Mn}^{55}(\text{n}, \beta^-)\text{Fe}^{56}$ reaction.

Table B-21 summarizes the predicted changes in composition induced by thermal-neutron irradiation of Type 347 stainless steel. These values represent only significant additions and no attempt has been made to correct for burnout of any element except for manganese.

TABLE B-21. CALCULATED COMPOSITION CHANGES IN TYPE 347 STAINLESS STEEL AS A RESULT OF NEUTRON-INDUCED TRANSMUTATIONS

Element	Initial Content, w/o	Calculated Change ^(a) , w/o	Origin of Transmutation ^(b)
Manganese	1.53	- 0.61	
Sulfur	0.016	+ 3×10^{-8}	Silicon (0.65 w/o)
Phosphorus	0.024	+ 1×10^{-6}	Silicon (0.65 w/o)
Zinc	0.005	+ 0.0003	Nickel (10.41 w/o)
		+ 0.0107	Copper (0.28 w/o)
Gallium	0.006	+ 1×10^{-8}	Copper (0.28 w/o)

(a) No corrections are made for possible burnout of element by neutron-induced transmutations, except for manganese.

(b) Element concentration in Type 347 stainless steel.

It can be seen that the only significant compositional changes predicted by the transmutation calculations are the buildup of zinc to about three times its original composition and the depletion of manganese.

It must again be stressed that only thermal-neutron activation is being considered. The results from these studies indicate that, under the assumed irradiation conditions, the transmutation effects taking place from silicon, nickel, and copper are rather small. This is not to say, however, that the effects due to bombardment by neutrons of epithermal and fast energies are insignificant. The contributions from the high cross sections in the resonance region may also contribute significantly to transmutations. It can also be concluded that the dependence of the transmutations on unknown cross sections is generally small. This indicates that analysis of long chain transmutations may be performed with sufficient accuracy by a digital computer.

To establish whether compositional changes were occurring in the Type 347 stainless steel, analyses were performed on the high irradiated material. To accomplish this, wet chemical analyses, emission spectrographic analyses, and microprobe analyses were performed.

Standard wet chemical analyses were performed on the irradiated stainless steel for manganese and sulfur. Simultaneous chemical analyses were also performed on unirradiated control samples. Results of the analyses are shown in Table B-22. These analyses confirm the predictions based on the calculations in that the sulfur content is unaffected and the manganese content is reduced by about the amount predicted.

TABLE B-22. CHEMICAL ANALYSIS OF IRRADIATED
TYPE 347 STAINLESS STEEL

Fast Fluence (>1 Mev), nvt	Thermal Fluence, nvt	Manganese, w/o	Sulfur, w/o
Unirradiated control	0	1.53 ± 0.04	0.015 ± 0.001
2.1×10^{22}	4×10^{22}	1.13	0.019
2.1×10^{22}	4×10^{22}	1.04	0.012

A series of spark emission spectrographic analyses was performed on irradiated and unirradiated Type 347 stainless steel samples. The spark emission from both the unirradiated and irradiated samples was obtained from the fracture surface of specimens, annealed for 1 hr at 1350 C, and then tested at 750 C. This specific specimen was chosen because it exhibited the largest degree of post-irradiation embrittlement. Consequently it was believed that if the embrittlement were due to an element, this element should be most prevalent on the fracture surface of this specimen. The analysis was taken from the fracture surface of the

irradiated specimen because it was suspected that a radiation-induced compositional change may have caused the intergranular fractures at elevated temperature. The spark vaporized a volume of material which included the whole surface area (0.02 in.^2) of the fracture and extended to a depth of 2 to 3 mils. This depth was sufficient to include about one to two whole grains in the analysis. Therefore, the analysis was not limited to the immediate grain-boundary area as planned. The results of the emission spectrographic analysis are given in Table B-23. For comparison, analyses of the unirradiated stainless steel are also included. These include wet chemical analyses and two emission spectrographic analyses, one of which was obtained from a cut surface and the other from the fracture surface.

TABLE B-23. COMPOSITIONAL ANALYSIS OF IRRADIATED AND UNIRRADIATED
TYPE 347 STAINLESS STEEL

Element	Composition of Unirradiated Material, w/o			Composition at Fracture Surface of Irradiated Material ^(b) , w/o	
	Chemical ^(a)	Cut Section ^(b)	Fracture Surface ^(b)	Expected ^(c)	Analyzed
Cobalt	0.10	0.10	0.10	--	0.10
Chromium	17.90	17.50	15.00	--	15.10
Copper	0.30	0.30	0.37	0.38	0.52
Manganese	1.72	1.70	1.10	0.66	0.80
Molybdenum	0.20	0.24	0.38	--	0.29
Lead	0.03	0.03	0.02	--	0.06
Silicon	0.65	0.62	0.86	--	0.64
Tin	0.04	0.04	0.05	--	0.07
Titanium	0.10	0.10	0.13	--	0.15
Vanadium	0.03	0.03	0.06	--	0.26
Zinc	0.005	0.03	0.06	0.07	0.20
Tungsten	0.04	0.04	0.10	--	0.04
Niobium	0.67	0.70	0.80	--	0.70
Phosphorus	0.024	0.027	0.020	--	0.027

(a) Wet chemical and emission spectrographic analyses performed at Battelle-Columbus.

(b) Emission spectrograph analysis performed by Idaho Nuclear.

(c) Based on the fracture surface of unirradiated stainless steel and changes due to irradiation (2.1×10^{22} nvt).

With a few minor exceptions (zinc, manganese) which may indicate segregation, all of the analyses on the unirradiated specimens are about the same. From these analyses it can be seen that the manganese content is reduced by irradiation, as predicted by the calculations and verified by the chemical analysis. The analyses also show a significant buildup of copper and considerable buildup of zinc and vanadium. The copper buildup is somewhat more than expected from the nickel while the increase in zinc is considerably higher than expected. Increased vanadium content possibly results from chromium undergoing a (n,γ) reaction followed by electron capture. The larger than predicted increase of the zinc content may be only apparent as a result of segregation or else be due to production of zinc by fast and epithermal neutrons which were not considered.

A further attempt was made to establish the compositional differences between the unirradiated and irradiated stainless steel. Microprobe analyses were performed on the matrix and across the grain boundaries of both unirradiated and irradiated stainless steel. A specially shielded Material Analysis Company "Model 450" electron microprobe at the Battelle-Columbus Hot-Cell Facility, was utilized for these analyses. A metallographic specimen from an irradiated specimen was used which had been tested at 600 C and showed a large ductility loss.

It was not possible to detect any zinc or vanadium in the irradiated sample. Copper could barely be detected above the high background radioactivity of the irradiated sample.

Hydrogen Analysis. Calculations were made to predict the amounts of hydrogen and helium that were produced in Type 347 stainless steel after a fast fluence of 2.1×10^{22} nvt (Table B-24). Hydrogen was considered important because large amounts of hydrogen are produced by (n, p) reactions and hydrogen has been found to embrittle metals. Therefore, it was decided to establish the effect of hydrogen (in the amounts produced by irradiation) on the elevated-temperature tensile properties of Type 347 stainless steel.

TABLE B-24. HYDROGEN AND HELIUM PRODUCTION BY NUCLEAR REACTIONS IN TYPE 347 STAINLESS STEEL DURING IRRADIATION^(a)

Element	Cross Section for (n, p) Reactions ^(b) , millibarns	Hydrogen Atoms per Gram of Stainless ^(c) , 10 ¹³	Cross Section for (n, a) Reactions ^(b) , millibarns	Helium Atoms per Gram of Stainless ^(c) , 10 ¹³
Manganese	0.4	158	0.13	52
Phosphorus	31	483	1.3	21
Sulphur	57	354	14	88
Silicon	3.8	1160	47	288
Nickel	76	169,000	0.59	1320
Chromium	1.8	7620	0.38	1640
Niobium	1.8	164	0.04	4
Iron	4.7	72,500	0.44	6870
Titanium	3.1	84	0.52	13
Copper	6.3	378	0.55	31
Nitrogen	--	--	41	111
Boron	--	--	3840×10^3 ^(d)	660 ^(d)
Total	--	251,901	--	11,098

(a) Only elements are included which contribute significantly to total hydrogen and helium production.

(b) Represents averaged cross section for fast neutrons, which takes isotopic abundance into account⁽¹⁵⁾.

(c) Based on a fast fluence of 2.1×10^{22} nvt.

(d) Values pertain to thermal neutrons.

Hydrogen is generally introduced into metals by cathodically charging them in an acid solution. However, very thin (~5 mil) sheet specimens have to be utilized to assure an even distribution of hydrogen because of the low charging temperatures and consequently low diffusion rates. Since tensile specimens with 0.16-in. diameters were to be utilized in this study, the hydrogen was introduced by annealing the specimens in a hydrogen atmosphere at elevated temperature. By utilizing the diffusion coefficients of hydrogen in austenitic stainless steel it was calculated that an equilibrium hydrogen concentration (~6 ppm by weight) at 950 C would be reached in 18 hr for the 0.16-in.-diameter gage section⁽¹⁶⁾. An anneal of 950 C for 18 hr is not expected to cause any measurable grain growth and thus will not change the properties of the stainless steel.

After annealing in hydrogen, the stainless steel specimens were quenched in water to retain the hydrogen in the specimens. The calculations given in Table B-24 indicate that about 4 ppm of hydrogen by weight should be produced in the stainless steel, as a result of (n,p) reactions. However, it was decided to place as much hydrogen into the stainless steel as practically possible without altering the properties of the steel. The factors which influenced this consideration were the uncertainties in (n,p) cross sections and the accuracy of hydrogen diffusion coefficient in stainless steel since an extrapolated value was used. To determine the hydrogen distribution in the specimens a 0.16-in.-diameter stainless steel rod was included with the tensile specimens during hydriding. Five successive layers were removed from the rod by machining, and these were analyzed for hydrogen by vacuum fusion techniques. The analysis showed that the hydrogen distribution in the hydrogen-impregnated sample was fairly uniform.

Tensile tests were performed on hydrogen-impregnated specimens and control specimens at room temperature 315, and 750 C. Results of the tensile tests are given in Table B-25. These tensile test results indicate that hydrogen has no measurable effect on the tensile properties of Type 347 stainless steel at any of the testing temperatures.

TABLE B-25. TENSILE PROPERTIES OF HYDRIDED^(a) AND UNHYDRIDED TYPE 347 STAINLESS STEEL

Test Tempera- ture, C	0.2 Percent Offset Yield		Ultimate Tensile		Total	
	Strength, 1000 psi		Strength, 1000 psi		Elongation, percent	
	Unhydrided	Hydrided	Unhydrided	Hydrided	Unhydrided	Hydrided
25	37.0	34.2	90.0	91.0	64.0	63.0
316	26.8	23.8	64.2	60.4	36.0	38.0
750	18.5	18.2	34.5	31.7	68.5	67.0

Table B-26 shows the hydrogen analysis of the stainless steel specimens after tensile testing. It can be seen that testing at room temperatures and 315 C does not result in any reduction of hydrogen content but that testing at 750 C restores the hydrogen level to that found normally in austenitic stainless steels.

TABLE B-26. HYDROGEN CONTENT OF TYPE 347
STAINLESS STEEL SPECIMENS

Material Condition	Hydrogen Content, ppm by weight (± 0.5 ppm)
As received	0.6, 1.0
Hydrided; tested at RT	4, 7, 6.5
Hydrided; tested at 315 C	6.2
Hydrided; tested at 750 C	0.9

It can be concluded from the test results that the extreme embrittlement that is observed in irradiated austenitic stainless steels at elevated temperature is not due to the hydrogen generated by (n, p) reactions during irradiation.

Helium Analysis. Selected specimens were sent to Atomics International for helium analysis. The analyses were obtained from approximately 1-g samples which were melted and vaporized in a tungsten helix coil. The helium released was mixed with a He^3 spike addition of known volume and was introduced into a mass spectrometer for measurement of the ratio of He^4 to He^3 . Analyses were performed in duplicate with excellent reproducibility, differences from the average being a maximum of 1 percent of the concentration values. In addition, mass spectrographic analyses were performed on irradiated and unirradiated samples of high-purity nickel, for which helium analyses also were available.

Results of these analyses are presented in Tables B-27 and B-28. Three observations concerning the data presented in Table B-27 should be noted. These are:

- (1) Large amounts of helium can be generated, the amount increasing exponentially with increasing neutron exposure.
- (2) The amounts are not predictable on the basis of known (n, α) reactions.
- (3) The amounts of helium, in excess of those predictable, are related to the presence of nickel in the irradiated materials.

TABLE B-27. NEUTRON EXPOSURE AND HELIUM CONTENT OF VARIOUS IRRADIATED ALLOYS

Alloy(a)	Fast Fluence (>1 Mev), 10 ²¹ nvt	Thermal Fluence, 10 ²¹ nvt	Helium Content, ppm (atomic)	
			Predicted	Measured
Nickel (99.999 percent)	1.4	2.6	6.3	77.3
Tenelon	1.5	2.7	6.1	7.9
UMCo-50	1.5	2.7	1.9	2.5
Type 348 stainless	3.3	3.7	10.8	23.5
Type 348 stainless	4.4	5.0	11.7	37.2
Type 347 stainless	21.0	23 - 42 ^(b)	18.0	917
Type 347 stainless	30.0	33 - 60 ^(b)	24.1	2016

(a) Composition (in w/o):

Tenelon: Fe-18Cr-14Mn-0.4N-0.1C.

UMCo-50: Co-27Cr-24Fe-0.07C.

Type 347 stainless: Fe-18Cr-10Ni-1.7Mn-0.7Nb-0.6Si-0.05C.

Type 347 stainless: Fe-18Cr-10Ni-1.7Mn-0.6Nb-0.5Si-0.04C.

(b) Estimated from fast fluence.

TABLE B-28. MASS SPECTROGRAPHIC ANALYSIS OF NICKEL

Isotopic Number	Element	Initial Content, ppm	Final Content, ppm
56	Fe	20	150
59	Ni(Co)	2	~5,000 - 10,000
63	Cu	1	500
65	Cu	0.5	30

The results of mass spectrographic analysis of the high-purity nickel specimen given in Table B-28 list only those elements which showed a measurable change after irradiation. The changes which are regarded as significant are the increases in level of elements of Mass 56 and 59.

The experimental helium analysis data also are plotted in Figure B-58 on a log-log scale. The straight line drawn through the data points has a slope of 2, showing a dependence of helium content on the square of the fluence.

FIGURE B-58. DEPENDENCE OF HELIUM CONTENT IN NICKEL-BEARING MATERIALS ON NEUTRON FLUENCE

The data of Weitman, et al. (17), who related the helium content to a two-step neutron reaction with nickel, also are summarized in this plot, averaging groups of data points, and exhibit a similar dependence. The displacement between the curves for the two sets of data is undoubtedly associated with differences in experimental and analytical techniques. It is important to note that Weitman, et al. (17), postulated an approach at relatively low fluence, to a linear dependence between helium content and fluence. However, when examined in the light of the higher fluence data of the present program, it is obvious that the helium data of Weitman, et al., also can be represented as following a dependence on the square of the fluence.

If a two-step reaction is considered for the production of helium from nickel, it is predicted that for high fluences or high combinations of $\phi \sigma_2 t$,

$$N_{\text{He}} = \sigma_1 N_x \phi t ,$$

and for low fluences or low combinations of $\phi \sigma_2 t$,

$$N_{\text{He}} = 1/2 \sigma_1 \sigma_2 N_x (\phi t)^2 ,$$

where

σ_1 = cross section for Reaction (1), which follows

σ_2 = cross section for Reaction (2), which follows

N_x = number of ^{58}Ni atoms originally present

ϕ = neutron flux.

The mass spectrographic data presented in Table B-28 can now be used to identify the specific reactions leading to the helium generation in nickel. These are shown below along with predicted and measured (in brackets) amounts generated of the various constituents in the reactions. Agreement between measured and predicted amounts of the constituents involved in the reactions is considered within the limits of accuracy for the analyses performed.

The cross section for Reaction (1) is taken from published data while the cross section for Reaction (2), in barns, is calculated from the nickel data shown in Table B-27. In making the calculation it was assumed that thermal neutrons were involved in both reactions; this, in view of the relatively significant cross section calculated, appears to be a valid assumption.

As indicated earlier, at some high fluence, the dependence of helium content on the square of the fluence will give way to a linear dependence. Using the cross section values given above it is found that deviations from the square dependence begin to become noticeable only when fluences of 10^{23} nvt are achieved. The beginning of this deviation is illustrated in Figure B-58. Consequently, for all practical ranges of neutron exposure the square dependence is observed. When exposures exceed a fluence of 10^{25} nvt the dependence becomes essentially linear.

Density Changes. Density measurements were made on specimens of Type 347 stainless steel irradiated at 50 C and also on specimens of Type 347 and Type 348 stainless steel irradiated at 400 and 650 C.

Irradiation of Type 347 stainless steel at 50 C to fast fluences as high as 2.2×10^{22} nvt did not cause any measurable density changes. Neither did annealing for 1 hr at 980 C. However, 1-hr anneal at 1350 C resulted in a 13 percent density decrease (Table B-29). The microstructure of the irradiated material that was annealed at 1350 C (Figure B-59) shows a large number of bubbles along the grain boundaries. These bubbles are believed to result from helium precipitation.

TABLE B-29. EFFECT OF ANNEALING ON THE DENSITY OF IRRADIATED TYPE 347 STAINLESS STEEL^(a)

Annealing Temperature ^(b) , C	Density Decrease ^(c) , percent
980	0
1200	1.5
1300	1.9
1350	13.0

(a) Specimens were irradiated at 50 C to a fast fluence of 2.2×10^{22} nvt.

(b) All anneals were for 1 hr.

(c) Measurements were performed by alternate weighing in air and CCl_4 . Accuracy of density measurements is ± 0.3 percent.

FIGURE B-59. MICROSTRUCTURE OF IRRADIATED (2.2×10^{22} NVT) TYPE 347 STAINLESS STEEL AFTER 1-HR ANNEAL AT 1350 C

It was also noted that grain size in the irradiated specimen increased only 50 percent while the grain size in an unirradiated specimen increased by a factor of 10 during the anneal at 1350 C. This implies that the helium bubbles are pinning the grain boundaries and preventing their growth.

Density measurements were performed on specimens of Type 347 and Type 348 stainless steel irradiated at 400 and 650 C, in both the as-irradiated condition and after annealing for 1 hr at 980 C. Results of these density measurements are summarized in Table B-30. The data indicate that irradiation at elevated temperatures causes a reduction in density which is restored by a post-irradiation anneal at 980 C. A satisfactory explanation for these observations is not available at the present time.

TABLE B-30. DENSITY OF IRRADIATED STAINLESS STEEL BEFORE AND AFTER ANNEALING FOR 1 HR AT 980 C

Material Condition	Density of Stainless Steel ^(a) , g per cm ³ (± 0.01)	
	Type 347	Type 348
Unirradiated control	7.91	7.92
Irradiated at 400 C	7.88	7.83
Annealed after 400 C irradiation	7.92	7.92
Irradiated at 650 C	7.85	7.88
Annealed after 650 C irradiation	7.92	7.92

(a) At least two specimens were measured for each condition.

Transmission Electron Microscopy. Specimens of Type 347 stainless steel that were irradiated at 50, 400, and 650 C were examined by transmission electron microscopy (TEM). These TEM observations were performed on specimens in the as-irradiated condition and after annealing for 1 hr at 980 C. The specimens had been tested in tension before the observations by TEM were made and the results of the tests are related to the microstructures observed in Figure B-60.

The predominant features of the microstructure of the unirradiated specimens include dislocation network or cell structure which is approximately 0.5μ in size, the general occurrence of twins, and a distribution of inclusions. In addition, slip traces and stacking faults are observed.

TEM results from specimens that were tested at 525 and 600 C after irradiation at 50 C did not reveal the presence of any voids or bubbles. Thus, the helium in these samples is either in solution or the bubbles are too small to be readily observed. At 600 C, this structure coincides with the lowest ductility of any specimen tested.

Examination of the irradiated and annealed material by TEM revealed a general distribution of 200 to 500-A-diameter bubbles. It was concluded that these

FIGURE B-60. RESPONSE OF DIFFERENT UNIRRADIATED AND IRRADIATED TYPE 347 STAINLESS STEEL MICROSTRUCTURES TO TENSILE TESTING AT ELEVATED TEMPERATURES

were helium bubbles rather than voids due to vacancy agglomeration, because vacancy agglomeration is not expected to occur at an irradiation temperature of 50 C and also no bubbles were observed in the as-irradiated condition. Observations by others have shown that voids due to vacancies are completely removed from irradiated stainless steel by annealing at 815 C⁽¹⁸⁾, while helium bubbles cannot be removed by annealing and grow with increasing annealing temperature⁽¹⁹⁾. These helium bubbles were fairly uniformly distributed throughout the material, but with a tendency to be located at grain and twin boundaries. The appearance of helium bubbles is accompanied by an increase in ductility. Thus, while helium may be responsible for the elevated-temperature embrittlement observed in stainless steels it appears that small or unresolved bubbles or clusters are more effective in embrittlement than are large helium bubbles.

The occurrence of twins, slip traces, stacking faults, and inclusions was not significantly different in either the irradiated or the unirradiated material. In addition to the helium bubble observations, significant differences in dislocation cell structure were observed. The cell structure observed in the unirradiated specimen was not observed at all in the as-irradiated specimens tested at 525 and 600 C. However, cell structure was present in the irradiated specimen which was annealed for 1 hr at 980 C and then tested at 600 C.

Cell structure forms when screw dislocations cross-slip from their original slip planes. Formation of cell structure is favored by increasing strain, and the cell structure becomes more distinct and the cells larger as the testing temperature becomes higher. Cross-slip, and consequently cell formation, is hampered by precipitates⁽²⁰⁾. Thus, it might be expected that the irradiation-induced loops, bubbles, or other defects could interfere with cross-slip and prevent cell formation. Also it has been found that formation of a cell structure decreases with decreasing stacking fault energy of the material. The irradiation-induced solid transmutation products, particularly after the long-time irradiation, may reduce the stacking fault energy and thus minimize the tendency toward formation of the cell structure. However, this effect must be minor since some cell formation still occurs in the specimen annealed at 980 C and then tested at 600 C.

Discussion of Results

It is quite apparent from Figures B-35 through B-39 that the degree of radiation-induced changes in tensile properties is dependent on the irradiation temperature. The changes in properties are a maximum for irradiation at 400 C, intermediate for irradiation at 50 C, and smallest for irradiation at 650 C. Bloom, et al.⁽²¹⁾, have shown that the change in yield strength induced by irradiation is due to the size and distribution of the defect clusters that were produced during irradiation. Irradiation at low temperatures results in a large density of small defects. Irradiation at higher temperatures results in lower defect densities but larger individual defects. The maximum increase in yield strength occurs at about 150 C and the yield strength decreases slowly from 150 C to 300 C, above which it decreases rapidly. The authors concluded that there was an optimum defect size for maximum strengthening, but that the amount of strengthening also

depends on the defect density. Recent studies have shown that strengthening in irradiated stainless steel is also caused by helium bubbles⁽²²⁾.

At the irradiation temperature of 400 C, defect clusters are expected to be somewhat larger than the optimum size for strengthening and consequently the strengthening from defect clusters is not expected to be a maximum. However, at an irradiation temperature of 400 C it is expected that small helium bubbles will form and make a significant contribution to strength. At the irradiation temperature of 650 C the defect clusters would be annealed out during irradiation. This is borne out by the fact that irradiation at 650 C does not result in significant increases of yield strength.

At low testing temperatures (<400 C) irradiation affects the tensile elongation of stainless steel, principally as a result of the presence of defect clusters that strengthen the material with an associated reduction in ductility. Helium bubbles may also contribute to these changes in strength and ductility at low temperatures as observed for specimens irradiated at 400 C. The development of an extremely fine array of bubbles at the grain boundary as a result of irradiation at the temperature was revealed by fractography. The array of bubbles apparently presented a path for fracture at very low total strains.

The increase in ductility and the decrease in strength with continuing irradiation at 50 C, after an initial saturation effect, is probably due to a softening mechanism which has been observed by other investigators⁽²³⁾. It could be theorized that as irradiation proceeds, such a large number of small defect clusters (due to low irradiation temperature) are formed that they overlap each other. Consequently, they interact with each other, thereby producing fewer but larger clusters. The combination of larger but fewer defect clusters provides less resistance to dislocation movement, and ductility is increased and strength decreased.

At higher testing temperatures (above about 450 to 550 C depending on material), irradiation causes a drastic reduction in ductility (Figures B-18, B-24, B-26, B-28, B-29, B-36, B-37, and B-38). This reduction in ductility has generally been attributed to helium^(11,12). Barnes⁽¹¹⁾ has suggested that helium coalesces into bubbles at grain boundaries due to stress. Once the bubble starts growing less stress is required for continued growth of the bubble. The final bubbles would not necessarily be spherical, and the bubbles could be described as fissures. The bubbles (or fissures) would continue to grow with continued stress until they came together and caused fracture. Kramer has suggested that moving dislocations actually sweep helium atoms from the matrix to the helium bubbles at the grain boundaries⁽²⁴⁾. This mechanism is not consistent with the TEM observations reported here that the growth of helium bubbles to a visible size, as a result of annealing, is accompanied by an increase in tensile ductility.

It has been also suggested that helium may cause intergranular fracture in stainless steel by forming small bubbles within the grain. These small bubbles impede dislocation movement and consequently strengthen the grains⁽²⁵⁾. Although the grain boundaries are not directly affected they become weaker relative to the grains, and consequently failure occurs in the grain boundaries. It is

quite possible that the mechanism proposed is applicable to the as-irradiated material since in that case the strength is higher and the ductility lower. However, by observing the stress-strain diagrams of materials in the irradiated and annealed condition (Figures B-25, B-27, B-40, and B-41), it can be seen that the work-hardening characteristics of the irradiated and annealed material are similar to those of the unirradiated material. Thus, it does not appear that, at elevated temperatures where helium bubble growth can occur, this mechanism could be effective.

The more probable mechanism is similar to that suggested by Rhines and Wray⁽²⁶⁾. In unirradiated metals and alloys, a ductility minimum is found to occur at a temperature somewhat below the recrystallization temperature. The ductility minimum is presumed to occur with increasing temperature when stress causes grain-boundary shearing and the formation of fissures in the grain boundaries. These grain-boundary fissures then promote intergranular fracture. Grain-boundary migration tends to minimize the effect of these fissures by continuously healing them. As the temperature is increased and the migration of the grain boundaries becomes sufficiently rapid, the fissures are healed quickly such that the ductility increases again with increasing temperature. In the case of irradiated stainless steel helium bubbles pin the grain boundaries and thus prevent their migration. Grain-boundary pinning was observed in highly irradiated stainless steel in this program; a 1-hr anneal at 1350 C caused only a 50 percent increase in grain size, while unirradiated stainless steel underwent an eightfold increase in grain size as a result of an identical heat treatment. Since only minimal migration occurs, the cracks which result from grain-boundary shearing are not healed and consequently fracture occurs without significant elongation. It has been shown that helium causes grain-boundary pinning in aluminum⁽²⁷⁾, but no comparable data are available for its behavior in stainless steel.

Metallographic examinations indicated that although the fracture of the irradiated material is intergranular there are very few wedge-type cracks present in the material (Figures B-21, B-31, B-32, B-33, and B-43). This seems to indicate that the ease of crack propagation is considerably enhanced by irradiation, and as soon as a crack is formed it propagates to fracture. The enhancement in ease of crack propagation results from grain-boundary pinning which prevents the healing of fissures. This also leads to lower ultimate strengths (Figures B-25, B-27, B-40, and B-41) since fracture occurs as soon as a crack is formed.

The presence of nickel is not necessary for elevated-temperature embrittlement. However, alloys which contain nickel are embrittled to a greater extent because of the high helium generation rates by thermal-neutron reaction with nickel. Böhm and Hank have suggested that irradiation increases the tendency toward intergranular fracture at elevated temperature⁽²⁸⁾. Thus it seems that the alloys which would be the most resistant to irradiation-induced elevated-temperature embrittlement are the ones which have a tendency toward transgranular fracture and contain the minimal amount of nickel.

At elevated temperatures both the stress rupture life and fatigue life are reduced as a result of irradiation-induced elevated-temperature embrittlement.

This is apparent since intergranular cracks are not observed in the fracture area, as is the case for unirradiated material, indicating that the first crack formed propagates to failure of the specimen.

Conclusions

Irradiation-induced changes of tensile properties in austenitic stainless steel are dependent on irradiation temperature. For low-temperature tests (< 450 C) the changes in mechanical properties are a maximum for irradiation at 400 C, intermediate for irradiation at 50 C, and smallest for irradiation at 650 C.

Irradiation softening takes place in stainless steel irradiated at 50 C to fast fluences of 3×10^{22} nvt. This softening is believed to be due to coalescence of small defects into larger defects.

Irradiation-induced elevated-temperature embrittlement was found to start at testing temperatures of 450 to 550 C (depending on material) and at 750 C was found to be independent of irradiation temperature.

Postirradiation annealing resulted in almost complete restoration of pre-irradiation tensile properties for testing temperatures of 500 C and below. However, it did not remove the effects of elevated-temperature embrittlement as manifested by reductions in ultimate strength and ductility.

Testing of as-irradiated specimens at temperatures above 500 C resulted in intergranular fracture with evidence of helium bubbles at grain-boundary facets.

The mechanism of irradiation-induced elevated-temperature embrittlement is believed to be an aggravation of the ductility minimum observed in tensile tests of most face-centered-cubic alloys.

The ease of crack propagation is found to be significantly enhanced by irradiation.

Large amounts of helium are produced by thermal neutrons from nickel by the two-step process: $\text{Ni}^{58} (n, \gamma) \text{Ni}^{59}$ and $\text{Ni}^{59} (n, \alpha) \text{Fe}^{56}$. Stainless steel in which the helium is distributed in unresolvable form is more brittle than stainless steel in which the helium is contained in resolvable (500 to 2000 Å) bubbles.

The presence of nickel is not necessary for elevated-temperature embrittlement to occur, but alloys containing nickel will be embrittled to a greater extent than nonnickel alloys because of the large amounts of helium generated from nickel by thermal neutrons.

The tendency for intergranular fracture at elevated temperature in all alloys tested was promoted by irradiation. In the case of nickel the fracture mode was changed from transgranular to intergranular.

High thermal fluences were found to produce significant amounts of zinc from copper by transmutations. Also 60 percent of the manganese was burned out by a thermal fluence of 4×10^{22} nvt. However, these compositional changes do not cause significant changes in mechanical properties.

The amount of hydrogen produced (4 ppm by weight) by a fast fluence of 2.1×10^{22} nvt was not found to influence the mechanical properties of stainless steel at elevated temperatures.

Irradiation (5.7×10^{21} nvt) was found to decrease the fracture energy of Charpy V-notch specimens at temperatures of -196 C to 315 C. However, since the Izod specimens did not fracture, irradiation was found to increase the absorbed energy during testing.

The room-temperature fatigue life of Type 347 stainless steel was enhanced by irradiation (5.5×10^{21} nvt) at low total strains. At fast fluences of 1.1×10^{22} nvt and 1.6×10^{22} nvt the fatigue life was lower for all strains.

At 650 C, irradiation (2.1×10^{22} nvt) reduces the fatigue life by a factor of about six due to elevated-temperature embrittlement.

Stress rupture life of irradiated (2.1×10^{22} nvt) stainless steel is reduced by a factor of six to ten as a result of elevated-temperature embrittlement.

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Effect of Fast-Neutron Irradiation (EBR-II) on the Mechanical Properties of Austenitic Stainless Steel

The development of Liquid-Metal Cooled Fast-Breeder Reactors (LMFBR's) requires that the effect of neutron irradiation on the mechanical properties of austenitic stainless steels, which are expected to be the main structural material in LMFBR's, be well known. Furthermore, because neutron spectrum varies with location in a reactor, it is desirable to know how the neutron damage varies with spectrum. To determine the effect of a predominantly fast-flux spectrum on tensile properties, specimens of Type 347 and Type 348 stainless steel were irradiated in the EBR-II. Specimens of the same heats of the stainless steels were also irradiated in the more thermalized spectrum of the ETR at identical temperatures (see the previous section). It was hoped to obtain a comparison of the tensile property changes resulting from different neutron spectrums.

Material Characteristics

Specimens were irradiated in a capsule in the EBR-II. The capsule contained specimens of Type 347 and Type 348 stainless steel as well as specimens of Tenelon, UMC0-50, and Haynes Stellite 25. However, the specimens of Tenelon, UMC0-50, and Haynes Stellite 25 were not tested because at the time the irradiation was completed they were no longer of interest as potential structural materials. Consequently, only the material characteristics of the stainless steels will be discussed here.

The chemical composition of the stainless steels is given in Table B-31. The main difference between Type 347 and Type 348 stainless steel is in the amount of allowable tantalum. Specifications for Type 347 stainless steel require that the niobium + tantalum content should be ten times the carbon content. For Type 348 stainless steel the tantalum content is limited to 0.10 w/o; also, the cobalt content is limited to 0.20 w/o. These limitations are placed on the steel because Type 348 stainless steel is intended for use in nuclear reactors and both tantalum and cobalt become highly radioactive after reaction with neutrons and therefore a health hazard.

Both of the stainless steels were hot rolled, solution annealed, and cold straightened. The average grain sizes for the Type 347 and Type 348 stainless steels were 12 μ and 10 μ , respectively. A typical microstructure of the Type 347 stainless steel is shown in Figure B-61a while the microstructure of Type 348 stainless steel is illustrated in Figure B-61b.

Specimens were machined from the centers of two 5/8-in. -diameter bars. The dimensions of the Type 347 stainless steel specimens are given in Figure B-62a, while Figure B-62b illustrates the dimensions of the Type 348 stainless steel specimens. Specimens with button heads were utilized because these specimens could be loaded more easily into tensile grips.

TABLE B-31. CHEMICAL ANALYSIS OF TYPE 347 AND TYPE 348
STAINLESS STEEL USED IN THE
IRRADIATION STUDIES^(a)

Element	Analysis, w/o	
	Type 347 Stainless	Type 348 Stainless
Iron	Bal	Bal
Chromium	17.9	17.5
Nickel	10.4	9.67
Manganese	1.72	1.67
Silicon	0.65	0.51
Niobium	0.67	0.58
Copper	0.30	0.06
Molybdenum	0.20	0.06
Cobalt	0.19	0.18
Titanium	0.10	NA
Carbon	0.057	0.04
Phosphorus	0.024	0.014
Sulfur	0.016	0.009
Tantalum	0.01	0.03
Boron	0.00005	0.0008
Nitrogen	0.030	0.037

(a) NA = not analyzed.

250X

5A396

a. Type 347 Stainless Steel

250X

OC585

b. Type 348 Stainless Steel

FIGURE B-61. MICROSTRUCTURES OF THE TYPES 347 AND 348 STAINLESS STEEL USED IN THE IRRADIATION STUDIES

a. Short Specimen

b. Long Specimen

FIGURE B-62. DESIGN OF TENSILE SPECIMENS THAT WERE IRRADIATED AND TESTED

Irradiation Conditions

The specimens were placed in individual specimen holders with four specimens to each specimen holder. Sleeves were placed around the gage sections of the tensile specimens to assure contact so that heat flow would occur. Figure B-63 is a schematic drawing of the specimens in the holder. The individual specimen holders were placed in one of the tubes which make up the subassembly of seven tubes.

A gas gap of specified size was left between the specimen holders and the ID of the subassembly tube. The size of the gas gap was selected to achieve the desired irradiation temperatures and was based on estimates of gamma heating for the specific location. The gamma heating study on which the estimates were based and the recommended gas gaps were determined by ORNL personnel.

Figure B-64 shows the relative axial location of the specimen holders in Subassembly X0-34 which was irradiated in Row 7 of the EBR-II. The specimens contained in each specimen holder are enumerated in Table B-32.

Although SiC temperature sensors were incorporated in selected specimen holders they were not analyzed because of the lowering of program priority. Fluence of the specimens was measured by ORNL personnel to be 3.9×10^{22} nvt ($E > 0.1$ Mev) at the EBR-II midplane. Fluence values for other specimens were obtained by normalizing the fluence value to the axial flux profile in Row 7 locations.

Tensile Testing

Tensile testing was done on an Instron testing machine. Before testing, the specimens were held at the testing temperature for 30 min to assure thermal equilibrium. The strain rates varied from 0.005 to 0.5 per min.

Specimens Irradiated at 400 C. Tensile tests were performed on the specimens irradiated at 400 C at temperatures ranging from room temperature to 750 C. Results of these tests are summarized in Table B-33. The uniform and total elongations of the specimens irradiated at 400 C are shown in Figures B-65 and B-66. For comparison, the data for unirradiated specimens and for specimens irradiated in the ETR to fast fluence levels similar to those experienced in the EBR-II are also included. From this comparison it is apparent that irradiation in the ETR is more detrimental to ductility at all testing temperatures. The post-irradiation ductility of the Type 348 stainless steel is slightly below that of the Type 347 stainless steel, although differences are not major.

TABLE B-32. LOADING SEQUENCE AND SPECIMEN INFORMATION FOR
TUBE IN SUBASSEMBLY XO-34

Compartment Identification	Specimen Quantity	Material	Type of Specimens	Irradiation Temperature, C	Overall Length, in.
1	4	Type 347 stainless	S-tensile	400	1.997
2	4	Type 347 stainless	Fatigue	400	2.302
3	2	UMCo-50	L-tensile	400	3.003
	2	Haynes Stellite 25	L-tensile	400	--
4	4	--	AI specimens	400	--
5	4	Tenelon	L-tensile	650	3.001
6	3	Type 348 stainless	L-tensile	650	2.997
	1	SiC	Temperature sensor	650	--
7	3	Type 347 stainless	S-tensile	650	2.002
	1	SiC	Temperature sensor	650	--
8	4	Type 347 stainless	S-tensile	400	2.001
9	4	Type 347 stainless	S-tensile	400	2.003
10	4	Type 348 stainless	L-tensile	400	3.000
11	4	Tenelon	L-tensile	400	3.001
12	4	Type 347 stainless	S-tensile	400	1.995
13	4	UMCo-50	L-tensile	400	3.000
14	4	Type 347 stainless	Tubes	400	4.401
15	4	Type 347 stainless	Tubes	400	4.397
16	4	Tenelon	L-tensile	400	3.003
17	4	Type 347 stainless	Tubes	400	4.396

TABLE B-33. TENSILE PROPERTIES OF TYPE 347 AND TYPE 348 STAINLESS STEEL BEFORE AND AFTER IRRADIATION IN THE EBR-II

Stainless Steel Type	Testing Temperature, C	Fast Fluence, 10 ²² nvt	Strain Rate, in. /in. /min	0.2 Percent	Ultimate Strength, 10 ³ psi	Elongation, percent	
				Offset Yield Strength, 10 ³ psi		Uniform	Total
<u>Unirradiated Material</u>							
347	Room	0	0.005	37.0	90.0	52.0	64.0
	315	0	0.005	26.8	64.2	31.9	36.0
	500	0	0.005	24.0	47.5	22.4	26.6
	600	0	0.005	23.0	40.5	17.8	27.5
	750	0	0.005	22.0	33.0	16.0	26
348	Room	0	0.005	36.2	93.0	58	61.4
	315	0	0.005	27.8	62.8	26.7	29.4
	500	0	0.005	23.8	53.2	22.7	25.2
	600	0	0.005	23.4	49.1	21.5	30.3
	750	0	0.005	19.0	27.3	4	35
<u>Irradiated at 400 C</u>							
347	25	3.9	0.005	92.0	124	30.4	37.8
	315	3.9	0.005	82.5	98	10.9	16.9
	500	3.5	0.5	73.0	82.0	7.9	14.3
	500	3.5	0.005	73.0	83.9	9.0	14.1
	600	3.5	0.5	58.2	71.2	7.1	11.7
	600	3.9	0.005	61.6	72.6	6.5	8.3
	750	3.5	0.5	43.5	47.0	6.0	8.2
	750	3.5	0.05	36.3	40.1	4.2	5.6
	750	3.9	0.005	27.1	28.6	3.2	4.2
348	315	2.7	0.005	89.6	102.0	6.6	10.5
	500	2.7	0.005	71.8	81.0	5.1	7.8
	600	2.7	0.005	60.9	66.0	3.6	5.2
<u>Irradiated at 650 C</u>							
348	315	3.4	0.005	36.4	66.8	22.3	25.7
	500	3.4	0.005	31.7	56.1	22.4	25.4
347	600	3.9	0.5	41.0	56.3	15.8	19.8
	600	3.9	0.005	40.5	47.5	7.5	8.2
348	750	3.4	0.005	16.8	18.9	7.2	14.5

FIGURE B-65. UNIFORM ELONGATION OF TYPE 347 STAINLESS STEEL IRRADIATED AT 400 C

FIGURE B-66. TOTAL ELONGATION OF TYPE 347 STAINLESS STEEL IRRADIATED AT 400 C

Figures B-67 and B-68 show the yield strength and ultimate strength of irradiated Type 347 stainless steel as a function of testing temperature. For comparison, the strengths of unirradiated specimens, as well as the strength of specimens irradiated in the ETR to similar fast fluence levels, are also included. From this comparison it is apparent that irradiation in the ETR causes larger increases in strength than irradiation in the EBR-II. The irradiation-induced strength increases of Type 348 stainless steel were similar to those of Type 347 stainless steel.

The effect of strain rate on the tensile properties of irradiated Type 347 stainless steel also was investigated. Results of these tensile tests are given in Table B-33. These test results showed that at 500 C the tensile properties were essentially independent of the strain rate, while the lower strain rate resulted in somewhat lower ductilities at 600 C. At 750 C, the irradiated material showed significant strain-rate dependence, lower strength and ductility being associated with decreasing strain rate.

Metallographic examinations were performed on selected specimens which had been irradiated in the EBR-II. Figure B-69 compares the fracture areas of Type 347 stainless steel specimens that were irradiated at 400 C and then tested at 600 C at different strain rates. The higher strain rate of 0.5 per min resulted in somewhat higher ductility, but the fracture appearance is almost identical. The fracture in both cases was intergranular, with very little necking taking place.

Figure B-70 illustrates the fracture area of a specimen that was irradiated at 400 C and tested at 315 C. This testing temperature was of interest because it was not known for certain whether the fracture at this temperature was intergranular or transgranular for an irradiated specimen. From the metallography it is apparent that the fracture was transgranular, with considerable necking taking place for both the unirradiated and irradiated conditions.

Specimens Irradiated at 650 C. The effect of irradiation at 650 C on tensile properties is much less pronounced than is irradiation at 400 C. At the higher irradiation temperature partial recovery of irradiation damage processes accompany irradiation. However, noticeable increases in strength and decreases in ductility are observed (see Table B-33). Yield strength and total elongation values for Type 348 stainless steel are shown plotted in Figures B-71 and B-72, respectively, for comparison with data for unirradiated material and for specimens irradiated in the ETR. The EBR-II values are intermediate to the unirradiated and ETR-irradiated property values. Thus, as for irradiation at 400 C, the more thermalized spectrum of the ETR produces greater changes in mechanical properties than does the harder EBR-II spectrum for essentially equivalent fast fluence exposures.

The Type 347 stainless steel specimens tested at 600 C exhibited unusually large strength increases and ductility decreases. This behavior may reflect an aging phenomenon during either irradiation or testing.

FIGURE B-67. YIELD STRENGTH OF TYPE 347 STAINLESS STEEL IRRADIATED AT 400 C

FIGURE B-68. ULTIMATE STRENGTH OF TYPE 347 STAINLESS STEEL IRRADIATED AT 400 C

100X

HC25101

a. Strain Rate of 0.5 per Min

100X

HC25091

b. Strain Rate of 0.005 per Min

FIGURE B-69. FRACTURE APPEARANCE OF IRRADIATED TYPE 347 STAINLESS STEEL SPECIMENS TESTED AT 600 C

100X

4F930

a. Unirradiated

100X

HC25111

b. Irradiated at 400 C

FIGURE B-70. FRACTURE APPEARANCE OF UNIRRADIATED AND IRRADIATED
TYPE 347 STAINLESS STEEL TESTED AT 315 C

FIGURE B-71. YIELD STRENGTH OF TYPE 348 STAINLESS STEEL IRRADIATED AT 650 C

FIGURE B-72. TOTAL ELONGATION OF TYPE 348 STAINLESS STEEL IRRADIATED AT 650 C

Irradiated and Annealed Specimens. Selected tensile specimens were annealed for 1 hr at 980 C after irradiation. These specimens were then tested in tension at various temperatures; the results are given in Table B-34. Both the preirradiation strength and ductility were restored by the anneal if the testing temperature was 600 C or below. However, at a testing temperature of 750 C both the strength and ductility of the irradiated and annealed material were lower than those of the unirradiated material. Material irradiated at 650 C was found to exhibit lower ductility when annealed and then tested at 750 C than material irradiated at 400 C and similarly annealed and tested. The difference may be due to the difference in fluence exposure level and cannot be related directly to difference in temperature of irradiation.

Density Measurements

Immersion density measurements were made on unirradiated, irradiated, and irradiated and annealed specimens; results are summarized in Table B-35. It appears that irradiation at 400 C causes measurable reductions in density while irradiation at 650 C does not; this is consistent with the behavior of other austenitic stainless steels. Postirradiation annealing for 1 hr at 980 C restored the preirradiation density for specimens irradiated at 400 and 650 C. It can therefore be assumed that the irradiation-induced density decreases were due primarily to vacancy clusters which were removed by the high-temperature anneal.⁽¹⁾

Helium Analysis

Helium analyses were performed on the irradiated Type 347 and Type 348 specimens at Atomics International. The helium contents were found to be 3.5 and 2.5 ppm (atomic) for Type 347 and Type 348 stainless steel, respectively. McElroy and Farrar have shown that the expected helium generation from stainless steel irradiated in the EBR-II is dependent on the impurity level of the material as well as the radial and axial locations⁽²⁾. Consequently, only estimates of helium generation in stainless steel irradiated in the EBR-II can be made. The analyzed helium contents fall within the approximate ranges of expected helium generation.

Conclusions

On the basis of testing of irradiated specimens of Type 347 and Type 348 stainless steel, a number of conclusions can be drawn.

Irradiation in the EBR-II at 400 and 650 C results in strength increases and ductility decreases which are most pronounced for the lower irradiation temperature.

TABLE B-34. TENSILE PROPERTIES OF IRRADIATED STAINLESS STEEL
AFTER ANNEALING FOR 1 HR AT 980 C

Stainless Steel Type	Irradiation Temperature, C	Testing Temperature, C	Fast Fluence, 10^{22} nvt	0.2 Percent Offset Yield Strength, 10^3 psi	Ultimate Strength, 10^3 psi	Elongation, percent	
						Uniform	Total
347	400	Room	0.1	40.6	91.2	60.5	75.2
347	400	315	0.1	27.7	65.0	31.8	37.6
347	400	500	0.1	27.1	60.0	28.9	37.3
348	400	600	2.7	23.2	48.2	22.5	35.9
347	400	750	0.1	17.8	27.7	9.4	12.5
347	650	750	3.9	21.2	26.0	6.2	8.0

TABLE B-35. EFFECT OF IRRADIATION AND POSTIRRADIATION ANNEALING
ON THE DENSITY OF AUSTENITIC STAINLESS STEEL

Stainless Steel Type	Fast Fluence, 10^{22} nvt	Irradiation Temperature, C	Density of Material in Indicated Condition, g per cm^3		Decrease in Density Due to Irradiation, percent	Density of Irradiated Material After Annealing for 1 Hr at 980 C, g per cm^3
			Unirradiated	Irradiated		
347	0.1	400	7.915	7.913	0.02	7.915
	2.7 to 3.9	400	7.915	7.890	0.31	7.918
	2.7 to 3.9	650	7.915	7.908	0.09	7.912
348	2.7 to 3.9	400	7.919	7.862	0.71	7.921
	2.7 to 3.9	650	7.919	7.914	0.06	--

There is little difference in the tensile properties of Type 347 and Type 348 stainless steel before or after irradiation.

Changes in properties, strength and ductility, are greater as a result of irradiation to similar fast-fluence levels in the ETR than in the EBR-II. The difference is most pronounced after irradiation at 400 C but is also evident after irradiation at 650 C.

The most obvious difference resulting from irradiation in the EBR-II as compared with the ETR is in the amount of helium generated in the stainless steel. For equivalent fast-fluence exposures ($\sim 3 \times 10^{22}$ nvt), helium generation in the EBR-II irradiations was about 3 ppm while stainless steel irradiated in the ETR analyzed at 2000 ppm. The reduced ductility accompanying irradiation in the ETR, in particular, is believed associated with helium grain-boundary concentrations.

Irradiation at 400 C resulted in significant density decreases for both stainless steels, while density after irradiation at 650 C was essentially unchanged. The density change is recovered by annealing at 980 C. This behavior is consistent with void formation behavior as a result of vacancy clustering and precipitation that is observed for Types 304 and 316 stainless steels. Peak density changes occurred at the lower irradiation temperature and not at 650 C where increased vacancy-interstitial mobility leads to annihilation of the vacancy clusters responsible for the density decreases.

The tensile properties of irradiated stainless steel are insensitive to strain rate at a test temperature of 500 C. However, at 750 C both strength and ductility are decreased by a decrease in strain rate.

- (1) Brager, H. R., and Robbins, R. E., "Stabilized Cavities in Irradiated Austenitic Stainless Steel", Trans. AIME, 242, 2010 (1968).
- (2) McElroy, W. N., and Farrar, H., "Helium Production in Stainless Steel and Its Constituents as Related to LMFBR Development Programs", HEDL-SA-193 (June, 1971).

RESPONSE OF DIFFERENT MICROSTRUCTURES TO IRRADIATION-INDUCED EMBRITTLEMENT AT ELEVATED TEMPERATURES

M. Kangilaski, A. A. Bauer, and J. W. Sheckherd

This effort was aimed at reductions in irradiation embrittlement and in fast-neutron-induced void formation by microstructural control. Two approaches were taken. In one, a series of specimens was prepared for irradiation in the EBR-II in order to investigate the role of grain boundaries and of grain-boundary orientation. This approach recognizes that elevated-temperature embrittlement is associated with intercrystalline cracking and that cracks develop on grain boundaries oriented perpendicular to the applied stress. The specimens are presently being irradiated and are to be examined by ORNL. The second approach involved shock hardening as a means of both strengthening stainless steel and

producing a dislocation substructure which will act as a vacancy sink to prevent the formation of voids. The mechanical and impact properties of shock-hardened material studied as part of this effort are summarized in the pages that follow.

Mechanical Properties of Shock-Strengthened Austenitic Stainless Steel

The results of this study were published in June, 1971, as a topical report, BMI-1909, "Mechanical Properties of Shock-Strengthened Austenitic Stainless Steel", and details of the work may be found therein. The conclusions reached as a result of the study are given here.

Tests were carried out to explore shock-strengthening parameters and how these shocking parameters influenced the mechanical properties of Types 304 and 316 stainless steel. The testing performed was sufficient to establish certain trends:

- (1) When a shock wave is passed through a material the yield strength and proportional limit are significantly increased, the ultimate strength is moderately increased, and ductility is generally decreased, although an intermediate increase in ductility with increasing shocking pressure was noted in material shock strengthened by means of a flyer plate.
- (2) The magnitude of the change in tensile properties increases with shocking pressure and holds for all testing temperatures.
- (3) Shock strengthening by contact explosives results in larger changes in mechanical properties than shock strengthening by flyer plate at equivalent pressures. The larger effect is attributed to the longer shock duration when contact explosives are used.
- (4) With an increase in shocking temperature (at equivalent shocking pressures), small changes are produced in room-temperature tensile properties and larger changes are produced in elevated-temperature tensile properties. This trend is attributed to the formation of a defect structure which is more stable at elevated temperatures.
- (5) The microstructure of shock-strengthened austenitic stainless steel exhibits fine striations, as observed by light microscopy. Transmission electron microscopy reveals a high dislocation density and the presence of mechanical twins. The density of both the twins, or striations, and dislocations increases with shocking pressure.
- (6) Annealing of the shock-strengthened material results in the disappearance of the defect structure and the changes in

mechanical properties, provided that the annealing temperature is high enough. A 1-hr anneal at 900 C results in complete removal of the defect structure.

- (7) Testing of shock-strengthened specimens annealed at 750 C for 960 hr indicates that some of the shock-induced changes in tensile properties are removed. However, the yield strength of the shocked and annealed material was found to be 41,400 psi at 650 C compared to 23,000 psi for the unshocked material.
- (8) Shock strengthening was found to reduce the minimum creep rate of Type 316 stainless steel by a factor of about 100 at 650 C for stresses ranging from 25,000 to 35,000 psi; the minimum creep rate of Type 304 stainless steel was reduced by a factor of 1000 at 650 C for stresses ranging from 25,000 to 35,000 psi.
- (9) The use of contact explosives to shock strengthen material at a pressure of 80 kbar resulted in creep rates at 650 C about equivalent to those for material shock strengthened at 320 kbar by a flyer plate.

Impact Properties of Shock-Strengthened Austenitic Stainless Steel

This section discusses the dynamic strength properties of shock-strengthened Type 316 stainless steel as determined by instrumented Charpy impact tests. Tensile and creep properties of the same material shocked under identical conditions are reported in BMI-1909⁽¹⁾. That report also includes a thorough characterization of the shocked material by light microscopy, transmission electron microscopy, and X-ray diffraction.

Shocking Conditions

The chemical compositions of the Type 316 stainless steels which were used in this study are given in Table B-36. For shock strengthening, 6 by 4 by 0.5-in. plates were used. The plates had been hot rolled and then solution annealed. Grain sizes for the different plates are also given in Table B-36.

The Type 316 stainless steel plates were shock strengthened by use of a flyer plate which is driven explosively to produce a shock wave in the material by its impact. By adjustment of the mass ratio of the explosive charge to flyer plate a wide range of pressures is attainable. The shock pressure pulse imparted to the material approximates a square wave, in the case of flyer plates, of the same or lower shock impedance than the material to be shocked. In the case of a flyer plate impacting a material of lower shock impedance, the initial pressure pulse is relieved in a series of pressure steps. The duration of the square, or

initial, pressure pulse in the impacted material is governed by the thickness of flyer plate employed and is given approximately by twice the flyer plate thickness divided by the shock-wave velocity in the flyer plate. The peak pressure developed is determined by the flyer-plate impact velocity and the Hugoniot (dynamic pressure-volume) relations of the flyer plate and shocked materials. The peak pressure is maintained (approximately) as the shock pulse progresses through the material until the square top on the pressure pulse has been eroded by the faster traveling rarification wave which terminates the pressure pulse. This condition occurs generally at about four to seven flyer plate thicknesses for impacts involving materials having similar Hugoniot properties. The peak pressure then attenuates in the manner characteristic of a triangular wave, which the square wave has now become. In general, attenuation of a wave generated by a thin flyer plate will be more rapid than from a thick one, with the rate of pressure attenuation decreasing with increasing distance of travel. With regard to hardening effects, the primary variable is the peak shock pressure, although a smaller dependence on the pressure pulse duration has also been noted for some materials.

TABLE B-36. COMPOSITION AND GRAIN SIZE OF THE TYPE 316 STAINLESS STEEL PLATES UTILIZED IN THE SHOCK-STRENGTHENING EXPERIMENTS

Material Designation	Grain Size, μ	Composition, w/o									
		Fe	Cr	Ni	Mo	Mn	Si	Cu	C	P	S
B	125	Bal	18.0	12.4	2.25	1.74	0.41	0.22	0.042	0.039	0.010
C	62	Bal	17.7	12.6	2.23	1.96	0.66	0.13	0.070	0.023	0.029

In application of the flyer plate technique, impact of the flyer normal to the surface to be hardened is usually sought in order to minimize dimensional distortion of the material. The experimental setup which was used in this study is illustrated in Figure B-73. In this technique, the initial angle between the flyer plate and surface to be impacted is adjusted to equal the plate bend angle generated on detonation of the explosive, so that the desired simultaneity of impact on the surface of the materials to be shocked is achieved. In the design of an experiment, the flyer plate thickness and velocity are determined from the desired depth of hardening and the desired impact pressure, using the Hugoniot relations of the flyer and target materials, respectively. Using design curves, developed for the explosive to be used, the explosive charge and angle are then established. The different shocking conditions and the specific materials which were utilized are enumerated in Table B-37.

FIGURE B-73. EXPERIMENTAL SETUP UTILIZED IN SHOCK STRENGTHENING AUSTENITIC STAINLESS STEEL

TABLE B-37. SHOCKING CONDITIONS

Designation	Pressure, kbar	Shocking Temperature, C	Material ^(a)
B-2	320	445	B
B-3	320	665	B
C-1	80	25	C
C-2	160	25	C

(a) See Table B-36 for code identification.

Experimental Procedures

The dynamic properties of unshocked and shocked Type 316 stainless steel were evaluated by instrumented Charpy impact tests. Tests were done on standard V-notch Charpy specimens at temperatures ranging from 25 to 650 C by using recommended test procedures (ASTM Designation: E23-66). Elevated temperatures were obtained by radiant heating in an electric muffle furnace. Specimens were heated to 3 percent over the nominal test temperature to compensate for temperature loss incurred during the approximately 5 sec needed to

complete the test. The overheat percentage was established over the temperature range of interest by removing a thermocoupled dummy specimen from the furnace and placing it on the anvil of the impact machine in the normal testing manner. The temperature drop at the notch root was measured by a spot-welded Chromel-Alumel thermocouple. The thermocouple output was monitored on a high-speed autographic recorder for the first 5 to 10 sec after removal from the furnace.

Tests were conducted on a conventional 240 ft-lb Satec impact machine. This machine has been certified with U. S. Army Materials and Mechanics Research Agency standard specimens in the past year. The instrumented components of the system consist of a strain-gaged tup, a high-gain dynamic amplifier, and a Textronic 564 fast-writing storage oscilloscope. The instrumented tup serves as a load measuring device (much like a tensile load cell), and through signal conditioning provides a load-time history of the test specimen. This information is recorded on film using an oscilloscope camera. The x-axis of this record can be converted to deflections using the effective pendulum velocity, and the y-axis is dynamically calibrated for load in pounds.

Interpretation of Instrumented Charpy Data

The only measured parameter in conventional Charpy testing is total energy. However, with load measuring capabilities the Charpy test can be considered a dynamic notch-bar bend test, and the many well developed notch-bar bending theories can be applied to the data analysis. These theories are based on sound metallurgical relationships and have been experimentally evaluated by other investigators⁽²⁻⁵⁾. Failure in notch-bar bending has been classified into six distinctive types⁽⁶⁾, as shown in Figure B-74. Distinguishing features of the load-time oscillograph are used to analyze the fracture behavior of the material. These features are shown in the idealized load-deflection record of Figure B-75. The symbols used in this figure are defined below:

P_{GY} = load at general yielding

P_{max} = maximum load

P_1 = load at the onset of fast fracture

d_{max} = deflection at maximum load

d_1 = deflection at the onset of fast fracture.

Figure B-75 also shows how total energy (W_T) can be subdivided into components of energy absorbed prior to crack initiation (W_I) and energy absorbed prior to unstable crack propagation (W_F). The energy, W_T , on the load deflection curve is equivalent to that measured on the pendulum dial.

Data from the time-load record are analyzed for dynamic yield stress, fracture toughness, and critical flaw size for each temperature. The dynamic yield stress is calculated from the measured general yield load, P_{GY} , and is related to the uniaxial tensile yield stress, σ_{ys} , by (7):

FRACTURE TYPE	LOAD-DISPLACEMENT CURVES	RAW DATA	REMARKS
I		P_F, d_I, W_T	Brittle fracture
II		P_{GY}, P_I, d_I, W_T	Brittle fracture
III		P_{GY}, P_I, d_I, W_T	Brittle fracture followed by fracture indicative of shear lip formation
IV		$P_{GY}, P_{max}, P_I, d_{max}, d_I, W_T$	Stable crack propagation followed by unstable brittle fracture and fracture indicative of shear lip formation
V		$P_{GY}, P_{max}, P_I, d_{max}, d_I, W_T$	Stable crack propagation followed by fracture indicative of shear lip formation
VI		$P_{GY}, P_{max}, d_{max}, W_T$	Stable crack propagation followed by gross deformation

FIGURE B-74. THE SIX TYPES OF FRACTURE FOR NOTCHED BAR BENDING

$$\sigma_{ys} = 33.3 P_{GY} \quad (3)$$

This factor is for a standard Charpy V-notch specimen, and is dependent upon the flank angle of the notch and assumed yield criterion (Tresca for this case).

FIGURE B-75. IDEALIZED LOAD-DEFLECTION RECORD

Fracture toughness refers to a critical stress intensity parameter, K_C , that can be related to structural service performance by a relationship of the form:

$$K_C \propto \sigma \sqrt{c} \quad (4)$$

where σ is the nominal stress in the vicinity of a flaw having a size indicative of the linear dimension, c . For the Type 316 stainless steel, all specimen fractures occurred beyond general yielding and, therefore, linear elastic fracture mechanics was not directly applicable. There is considerable controversy in the technical community regarding the calculation of a meaningful fracture toughness value from data derived from a specimen fracture beyond general yielding. For this study, two different approaches to the calculation of fracture toughness were employed. These methods are from evaluation of previous unpublished work by D. R. Ireland involving several methods of fracture toughness calculations ⁽⁶⁾.

The first method employs the assumption of equivalence of critical strain-energy release rate, G_C , to the ratio of impact energy to fracture surface area [i.e., area = 0.394 (0.394-a)]. The relationships used to calculate fracture toughness are:

$$K_c = (G_c E)^{1/2} \quad (5)$$

and

$$K_c = \left[\frac{W_i E}{0.394 (0.394 - a)} \right]^{1/2}, \quad (6)$$

where E is the elastic modulus and W_i is the impact energy to initiate a crack.

The second method employed for calculation of fracture toughness uses the somewhat more conventional crack-opening-displacement (COD) approach. The COD approach essentially uses the deflection measurements to calculate the strain at the root of the notch. COD is intended to represent this strain and can be calculated from the following:

$$\text{COD} = 0.51 (0.394 - a) d_{\max}, \quad (7)$$

where $d_{\max}$ is the appropriate deflection. The generally accepted relation of COD to G_c is

$$G_c = \text{COD} \times \sigma_{ys}, \quad (8)$$

and K_c is found by combining Equations (5), (7), and (8):

$$K_c = (\text{COD} \times \sigma_{ys} \times E)^{1/2}. \quad (9)$$

For these impact tests, σ_{ys} in Equation (9) is that determined by Equation (3).

One of the apparently more useful techniques for evaluating the structural performance of a material is by comparison of a hypothetical parameter identified as critical crack length (c). The c -value is a direct reflection of the dynamic yield strength and the K_c fracture toughness properties. In this context "critical" reflects the lowest fracture toughness at which the flaw will propagate or grow in size. The relationship of these parameters is given by

$$K_c = \sigma \sqrt{\pi c}, \quad (10)$$

where K_c is the fracture toughness, σ is the nominal stress normal to the flaw plane, and c is a linear measure of the flaw size. This hypothetical flaw can be regarded as being a two-dimensional object and is assumed to be a surface flaw in an infinitely wide plate of unit thickness. The flaw is assumed to have the fixed geometrical shape shown in Figure B-76. For this arbitrary evaluation of critical crack length, σ is assumed to be the dynamic yield stress which is determined by Equation (3), and the fracture toughness is the COD value, which is believed to be a conservative K_c value. The critical crack length, c , is calculated from the following rearranged form of Equation (10):

$$c = \frac{1}{\pi} (K_c / \sigma_{ys})^2.$$

Although this c -value is intended for use only as a comparative measure of material properties, it should be noted that the relationship shown in Equation (11) is very similar to that employed by Wilshaw and Pratt⁽⁸⁾ for studies of the relationship of metallurgical parameters to critical plastic zone size values in ferritic steels.

Results and Discussion

The effect of explosive shocking pressure on the dynamic mechanical properties of Type 316 stainless steel was determined by instrumented Charpy impact tests. Testing direction of specimens relative to the rolling and shocking directions of the plate is illustrated in Figure B-77. Testing temperatures were selected to overlap those used in uniaxial tensile tests, thus allowing a direct comparison of static and dynamic test data.

In the temperature range covered, the plastic constraint is reduced to a point that plastic deformation occurs across the entire specimen cross section. Thus, fracture is initiated by fibrous tearing at or close to the root of the notch and maximum load is reached prior to fracture load. Under these conditions while standard Charpy tests provide little insight into dynamic material behavior, instrumentation furnishes time-load data which can be evaluated by notch-bar bend theory and provide useful information.

Figures B-78 through B-80 show typical fracture surfaces and time-load records for various material conditions and test temperatures. The shocked specimens were observed to have laminations at the fracture surfaces. These laminations run parallel to the shock wave front, and are therefore assumed to be related to and produced by the shock wave. The gross fibrous tearing of materials shocked at 80 and 160 kbar gives an illusion of more pronounced lamination than in material shocked at 320 kbar. The latter, however, exhibits a higher density of fine laminar segments. These laminar defects undoubtedly have an adverse effect upon dynamic properties.

The time-load record for each specimen (such as the typical ones of Figures B-78 through B-80) is converted to load-deflection data. These data are used for the previously described calculations of energy, stress, fracture toughness, and critical crack length, thus furnishing information for engineering analysis. The test results from this investigation are presented in Table B-38.

Figures B-81 through B-85 graphically show the various energies (W_T , W_I , W_F), and loads (P_{GY} , P_{max}) as a function of test temperature for the control material and each shocking condition. In general, it can be observed that total energy, yield, and maximum loads decrease as the temperature increases; however, some shocked materials show a minimum total energy absorbed at approximately 500 C and then slight increases at 600 C and/or 650 C.

A comparative plot of the total energy absorbed during impact versus temperature for all materials is shown in Figure B-86. The shocked materials

FIGURE B-76. HYPOTHETICAL SURFACE FLAW OF CRACK LENGTH, c , IN INFINITE-WIDTH PLATE OF UNIT THICKNESS

FIGURE B-77. TESTING DIRECTION OF SPECIMENS RELATIVE TO THE ROLLING AND SHOCKING DIRECTIONS

2-1

Deflection (Full Scale) →

2-5

Deflection (Full Scale) →

2-7

Deflection (Half Scale) →

2X

4F580

Test Temperature, 25 C
Energy (W_T), 237.8 ft-lb
Type VI Fracture

2X

4F581

Test Temperature, 500 C
Energy (W_T), 203.3 ft-lb
Type VI Fracture

2X

4F582

Test Temperature, 650 C
Energy (W_T), 184.5 ft-lb
Type VI Fracture

FIGURE B-78. TYPICAL LOAD-TIME TRACE AND FRACTURE APPEARANCE
FOR UNSHOCKED TYPE 316 STAINLESS STEEL

3D1

Deflection (Quarter Scale) →

2X

4F587

Test Temperature, 25 C
 Energy (W_T), 119.5 ft-lb
 Type V Fracture

3D3

Deflection (Quarter Scale) →

2X

4F588

Test Temperature, 500 C
 Energy (W_T), 110.0 ft-lb
 Type VI Fracture

3C6

Deflection (Quarter Scale) →

2X

4F586

Test Temperature, 650 C
 Energy (W_T), 74.0 ft-lb
 Type VI Fracture

FIGURE B-79. TYPICAL LOAD-TIME TRACE AND FRACTURE APPEARANCE FOR TYPE 316 STAINLESS STEEL SHOCKED BY 160 KBAR AT AMBIENT TEMPERATURE

B-116

2C1

Deflection (Quarter Scale) →

2C3

Deflection (Quarter Scale) →

2C4

Deflection (Quarter Scale) →

2X

4F583

Test Temperature, 25 C
Energy (W_T), 34.5 ft-lb
Type III Fracture

2X

4F584

Test Temperature, 500 C
Energy (W_T), 23.0 ft-lb
Type III Fracture

2X

4F585

Test Temperature, 650 C
Energy (W_T), 23.5 ft-lb
Type III Fracture

FIGURE B-80. TYPICAL LOAD-TIME TRACE AND FRACTURE APPEARANCE FOR TYPE 316 STAINLESS STEEL SHOCKED BY 320 KBAR AT 665 C

TABLE B-38. MEASUREMENTS OBTAINED FROM TIME-LOAD RECORDS FOR SPECIMENS TESTED IN IMPACT BY INSTRUMENTED CHARPY TESTS

Material Identi- fication	Test Temperature, C	Energy, ft-lb			Fracture Type	Deflections, in.		Yield Stress, ksi		Fracture Toughness, ksi $\sqrt{\text{in.}}$		Critical Crack Length, in.
		W _T	W _I	W _F		d _{max}	d ₁	Static	Dynamic	W _I	COD _{max}	
B(a)	25	237.6	34.1	--	VI	0.181	--	33.7	65.7	308.8	236.0	4.104
	300	229.7	52.3	--	VI	0.363	--	22.8	35.9	359.3	231.0	13.169
	500	203.3	53.5	--	VI	0.449	--	19.9	28.7	345.8	219.6	18.644
	600	196.0	53.3	--	VI	0.468	--	19.9	27.9	336.4	215.3	18.956
	650	184.5	54.9	--	VI	0.491	--	19.9	27.9	336.5	217.4	19.345
B-2	25	77.5	24.0	31.7	V	0.089	0.112	108.5	115.4	259.2	219.3	1.149
	500	64.5	9.3	26.8	V	0.054	0.133	77.3	84.5	144.1	130.6	0.762
	500	56.0	10.3	22.3	V	0.059	0.114	77.3	84.5	151.9	136.6	0.832
	600	77.0	16.2	35.1	V	0.096	0.196	64.0	74.1	185.2	158.9	1.463
B-3	25	34.5	20.4	20.4	III	--	0.065	94.4	104.8	238.9	178.5	0.924
	500	23.0	6.7	6.7	III	--	0.037	72.7	81.7	122.2	106.4	0.539
	650	23.5	9.9	9.9	III	--	0.051	67.5	74.1	143.0	114.2	0.756
C-2	25	76.9	22.1	37.6	V	0.100	0.160	60.5	91.6	248.5	207.1	1.626
	500	77.0	17.5	36.7	V	0.105	0.204	51.7	68.5	197.8	164.1	1.825
	600	75.0	18.0	32.9	V	0.120	0.203	46.9	59.8	195.7	159.6	2.268
	650	74.0	17.2	--	VI	0.120	--	43.5	56.6	188.3	153.1	2.330
C-1	25	119.5	38.7	65.9	V	0.166	--	52.1	86.5	329.2	259.2	2.862
	500	110.0	28.1	--	VI	0.196	--	37.1	52.6	250.6	192.7	4.616
	500	103.0	22.5	--	VI	0.156	--	37.1	52.6	224.4	175.3	3.534
	600	109.0	21.9	--	VI	0.157	--	33.9	50.6	215.8	167.9	3.506
	650	111.5	21.0	--	VI	0.162	--	31.0	47.0	208.2	162.1	3.786

(a) Unshocked control material.

FIGURE B-81. TEMPERATURE DIAGRAM FOR LOADS AND ENERGIES OF UNSHOCKED TYPE 316 STAINLESS STEEL

FIGURE B-82. TEMPERATURE DIAGRAM FOR LOADS AND ENERGIES OF TYPE 316 STAINLESS STEEL SHOCKED BY 80 KBAR AT 25 C

FIGURE B-83. TEMPERATURE DIAGRAM FOR LOADS AND ENERGIES OF TYPE 316 STAINLESS STEEL SHOCKED BY 160 KBAR AT 25 C

FIGURE B-84. TEMPERATURE DIAGRAM FOR LOADS AND ENERGIES OF TYPE 316 STAINLESS STEEL SHOCKED BY 320 KBAR AT 445 C

FIGURE B-85. TEMPERATURE DIAGRAM FOR LOADS AND ENERGIES OF TYPE 316 STAINLESS STEEL SHOCKED BY 320 KBAR AT 665 C

FIGURE B-86. TEMPERATURE DIAGRAM FOR TOTAL ENERGY ABSORBED DURING IMPACT OF UNSHOCKED AND SHOCK-STRENGTHENED TYPE 316 STAINLESS STEEL

FIGURE B-87. TEMPERATURE DIAGRAM FOR FRACTURE TOUGHNESS OF UNSHOCKED AND SHOCKED TYPE 316 STAINLESS STEEL

show a relatively small total energy (W_T) change over the entire temperature range. The unshocked material undergoes a continual decrease in W_T with increased temperature, but still exhibits a very high absorption energy (184.5 ft-lb) at 650 C. The W_T for the tests at 25 C shows a decrease with increased shocking pressure, except for material shock strengthened by a pressure of 320 kbar at 445 C which possesses an energy level similar to that of the material shock strengthened by a pressure of 160 kbar at 25 C.

The room-temperature fracture toughness of all shocked material is less than that of the control material except for the material shocked at the lowest pressure (Figure B-87). Significantly, an increase in toughness is indicated in static tensile tests also where increases in both ductility and yield strength were obtained. Fracture toughness (K_C) decreases as the temperature increases, with all elevated-temperature values of the shocked material falling below those of the unshocked material. Material shocked by a pressure of 320 kbar at 445 C shows a minimum K_C at 500 C and then a large increase at 600 C. This trend may be indicative of a recovery process occurring at the higher temperatures.

The hypothetical critical crack length values are plotted as a function of temperature in Figure B-88. It can be seen that the unshocked values increase considerably with increased test temperature. However, this is not the case for shocked material, which maintains a rather constant low level over the entire temperature range. Increased shocking pressure causes a constant decrease in the critical crack length at all test temperatures. Figure B-89 shows this trend for 25 C and 650 C. This diagram also shows how the dynamic yield stress increases with increased shocking pressure. These two parameters are related to toughness in much the same manner as yield stress and ductility are in the uniaxial tensile test.

The ratio of maximum load (P_{max}) to general yield load (P_{GY}) as a function of shocking pressure is shown in Figure B-90. The ratios for all elevated-temperature tests fall very near the same curve, thus indicating quite similar dynamic strain-hardening characteristics at 500, 600, and 650 C. The unshocked material has an elevated-temperature P_{max}/P_{GY} ratio greater than 2, but it rapidly approaches unity as the shocking pressure increases. At room temperature the unshocked material has a much lower initial ratio of 1.48 and it shows only a slight decrease as a function of increasing pressure.

The ratio of dynamic yield stress to static yield stress is plotted as a function of temperature in Figure B-91. The ratio decreases as the temperature increases with a minimum reached at 500 to 600 C and a slight increase at 600 to 650 C. The ratio of dynamic to static yield stress at 25 C decreases from 1.95 for unshocked material to approximately 1.1 at pressures of 320 kilobar. This indicates a decrease in strain-rate sensitivity as the shocking pressure increases.

FIGURE B-88. TEMPERATURE DIAGRAM FOR CRITICAL CRACK LENGTH OF UNSHOCKED AND SHOCKED TYPE 316 STAINLESS STEEL

FIGURE B-89. DYNAMIC YIELD STRESS AND CRITICAL CRACK LENGTH OF TYPE 316 STAINLESS STEEL AS A FUNCTION OF SHOCKING PRESSURE

FIGURE B-90. RATIO OF MAXIMUM LOAD TO GENERAL YIELD LOAD AT VARIOUS TEMPERATURES AS A FUNCTION OF SHOCKING PRESSURE FOR TYPE 316 STAINLESS STEEL

FIGURE B-91. RATIO OF DYNAMIC YIELD STRESS TO STATIC YIELD STRESS FOR UNSHOCKED AND SHOCKED TYPE 316 STAINLESS STEEL AT VARIOUS TEMPERATURES

Conclusions

The dynamic strength properties of shock-strengthened Type 316 stainless steel were determined by instrumented Charpy tests. As a result of this study, a number of conclusions can be made.

The total energy needed to cause fracture in Type 316 stainless steel is decreased by shock strengthening. The decrease in fracture energy increases with shocking pressure at test temperatures ranging from 25 to 650 C.

Fracture toughness (K_{IC}) of Type 316 stainless steel is decreased by shock strengthening at temperatures ranging from 25 to 650 C, the only exception being the room temperature K_{IC} value of material shocked by a pressure of 80 kbar at 25 C. The increases in K_{IC} at testing temperatures of 600 C and above may indicate a recovery process for the material shocked by a pressure of 320 kbar.

The critical crack length is decreased by increasing shocking pressure. Increasing test temperature increases the critical crack length of unshocked material but the critical crack length of shocked material is essentially independent of testing temperature.

The dynamic yield strength (D_{YS}) is increased by increasing shocking pressures for all testing temperatures. This trend is expected since the static yield strength (S_{YS}) was also found to increase with increasing shocking pressure⁽¹⁾.

The ratio of dynamic yield to static yield strength (measure of strain-rate sensitivity) was found to decrease with increasing shocking pressure.

The dynamic strain-hardening ability of unshocked material is nearly doubled as test temperature is increased to 500 C. However, shocking even at the lowest pressure (80 kbar) almost completely removes the ability to strain harden. Further increases in shocking pressure reduce strain hardening still more until at 320 kbar the normally ductile Type 316 stainless steel fails in a nearly brittle manner, even when tested at 650 C.

Shocking appears to increase the number of internal flaws and the sensitivity of materials to internal flaws. These flaws appear to originate from the shock wave which passes through the material during shocking.

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EVALUATION OF LOW-CARBON FERRITIC 2-1/4 Cr-1 Mo STEEL

M. Kangilaski and A. A. Bauer

Introduction

The third effort under Task 14 involved investigation of the mechanical properties of low-carbon ferritic 2-1/4 Cr-1 Mo alloy steels as a means of evaluating their potential usefulness for LMFBR steam-generator tubing. The ferritic 2-1/4 Cr-1 Mo alloy steel is of interest for use as steam generator tubing in LMFBR's since the potential for stress corrosion cracking on the water-steam side of the tubing precludes the use of austenitic steels, which will serve as the major structural material in other reactor components. However, standard commercial heats of this alloy contain up to about 0.15 w/o carbon and, on the sodium side of the steam generator, exhibit preferential carbon transfer to the austenitic components. Because of design problems associated with carbon and mass transfer deposits and with the changing properties of the components materials as transfer proceeds, it is desirable to either prevent or minimize this transfer.

One approach to achieving a carbon-stabilized austenitic-ferritic bimetallic system for use in a LMFBR is to equalize the carbon potential in the ferritic and austenitic components by reducing the carbon content of the ferritic steel. An alternative approach is to add a carbon-stabilizing element such as niobium or titanium to the ferritic alloy. The first approach is being evaluated in this effort.

Experimental Program

Bars of 2-1/4 Cr-1 Mo alloy material containing 0.012 or 0.042 w/o carbon were received from Climax-Molybdenum Company. Creep-rupture specimens were machined from the bars. These specimens were to be creep-rupture tested at temperatures ranging from 900 to 1100 F, supplementing previous tensile and creep-rupture tests performed by Climax-Molybdenum, in order to provide data for a parametric analysis of stress-rupture life and secondary creep rates.

Experimental tensile and creep-rupture data for these materials, supplied by the Climax-Molybdenum Company Research Laboratory, were first evaluated. Tensile data were available at 900 and 1100 F and creep-rupture data at 1000, 1100, 1200, and 1300 F; creep-rupture data were obtained at stress levels that produced rupture at times to a maximum of about 1000 hr. It was concluded that the major need for creep-rupture data existed in the lower range of test temperatures and at reduced stress levels or extended rupture life.

Test temperatures and stresses were selected on the basis of a parametric analysis of the Climax-Molybdenum data, and creep-rupture tests were initiated. Results currently available from the creep-rupture tests are shown in Table B-39. All specimens still on test have experienced exposure periods in excess of 10,000 hr.

TABLE B-39. RESULTS OF CREEP-RUPTURE TESTS ON LOW-CARBON FERRITIC 2-1/4 Cr-1 Mo STEEL SPECIMENS

Carbon Content, w/o	Test Temperature, F	Stress, ksi	Rupture Time ^(a) , hr	Minimum Creep Rate, percent per hr	Primary Creep Strain, percent
0.012	900	20.0	On test	0.000020	0.43
	950	19.5	On test	0.00015	0.35
	950	16.3	6939.1	0.000055	0.16
	1000	18.0	973.3	0.00075	0.28
	1000	15.5	2137.3	0.00032	0.070
	1000	12.0	5880.4	0.00007	0.17
	1100	8.7	1719.5	(b)	0.070
0.042	900	26.0	On test	0.000013	0.54
	950	25.0	On test	0.000035	0.52
	950	20.0	7303.5	0.000018	0.32
	1000	22.7	1271.1	0.00028	0.43
	1000	19.7	2343.9	0.000125	0.20
	1000	15.5	4920.7	0.000042	0.10
	1100	11.2	1627.6	(b)	0.09

(a) Specimens still on test have all experienced exposure periods in excess of 10,000 hr.

(b) No evident secondary creep stage at this temperature. Specimen appears to have entered tertiary stage directly after primary creep stage.

Larson-Miller plots of the Battelle and earlier Climax-Molybdenum⁽¹⁾ creep-rupture data are shown in Figure B-92. The two sets of data show good agreement. Included in this figure is a plot of data for annealed commercial material (0.11 w/o carbon) which was compiled in an ASTM publication⁽²⁾. A significant feature of the results for the two low-carbon alloys is the break in slope of the plots at stresses of about 13,000 and 15,000 psi, respectively, for the 0.012 and 0.042 w/o carbon alloys; below this stress, a consistent decrease in rupture life with decrease in carbon content is indicated. However, at higher stresses the plots for the two low-carbon alloys cross the average plot for the higher-carbon, commercial-grade material.

FIGURE B-92. LARSON-MILLER PLOTS OF STRESS-RUPTURE DATA FOR FERRITIC 2-1/4 Cr-1 Mo STEEL SPECIMENS

The anomalous behavior at high stresses appears to be related to strain hardening of the low-carbon material. If the data recorded in Table B-39 are examined, it will be seen that for all tests conducted at stresses above 15,500 psi, the primary creep strain was 0.20 percent or greater, regardless of test temperature. Thus, despite the fact that the 0.2 percent yield strengths range 5,000 to 10,000 psi above this stress limit, when it is exceeded, significant strain hardening is encountered for test temperatures of 1100 F and below.

An analysis of the combined Battelle-Climax-Molybdenum creep-rate data was performed. The combined results appear in Figure B-93. At Battelle, it was noted that, at 1100 F, transition through the second stage of creep occurred so rapidly that it was impossible to determine a minimum steady-state creep rate so that no Battelle data are included at this temperature.

The curves drawn in Figure B-93 are calculated from a parametric analysis of the creep-rate data. The analysis is summarized in Figure B-94, where the data are treated to fit a creep law of the form,

$$\dot{\epsilon} = k\sigma^n \exp(-\Delta H/RT) .$$

The 1200 and 1300 F creep rates determined by Sponseller, et al.⁽¹⁾, did not fit with the lower-temperature data and therefore were not included in the analysis, which should be considered applicable only to temperatures below 1100 F. Presumably, at higher temperatures, the creep mechanism is altered by annealing affects.

The creep exponent, n , was determined to be 5.0 and the activation energy for creep, 54,960 cal per mole. The activation energy for self-diffusion in an iron-2.9 w/o chromium alloy containing 50 ppm carbon has been reported⁽³⁾ as 71,500 cal per mole, which is somewhat higher than the creep activation energy determined here. The creep constants (k) indicate that the 0.042 w/o carbon material possesses almost 1.5 times the creep strength of the 0.012 w/o carbon material. Creep rates shown by dashed lines in Figure B-93 for normal commercial material⁽²⁾ show no evident relationship, in terms of carbon content, to those obtained with the experimental alloys.

Plans for Future Work

The creep tests on the alloys containing 0.012 and 0.042 w/o carbon will be completed and reported under the Task 32 effort.

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FIGURE B-93. CREEP-RATE DEPENDENCE ON STRESS AND TEMPERATURE FOR LOW-CARBON FERRITIC 2-1/4 Cr-1 Mo STEEL SPECIMENS

FIGURE B-94. PARAMETER PLOT OF CREEP-TEMPERATURE DATA FOR LOW-CARBON FERRITIC 2-1/4 Cr-1 Mo STEEL SPECIMENS

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