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PROGRESS ON LMFBR CLADDING, STRUCTURAL,
AND COMPONENT MATERIALS STUDIES
DURING JULY, 1969, THROUGH JUNE, 1970
ANNUAL REPORT

MASTER

BATTELLE MEMORIAL INSTITUTE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

Prepared for U. S. Atomic Energy Commission
Under Contract No. W-7405-eng-92

Report Date: July, 1970

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**PROGRESS ON LMFBR CLADDING, STRUCTURAL,
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Donald L. Keller

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REPORTS RELATING TO REACTOR MATERIALS, TECHNOLOGY, AND
SAFETY ISSUED DURING THE PERIOD JULY, 1969, THROUGH JUNE, 1970

- BMI-1865 "Fission-Product Deposition and Its Enhancement Under Reactor Accident Conditions: Deposition on Containment-System Surfaces", by Harvey S. Rosenberg, Joseph M. Genco, and David L. Morrison.
- BMI-1866 "Investigation of the Initiation and Extent of Ductile Pipe Rupture. Phase I", by Robert J. Eiber, Willard A. Maxey, Arthur R. Duffy, and Thomas J. Atterbury.
- BMI-1867 "An Evaluation of the Applicability of Existing Data to the Analytical Description of a Nuclear-Reactor Accident. Quarterly Progress Report for April Through June, 1969", by David L. Morrison, Wayne A. Carbiener, and Robert L. Ritzman.
- BMI-1868 "Annual Report - Progress on Development of Materials and Technology for Advanced Reactors During July, 1969, Through June, 1969", by Donald L. Keller.
- BMI-1870 "Progress on High-Temperature Fuels Technology During August, 1968, Through July, 1969. Annual Report", by Donald L. Keller, and Walston Chubb.
- BMI-1871 "An Evaluation of the Applicability of Existing Data to the Analytical Description of a Nuclear-Reactor Accident. Quarterly Progress Report for July Through September, 1969", by Wayne A. Carbiener, and Robert L. Ritzman.
- BMI-1872 "Progress on Development of Materials and Technology for Advanced Reactors During July Through September, 1969", by Donald L. Keller.
- BMI-1873 "Investigation of the Initiation and Extent of Ductile Pipe Rupture. Phase II. Progress Report for May-September, 1969", by Robert J. Eiber, Willard A. Maxey, Arthur R. Duffy, and Thomas J. Atterbury.
- BMI-1874 "Fission-Product Deposition and Its Enhancement Under Reactor Accident Conditions: Development of Reactive Coatings", by Harvey S. Rosenberg, George E. Cremeans, Joseph M. Genco, David A. Berry, and David L. Morrison.
- BMI-1875 "Progress on High-Temperature Fuels Technology During August Through October, 1969", by Donald L. Keller, and Walston Chubb.
- BMI-1876 "Investigation of the Initiation and Extent of Ductile Pipe Rupture. Phase II. Progress Report for October-December, 1969", by Robert J. Eiber, Willard A. Maxey, Arthur R. Duffy, and Thomas J. Atterbury.
- BMI-1877 "An Evaluation of the Applicability of Existing Data to the Analytical Description of a Nuclear-Reactor Accident: Quarterly Progress Report for October Through December, 1969", by Wayne A. Carbiener, and Robert L. Ritzman.
- BMI-1878 "Progress on Development of Materials and Technology for Advanced Reactors During October Through December, 1969", by Donald L. Keller.
- BMI-1879 "Progress on High-Temperature Fuels Technology During November, 1969, Through January, 1970", by Donald L. Keller, and Walston Chubb.
- BMI-1880 "Investigation of the Initiation and Extent of Ductile Pipe Rupture. Phase II. Progress Report for January-March, 1970", by Robert J. Eiber, Willard A. Maxey, Arthur R. Duffy, and Thomas J. Atterbury.
- BMI-1881 "An Evaluation of the Applicability of Existing Data to the Analytical Description of a Nuclear-Reactor Accident. Quarterly Progress Report for January Through March, 1970", by Wayne A. Carbiener, and Robert L. Ritzman.
- BMI-1882 "Progress on Development of Materials and Technology for Advanced Reactors During January Through March, 1970", by Donald L. Keller.
- BMI-1883 "Review of Through-Wall Critical Crack Formulations for Piping and Cylindrical Vessels", by Robert J. Eiber, Willard A. Maxey, Arthur R. Duffy, and Thomas J. Atterbury.

HIGHLIGHTS OF RESULTS DURING REPORTING PERIOD

Results of DRDT-supported studies at Battelle-Columbus during Fiscal Year 1970 on cladding, component, and structural materials of interest for LMFBR applications are summarized in this report. The work under way includes a program directed toward developing mechanical-property design data and a comprehensive, long-term study directed toward the elucidation of the effects of irradiation on cladding and structural alloys. Highlights of the results obtained are cited below.

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

In a program to evaluate the low-cycle-fatigue resistance of Incoloy 800, measurements were completed at 800 C on solution-annealed specimens. The curve fell somewhat above that developed earlier in tests at 1000 C. Attention was also given to the effect of incorporating hold times in compression or tension in the test stress cycle. For total strain ranges between 1.56 and 2.73 percent, cyclic fatigue life was greatly reduced at 1400 and 1200 F and slightly reduced at 1000 F when only tension 10-min hold times were applied. When only compression or both tension and compression 10-min hold times were used, the cyclic fatigue life at 1400, 1200, and 1000 F was but slightly reduced.

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

An evaluation of two potential methods to inexpensively determine radiation-induced changes in mechanical properties of cladding materials was undertaken. Testing of ring specimens fabricated from tubing proved unsatisfactory in that friction effects associated with the loading method complicated interpretation of the data. Experiments with a microhardness technique involving the use of variable-angle cone indenters to determine stress-strain properties produced consistent results only with nonirradiated material.

In studies of radiation effects on mechanical properties, a relationship was established between elevated-temperature (600 C) embrittlement and >50-A helium bubbles in the microstructure of Type 347 stainless steel. Such bubbles were observed in specimens annealed at 980 C but not in as-irradiated specimens, in which embrittlement effects were more severe. The effects of irradiation temperature on the mechanical properties of Types 347 and 348 stainless were studied also. In tensile tests below 450 C, specimens irradiated at 400 C exhibited the largest change in mechanical properties; those irradiated at 50 C showed an intermediate effect and those irradiated at 650 C the least change. Specimens tested at 750 C displayed an embrittlement effect that was independent of irradiation temperature. In efforts to explain the high helium contents of these irradiated alloys, calculations based upon Weitman's nickel transmutation hypothesis were carried out. Good correlations with analytical data were obtained.

In a study of the response of materials of different microstructures to irradiation-induced embrittlement at elevated temperatures, a capsule containing Type 316 stainless steel tensile specimens with oriented grain structures was prepared for insertion in the EBR-II. In work concerned with the use of high-defect structures to control void formation during irradiation, explosive shock hardening of Type 316 stainless was found to produce large increases in yield strength, moderate increases in ultimate strength, and reductions in ductility. Steady-state creep rates at 650 C were reduced. In annealing studies the shock-induced hardening was largely retained for 1500 hr at 700 C.

Creep studies were initiated on alloys containing 0.012 and 0.046 w/o carbon in an investigation to evaluate low-carbon ferritic 2-1/4 Cr-1 Mo steel for LMFBR steam-generator tubing.

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

J. S. Perrin

This program is being conducted for the AEC Division of Reactor Development and Technology to generate needed design data on reactor component materials by the performance of mechanical-property tests. The first phase of this work consists of obtaining low-cycle, strain-controlled fatigue design data on Incoloy 800 for submission to ASTM to establish a code.

Later phases of the work will be concerned with generating 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 Incoloy 800

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

Objective of Program

To develop low-cycle-fatigue design data for Incoloy 800 at temperatures from 800 to 1400 F.

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 data for Incoloy 800.

The experimental procedures used in this program are very similar to those used previously in elevated-temperature, low-cycle-fatigue evaluations of three austenitic stainless steel alloys⁽¹⁻⁴⁾. The tests involved 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 closed-loop, servocontrolled hydraulic system operated in axial strain control. Axial load is applied to an hourglass-shaped specimen heated by high-frequency induction (see Figure A-1). 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 (see Figure A-2). The diametral strain and load signals are combined and converted to an equivalent axial strain using an analog computer.

Significant Accomplishments During Fiscal Year 1970

A curve of total strain range versus fatigue life was generated at 800 F between 10^2 and 10^5 cycles for solution-annealed Incoloy 800. The curve was just slightly above earlier results for the same material at 1000 F and about a factor of two in cyclic fatigue life below earlier results for mill-annealed Incoloy 800 at 800 F.

The effect of hold times at constant strain on the low-cycle fatigue behavior of Incoloy 800 was investigated at 1000, 1200, and 1400 F and compared with zero hold-time results under continuous cycling. For total strain ranges between 1.56 and 2.73 percent, cyclic fatigue life was greatly reduced at 1400 and 1200 F and slightly reduced at 1000 F when 10-min tension-only hold times were applied. When

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FIGURE A-1. GAGE SECTION OF LOW-CYCLE-FATIGUE SPECIMEN WITH INDUCTION HEATING COIL

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FIGURE A-2. LOADING FIXTURE WITH LOW-CYCLE-FATIGUE SPECIMEN, INDUCTION HEATING COIL, AND DIAMETRAL EXTENSOMETER

compression-only or both tension and compression hold times were used, the cyclic fatigue life at 1400, 1200, and 1000 F was only slightly reduced.

Effects of a duplex solution anneal and higher carbon content (0.09 w/o instead of 0.05 w/o) were studied. The duplex-solution-annealed material had about the same or slightly lower fatigue lives than the normal material with tension-only hold times. The higher carbon content improved the tension-only hold-time fatigue resistance at 1000 and 1200 F, but gave lower fatigue resistance at 1400 F.

Summary of Research Carried Out

The objective of this program was to extend the understanding of the low-cycle-fatigue behavior of solution-annealed Incoloy 800 beyond that developed in earlier studies⁽⁵⁾. Data for constant-amplitude, completely reversed testing were to be developed at 800 F. The influence of 10-min hold times at maximum tensile strain on low-cycle-fatigue behavior was to be examined at 1000, 1200, and 1400 F and at total strain ranges near 2 percent. Also, the effects of compressive 10-min hold times, and both tensile and compressive 10-min hold times, were to be looked at briefly. In addition, the effects of two processing variables were to be studied both with and without tensile hold times.

All experimental work was done with the apparatus described earlier. Analog computers, programmers, diametral extensometers, and loading fixtures for two such systems were built for Battelle-Columbus by GE-NSP. To conserve the limited quantity of Incoloy 800 samples, both systems were checked out using several dummy specimens of Type 304 stainless steel before any Incoloy 800 material was tested. However, because Incoloy 800 exhibits much more discontinuous yielding at these temperatures than does Type 304 stainless steel, experimental difficulties were encountered in maintaining the stability of control during some of the first tests of Incoloy 800. In several cases, these instabilities resulted in loss of the specimen before successful completion of the test.

During each test, axial strain was programmed to follow either a triangular or trapezoidal waveform with a mean level of zero, as shown in Figure A-3. For the constant-amplitude, fully reversed tests with no hold times, the triangular waveform of Figure A-3a was used. For each hold-time test the waveform of either Figures A-3b, A-3c, or A-3d was used. In all cases the total strain range, $\Delta\epsilon_t$, and strain rate, $\dot{\epsilon}$, were kept constant throughout each test. The length of the hold time, Δt , was also maintained constant throughout each hold-time test. For each sample tested, load-versus-time curves such as those shown schematically in Figure A-3 were recorded continuously to provide information on strain hardening, cyclic relaxation behavior, and initiation of cracking. Also, load-versus-axial strain hysteresis loops were periodically recorded to check the relative amounts of elastic and plastic strain as a function of cycles.

Sixty-three specimens of solution-annealed Incoloy 800 were taken from three different heats of material (see Table A-1). All specimens were machined to the dimensions shown in Figure A-4 and supplied to Battelle-Columbus by Gulf General Atomics. Group A had a normal chemical composition and heat treatment. Group B was the same as Group A except for annealing process, and Group C was the same as Group A except for the higher than normal carbon content (0.09 w/o instead of 0.05 w/o).

As outlined in Tables A-2, A-3, and A-4, 25 specimens of Group A, 13 specimens of Group B, and 14 specimens of Group C have been evaluated. Most of the hold-time tests involved 10-min-long hold times in tension only. Choice of this type and length of hold time was based upon previous results for Type 304 stainless steel⁽⁴⁾, where it was shown that hold times in tension only had the most deleterious influence on cyclic fatigue life. The 10-min hold time was long enough to allow significant relaxation to occur, but short enough to allow completion of tests within a reasonable length of time.

FIGURE A-3. SCHEMATIC OF TYPICAL LOADING CONDITIONS FOR LOW-CYCLE-FATIGUE TESTS

Scale : Full

Tolerances :

.XX $\pm$.010.XXX $\pm$.002

FIGURE A-4. LOW-CYCLE-FATIGUE SPECIMEN

TABLE A-1. IDENTIFICATION OF SOLUTION-ANNEALED INCOLOY 800
LOW-CYCLE-FATIGUE SPECIMENS

	Heat HH8968A ^(a)	Heat HH0901A ^(b)	Heat HH3310A ^(a)
Group Identification	A	B	C
Number of Specimens	36	13	14
Chemical Analysis, w/o			
Carbon	0.05	0.05	0.09
Manganese	0.84	0.90	1.14
Iron	44.38	45.61	46.84
Sulfur	0.008	0.007	0.007
Silicon	0.29	0.24	0.52
Copper	0.45	0.22	0.26
Nickel	31.41	31.59	30.12
Chromium	21.61	20.31	21.00

(a) Hot-finished 3/4-in. -diameter round, solution-annealed at 2100 F for 1 hr.

(b) Hot-finished 5/8-in. -diameter round, annealed and reannealed with the final solution anneal at 2100 F for 1 hr.

TABLE A-2. SUMMARY OF FATIGUE TESTS OF SOLUTION-ANNEALED INCOLOY 800 BAR FROM GROUP A
(HEAT HH8968A) AT AN AXIAL STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

Specimen	Test Temperature, F	Tension Hold Time, min	Compression Hold Time, min	Axial Strain Range at $N_f/2$, percent			Stress Range at $N_f/2$, psi	Fatigue Life			
				Total	Plastic	Elastic		N_o , cycles	N_5 , cycles	N_f , cycles	t_f , hr
1A	1400	0	0	2.64	2.12	0.52	78,000	177	192	298	1.06
2A	1400	0	0	0.50	0.22	0.28	44,000	4,500	4,800	5,760	3.20
3A	1200	0	0	2.51	1.89	0.62	106,000	200	241	306	1.09
4A	1200	0	0	--	--	--	--	--	--	--	(a)
5A	1000	0	0	2.35	1.74	0.61	123,000	276	--	279	0.96 ^(b)
6A	1000	0	0	--	--	--	--	--	--	--	(c)
17A	800	0	0	2.02	1.61	0.41	121,000	950	1,080	1,209	3.36
19A	800	0	0	2.03	1.64	0.39	120,000	810	860	884	2.46
14A	800	0	0	1.08	0.78	0.30	91,000	4,300	4,900	5,280	7.33
15A	800	0	0	1.05	0.74	0.31	94,000	3,600	3,850	4,249	5.90
16A	800	0	0	0.38	0.14	0.24	82,000	40,500	42,000	43,855	24.4
18A	800	0	0	0.40	0.15	0.25	83,000	(d)	(d)	33,378	18.6
20A	800	0	0	0.24	0.03	0.21	71,000	(e)	(e)	228,216 ^(e)	95.0
13A	1400	10	0	2.73	2.39	0.34	88,500	80	91	122	20.7
7A	1400	10	0	2.28	1.90	0.38	64,300	110	122	141	24.0
8A	1400	10	0	1.76	1.51	0.25	61,600	142	165	219	37.3
21A	1400	10	10	2.29	2.09	0.20	73,700	190	220	329	111
22A	1400	0	10	2.28	2.02	0.26	87,100	220	230	312	53.0
9A	1200	10	0	2.00	1.55	0.45	86,800	114	128	154	26.2
10A	1200	10	0	1.56	1.17	0.39	80,100	151	157	193	32.8
23A	1200	10	10	2.27	1.98	0.29	109,000	185	220	370	129
24A	1200	0	10	2.25	1.84	0.41	118,000	225	240	269	45.7
11A	1000	10	0	2.26	1.59	0.67	109,000	455	464	488	83.0
12A	1000	10	0	2.24	1.71	0.53	104,000	380	412	491	83.4
25A	1000	10	10	2.29	1.57	0.72	140,000	430	480	565	192

(a) Extensometer slipped off specimen.

(b) Preliminary examination of fracture surface shows that a metallurgical flaw may have caused the shorter than anticipated fatigue life.

(c) Equipment malfunction occurred.

(d) No data because of recorder malfunction.

(e) Specimen failed at button end.

TABLE A-3. SUMMARY OF FATIGUE TESTS OF SOLUTION-ANNEALED INCOLOY 800 BAR FROM GROUP B (HEAT HH0901A) AT AN AXIAL STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$ WITH NO COMPRESSION HOLD TIME

Specimen	Test Temperature, F	Tension Hold Time, min	Axial Strain Range at $N_f/2$, percent			Stress Range at $N_f/2$, psi	Fatigue Life			
			Total	Plastic	Elastic		N_o , cycles	N_5 , cycles	N_f , cycles	t_f , hr
4B	1400	0	2.58	2.21	0.37	98,000	170	190	245	0.85
3B	1200	0	2.58	2.13	0.45	132,000	340	360	480	1.67
5B	1200	0	0.46	0.18	0.28	84,300	16,500	19,000	21,259	14.8
2B	1000	0	2.58	2.10	0.48	142,000	560	575	595	2.07
1B	1400	10	2.73	2.39	0.34	93,600	70	82	102	17.2
13B	1400	10	2.76	2.44	0.32	92,400	64	85	116	19.8
7B	1400	10	--	--	--	--	--	--	--	(a)
8B	1400	10	--	--	--	--	--	--	--	(b)
9B	1200	10	2.76	2.42 ^(c)	0.34 ^(c)	(d)	(d)	(d)	62	10.5
10B	1200	10	2.70	2.19	0.51	142,000	62	72	80	13.6
11B	1000	10	--	--	--	--	--	--	--	(b)
12B	1000	10	2.76	2.16	0.60	149,000	115	150	221	37.6
6B	1000	10	2.62	2.17	0.45	151,000	220	260	276	46.9

- (a) Temperature controller malfunction.
 (b) Stability problem in control loop caused test to go out of control.
 (c) At 3 cycles, not $N_f/2$.
 (d) No data because of recorder malfunction.

TABLE A-4. SUMMARY OF FATIGUE TESTS OF SOLUTION-ANNEALED INCOLOY 800 BAR FROM GROUP C (HEAT HH3310A) AT AN AXIAL STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$ WITH NO COMPRESSION HOLD TIME

Specimen	Test Temperature, F	Tension Hold Time, min	Axial Strain Range at $N_f/2$, percent			Stress Range at $N_f/2$, psi	Fatigue Life			
			Total	Plastic	Elastic		N_o , cycles	N_5 , cycles	N_f , cycles	t_f , hr
1C	1400	0	2.47	2.13	0.34	87,000	230	250	285	0.96
2C	1400	0	0.38	0.16	0.22	59,000	>5,500 ^(a)	>5,500 ^(a)	>5,500 ^(a)	3.1 ^(a)
3C	1200	0	2.89	2.30	0.59	131,000	260	300	362	1.26
4C	1200	0	0.40	0.16	0.24	74,300	12,600	14,400	16,357	9.1
13C	1000	0	2.69	2.28	0.41	157,000	315	322	335	1.16
6C	1000	0	0.41	0.15	0.26	71,000	15,455 ^(b)	15,455 ^(b)	15,455 ^(b)	8.6 ^(b)
7C	1400	10	2.75	2.26	0.49	97,000	50	61	91	15.5
8C	1400	10	2.65	2.31	0.34	92,000	58	71	92	15.6
9C	1200	10	2.75	2.23 ^(c)	0.52 ^(c)	128,000 ^(c)	95	110	131	22.3
10C	1200	10	2.66	2.18 ^(c)	0.48 ^(c)	134,000 ^(c)	85	95	108	18.4
14C	1200	60	2.32	2.04	0.28	115,000	57	75	125	126
5C	1200	300	2.34	2.10	0.24	104,000	70	76	114	571
11C	1000	10	2.62	2.01	0.61	154,000	265	352	402	68.3
12C	1000	10	2.64	2.06	0.58	155,000	370	460	642	109

- (a) Recorder malfunctioned and system did not automatically shut off at failure. The specimen was still cycling at 32,192 cycles.
 (b) There was no drop in load before failure cycle, and preliminary examination of the fracture surface showed evidence of a possible flaw at point of crack initiation.
 (c) At 30 cycles, where $\Delta\sigma$ was maximum.

Values of plastic and elastic strain range listed in Tables A-2 through A-4 were taken from the cyclically stable hysteresis loops (usually recorded near half the number of cycles to fracture, $N_f/2$). Stable stress range ($\Delta\sigma$) values 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. The four values listed for fatigue life are defined as follows:

- N_0 = number of cycles where tension load starts to drop off just before complete fracture occurs
- N_5 = number of cycles where tension load drops off 5 percent below a cyclically stable value just before complete fracture occurs
- N_f = number of cycles to complete fracture
- t_f = time to complete fracture.

Seven specimens from Group A were used to generate a curve of total strain range versus fatigue life at 800 F (see Figure A-5). Plotted points represent individual tests and the curve is a "best-fit" line through the points. Also shown in Figure A-5 are data from Conway, et al. (5), for the same material at 1000 F and for mill-annealed Incoloy 800 at 800 F. The 800 F curve for solution-annealed material falls slightly above the comparable 1000 F curve and significantly below the 800 F curve for mill-annealed material.

The values of N_5 and N_f reported in Tables A-2 through A-4 were compared with corresponding mean fatigue lives, $\bar{N}_5$ and $\bar{N}_f$, obtained from the results of Conway, et al. (5), by computing values of $N_5/\bar{N}_5$ and $N_f/\bar{N}_f$ for each test (see Table A-5). From the viewpoint of cyclic fatigue life, these values are damage ratios that can be used to assess the relative amount of fatigue damage done during each test. When these ratios are close to unity, the fatigue damage is similar to that for completely reversed, constant-amplitude strain cycling. However, when the values are significantly less than unity, it is likely that some material variation or test condition has had a degrading effect on the fatigue behavior.

Since Group A was the same heat of material studied by Conway, et al. (5), it is not surprising that the zero hold-time tests gave damage ratios close to unity. Results for zero hold-time tests for both Groups B and C were similar to those for Group A.

For all of the hold-time tests, average values of $N_5/\bar{N}_5$ and $N_f/\bar{N}_f$ are summarized in Table A-6. At 1400 F, Groups A and B show similar behavior, with Group C exhibiting lower fatigue resistance. At 1200 F, Groups A and B again exhibit similar behavior, with Group C showing superior fatigue resistance. At 1000 F, Group C shows better fatigue resistance than Group A, and Group A shows better resistance than Group B. For Groups A and B, cyclic fatigue life was reduced the most at 1200 F and the least at 1000 F. For Group C, it was reduced most at 1400 F and least at 1000 F. Increasing the hold time at 1200 F from 10 to 60 min decreased the cyclic fatigue life of Group C material; however, increasing it from 60 to 300 min made no significant change in fatigue life.

For the five tests where compression hold times were present, the damage ratios were only slightly less than unity. At 1400 and 1200 F, they were significantly greater than the damage ratios for tension-only hold times, and at 1000 F they were about the same. The results at 1400 and 1200 F are similar to those obtained previously for Type 304 stainless steel at 1200 F (4, 6).

An analysis of the cyclically stable stress-relaxation curves similar to that reported for Type 304 stainless steel (4, 6) was made, and the results are reported in Table A-7. The terms used in Table A-7 are defined in Figure A-6 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. Values of stress were calculated on the basis of the specimen cross-sectional area at temperature.

FIGURE A-5. FATIGUE LIFE AS A FUNCTION OF TOTAL STRAIN RANGE FOR SOLUTION-ANNEALED INCOLOY BAR AT 800 F AND AN AXIAL STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

TABLE A-5. SUMMARY OF CYCLIC-FATIGUE-LIFE DAMAGE RATIOS FOR SOLUTION-ANNEALED INCOLOY 800 BAR TESTED AT AN AXIAL STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

Specimen	Tension Hold Time, min	Compression Hold Time, min	Test Temperature, F	Total Strain Range, percent	Mean Fatigue-Life Value ^(a)		Actual Fatigue-Life Value		Damage Ratio	
					N_5	N_f	N_5	N_f	N_5/N_5	N_f/N_f
1A	0	0	1400	2.64	225	260	192	298	0.85	1.15
2A	0	0	1400	0.50	3,500	4,200	4,800	5,760	1.37	1.37
3A	0	0	1200	2.51	280	330	241	306	0.89	0.93
5A	0	0	1000	2.35	600	640	276	279	0.46	0.44
13A	10	0	1400	2.73	215	250	91	122	0.42	0.49
7A	10	0	1400	2.28	280	335	122	141	0.44	0.42
8A	10	0	1400	1.76	410	500	165	219	0.40	0.44
21A	10	10	1400	2.29	280	335	220	329	0.79	0.98
22A	0	10	1400	2.28	280	335	230	312	0.82	0.93
9A	10	0	1200	2.00	430	500	128	154	0.30	0.31
10A	10	0	1200	1.56	690	780	157	193	0.23	0.25
23A	10	10	1200	2.27	340	400	220	370	0.65	0.93
24A	0	10	1200	2.25	345	410	240	269	0.70	0.66
11A	10	0	1000	2.26	650	700	464	488	0.72	0.70
12A	10	0	1000	2.24	650	700	412	491	0.64	0.70
25A	10	10	1000	2.29	630	680	480	565	0.76	0.83
4B	0	0	1400	2.58	230	270	190	245	0.83	0.91
3B	0	0	1200	2.58	265	315	360	480	1.36	1.52
5B	0	0	1200	0.46	12,300	16,000	19,000	21,259	1.54	1.33
2B	0	0	1000	2.58	480	520	575	595	1.20	1.14
1B	10	0	1400	2.73	215	250	82	102	0.38	0.41
13B	10	0	1400	2.76	210	245	85	116	0.40	0.47
9B	10	0	1200	2.76	235	280	55	62	0.23	0.22
10B	10	0	1200	2.70	245	295	72	80	0.29	0.27
12B	10	0	1000	2.76	420	460	150	221	0.36	0.48
6B	10	0	1000	2.62	470	510	260	276	0.55	0.54
1C	0	0	1400	2.47	245	290	250	285	1.02	0.98
2C	0	0	1400	0.38	6,600	8,200	>5,500	>5,500	>0.83	>0.67
3C	0	0	1200	2.89	215	255	300	362	1.40	1.42
4C	0	0	1200	0.40	19,500	20,000	14,400	16,357	0.74	0.82
13C	0	0	1000	2.69	450	490	322	335	0.72	0.68
6C	0	0	1000	0.41	25,000	36,000	15,455	15,455	0.62	0.43
7C	10	0	1400	2.75	210	245	61	91	0.29	0.37
8C	10	0	1400	2.65	225	260	71	92	0.32	0.35
9C	10	0	1200	2.75	235	280	110	131	0.47	0.47
10C	10	0	1200	2.66	255	300	95	108	0.37	0.36
14C	60	0	1200	2.32	325	385	75	125	0.23	0.32
5C	300	0	1200	2.34	320	375	76	114	0.24	0.30
11C	10	0	1000	2.62	470	510	352	402	0.75	0.79
12C	10	0	1000	2.64	460	500	460	642	1.00	1.28

(a) These values were taken from data of GEMP-732⁽⁵⁾.

TABLE A-6. AVERAGE CYCLIC-FATIGUE-LIFE DAMAGE RATIOS FOR HOLD-TIME TESTS
OF THREE DIFFERENT HEATS OF SOLUTION-ANNEALED INCOLOY 800 BAR
AT AN AXIAL STRAIN RATE OF $4 \times 10^{-3} \text{ SEC}^{-1}$

Heat Identification	Tension Hold Time, min	Compression Hold Time, min	Test Temperature, F	Number of Tests	Average Damage Ratio	
					N_5/N_5	N_f/N_f
HH8968A (Group A)	10	0	1400	3	0.42	0.45
	10	10	1400	1	0.79	0.98
	0	10	1400	1	0.82	0.93
	10	0	1200	2	0.27	0.28
	10	10	1200	1	0.65	0.93
	0	10	1200	1	0.70	0.66
	10	0	1000	2	0.68	0.70
	10	10	1000	1	0.76	0.83
HH0901A (Group B)	10	0	1400	2	0.39	0.44
	10	0	1200	2	0.26	0.25
	10	0	1000	2	0.46	0.51
HH3310A (Group C)	10	0	1400	2	0.31	0.36
	10	0	1200	2	0.42	0.42
	60	0	1200	1	0.23	0.32
	300	0	1200	1	0.24	0.30
	10	0	1000	2	0.88	1.04

As a result of the experimental work conducted during the past year, five important observations were made about the elevated-temperature, low-cycle-fatigue behavior of solution-annealed Incoloy 800:

- (1) At 800 F, the curve for $\Delta\epsilon_t$ versus N_f between 10^2 and 10^5 cycles falls slightly above the corresponding curve at 1000 F. However, this curve at 800 F is about a factor of two in fatigue life below the corresponding curve for mill-annealed Incoloy 800 at 800 F.
- (2) When compared with the results obtained in continuous cycling tests, it is evident that the 10-min hold times at maximum tension strain only greatly reduce cyclic fatigue life at 1400 and 1200 F and reduce it somewhat at 1000 F. This reduction in fatigue life is greatest at 1200 F and least at 1000 F.
- (3) When compared with the results in continuous cycling tests, it is found that compression-only 10-min hold times and both compression and tension 10-min hold times reduce cyclic fatigue life to only a small degree at 1400, 1200, and 1000 F. At 1400 and 1200 F, the fatigue life is markedly greater than for tension-only hold times and at 1000 F the fatigue life is about the same as that for tension-only hold times.
- (4) The duplex solution anneal did not yield better low-cycle-fatigue behavior at 1000, 1200, and 1400 F than the regular solution anneal and gave slightly worse behavior with 10-min tension-only hold times.
- (5) Increasing the carbon content of solution-annealed Incoloy 800 from 0.05 to 0.09 w/o gave better hold-time fatigue resistance at 1000 and 1200 F, but worse hold-time fatigue resistance at 1400 F.

TABLE A-7. CYCLICALLY STABLE STRESS-RELAXATION DATA FOR LOW-CYCLE FATIGUE OF SOLUTION-ANNEALED INCOLOY 800
BAR AT AN AXIAL STRAIN RATE OF 4×10^{-3} SEC⁻¹

Specimen	Tension Hold Time, min	Compression Hold Time, min	Stress Range, ksi	Relaxed Stress Range, ksi	Tensile Stress, ksi		Compressive Stress, ksi		Relaxed Tensile Stress, ksi	Relaxed Compressive Stress, ksi	Mean Relaxed Tensile Stress, ksi	Time to Fracture at Tensile Stress, hr
					Maximum	Minimum	Maximum	Minimum				
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
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
1B	10	0	93.6	61.8	46.8	15.9	-46.8	-46.8	30.9	0	31.4	17.0
13B	10	0	92.4	59.2	46.2	13.1	-46.2	-46.2	33.1	0	29.6	19.3
7C	10	0	97.0	63.0	50.5	16.5	-46.5	-46.5	34.0	0	33.5	15.2
8C	10	0	92.0	62.0	46.0	16.0	-46.0	-46.0	30.0	0	31.0	15.3
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
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
10B	10	0	142	108	72.3	39.2	-69.3	-69.3	33.1	0	55.7	13.3
9C	10	0	128	94.6	65.7	32.9	-62.3	-62.3	32.9	0	49.3	21.8
10C	10	0	134	104	68.3	38.2	-65.7	-65.7	30.1	0	53.2	18.0
14C	60	0	115	79.5	59.2	24.1	-55.4	-55.4	35.1	0	41.7	125
5C	300	0	104	70.1	53.1	19.1	-51.0	-51.0	34.0	0	36.1	570
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
25A	10	10	140	131	72.0	66.8	-67.6	-64.0	5.2	3.6	69.4	94.2
12B	10	0	149	145	71.3	66.3	-77.8	-77.8	5.0	0	68.8	36.9
6B	10	0	151	138	77.5	65.3	-73.1	-73.1	12.2	0	71.4	46.0
11C	10	0	154	145	78.6	69.2	-74.8	-74.8	9.4	0	73.9	67.0
12C	10	0	155	145	79.3	69.3	-75.8	-75.8	10.0	0	74.3	90.3

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
 Mean Relaxed
 Tensile Stress = $(STMAX + STMIN)/2$

FIGURE A-6. DEFINITION OF TERMS USED IN REPORTING STRESS RELAXATION DATA FOR HOLD-TIME LOW-CYCLE-FATIGUE TESTS

Plans for Future Work

The effect of longer hold times and lower strain ranges on the low-cycle fatigue behavior of solution-annealed Incoloy 800 will be investigated. It is planned to study these effects by evaluating 14 specimens from the first heat according to the matrix listed in Table A-8. To check existing theories for relating creep and fatigue damage, several creep tests will be performed on the same Incoloy 800 material at 1000, 1200, and 1400 F.

TABLE A-8. PLANNED FATIGUE TESTS OF SOLUTION-ANNEALED
INCOLOY 800 BAR FROM GROUP A (HH8968A) AT
AN AXIAL STRAIN RATE OF 4×10^{-3} SEC⁻¹
WITH NO COMPRESSION HOLD TIMES

Test Temperature, F	Tension Hold Time, min	Total Strain Range, percent	Number of Replicates	Number of Tests
1400	10	0.5	2	2
	30-60(a)	0.5	1	1
1200	10	0.5	2	2
	30	0.5	1	1
	60	2.5, 0.5	1	2
	300	2.5	1	1
1000	10	0.5	2	2
	60	2.5, 0.5	1	2
	300	2.5	1	1
Total =				14

(a) Stress-relaxation tests will be conducted at 1400 F. The hold time will be adjusted to be no longer than the time for complete stress relaxation.

The influence of hold time on the behavior of Type 304 stainless steel will be investigated. This work will expand the previous results at 1200 F^(4, 6) and study behavior at 1000 F. Upon completion of this work advanced, complex creep/fatigue tests of Type 304 stainless steel will be conducted. The objective of these advanced tests will be to develop useful damage relationships for conditions of combined creep and fatigue damage.

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- (2) Slot, T., and Stenz, R. H., "Experimental Procedures for Low-Cycle Fatigue Research at High Temperatures", Exp. Mech., 8 (3), 107-114 (1968).
- (3) Slot, T., Stenz, R. H., and Berling, J. T., "Controlled-Strain Testing Procedures", Manual on Low-Cycle Fatigue Testing, ASTM-STP-465, American Society for Testing and Materials, Philadelphia (1969), pp 100-128.
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- (5) Conway, J. B., "Short-Term Tensile and Low-Cycle Fatigue Properties of Incoloy 800", GEMP-732 (1969).
- (6) Conway, J. B., "An Analysis of the Relaxation Behavior of AISI 304 and 316 Stainless Steel at Elevated Temperature", GEMP-730 (1969).

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

R. A. Wullaert and A. A. Bauer

This program is being carried out for the AEC Division of Reactor Development and Technology to determine the effects of reactor environments on various reactor cladding and structural materials. The portion of the program involving irradiation effects is directed toward obtaining an understanding of the radiation-induced high-temperature embrittlement observed in potential cladding materials for liquid-metal fast breeder reactors (LMFBR). Emphasis is being placed on the irradiation behavior of AISI Type 347 and Type 316 stainless steel.

The program is divided into efforts concerning (1) the mechanical properties of Type 347 stainless steel irradiated to very high thermal and fast fluences, (2) the effect of microstructure on the radiation-induced high-temperature embrittlement, (3) the development and evaluation of new techniques for determining radiation-induced mechanical property changes, (4) the effect of irradiation temperature, dose rate, and flux spectrum on the mechanical properties of cladding materials, and (5) the evaluation of requirements for and the determination of properties of LMFBR structural and component materials.

An Investigation of New and Unusual Test Techniques for Determining
the Mechanical Properties of Irradiated Cladding Materials

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M. Kangilaski, and R. A. Wullaert

Objective of Program

To determine the radiation-induced changes in mechanical properties of cladding materials by techniques not requiring extensive specimen preparation.

Approach Being Used

Materials previously irradiated and readily available will be used in these experiments. These will include stainless steel capsule components, stainless steel cladding from fuel irradiation programs, and miscellaneous irradiated structural materials. Whenever possible, unstrained portions of previously tested specimens (that is, tensile-specimen button heads) will be used, since their mechanical properties have been determined by conventional tests.

The techniques used will involve microhardness tests to develop stress-strain curves and tensile tests on ring specimens. In developing these techniques, special emphasis will be placed on understanding the state of stresses that are present during testing and the testing variables which may influence the state of stresses.

The stress-strain curves will be developed by using variable-angle cone indenters for hardness tests. This technique will allow a stress-strain relation of the form $S = Ae^n$ to be developed without resorting to conventional mechanical testing. In this test, the ratio of the applied load to the projected area of the indentation is used as the hardness value. Each conical indenter is calibrated to obtain two constants, ϵ_θ and c_θ . The ratio of the hardness to c_θ is considered to be the stress that causes an additional strain of ϵ_θ in the material being tested. By using a series of cone indentations, the stress-strain relation can be developed and radiation-induced changes studied.

Small sections of tubing will be tested by inserting two D-shaped grips into the ring of tubing and pulling the ring in tension. In order to understand the tensile testing of ring specimens, studies will be performed to determine the stress and strain distributions in the ring during testing. Effects of testing variables such as strain rate and bending (straightening of effective gage section) during testing will be investigated.

Significant Accomplishments During Fiscal Year 1970

The state of stress in a ring-type specimen during tensile testing was established by an elastic-plastic, finite-element, computer stress calculation. An approximate calculation treating the ring as a flexible membrane was also performed. It was found that the stress and strain distribution during the test depended on the friction between the ring and the D-section grip for narrow ring specimens (BMI-1872). It was concluded that unless the strain-hardening exponent, n , is very high, or a way is found to make the friction coefficient, μ , reproducibly very low (so that the ratio of μ/n is not more than a few tenths), significant variations in the load-elongation behavior defined by this test from that of a uniaxial tensile test will occur. Therefore, it was decided that this was not a good test for irradiated specimens, and efforts on this test were discontinued.

Variable-angle cone hardness indenters were used to obtain data representing the stress-strain behavior of stainless steel. This technique was successfully applied to unirradiated material but an adverse scatter in the data occurred when the technique was applied to irradiated material.

Summary of Research Carried Out

Tensile tests on ring-shaped sections of tubing provide a possible means of determining conventional tensile strength and elongation, if the state of stress and strain in the ring is known. The specimen deformation was evaluated by two types of analyses. The first was an elastic-plastic finite-element, computer stress calculation and the second was an approximate calculation treating the ring as a flexible membrane. The finite-element analysis required no assumptions concerning the state of stress in the ring and thus could show any bending action. In addition, it showed the exact elastic stresses during the initial part of the test, and the subsequent transition to the plastic state with the corresponding changes in the stress distribution. However, in this finite-element analysis, frictional effects could not easily be included and it was limited to small strains (i. e., changes in the cross-sectional area were not included). The finite-element analysis nevertheless did show that the effect of yielding was to reduce bending stresses to negligible levels. This suggested the validity of a membrane analysis, for which a simple closed-form solution was possible. While the approximate solution ignored bending stresses, it did include frictional effects and was applicable to arbitrarily large deformations.

A finite-element grid of a quadrant of the ring was constructed and Battelle's computer program FEELAP was used to perform an elastic-plastic stress analysis. Both plane and axisymmetric bodies of arbitrary cross section can be analyzed with the stress and displacement boundary conditions permitted. The von Mises yield criterion is used with generalized strain hardening.

The tubing selected for analysis was 0.995 in. in ID with a wall thickness of 0.065 in. The inside mandrel or D-section was assumed rigid and initially fitting the tubing section. Although it is expected that friction plays a role, zero friction between the tubing and D-sections was assumed to minimize the number of variables. A state of plane stress in tubing sections was assumed to model a narrow ring. Because of symmetry conditions it was necessary to model only one quadrant of the ring, which was divided into 300 finite elements. The material was assumed to follow the stress-strain behavior of annealed Type 304 stainless steel at 650 C (yield strength of

22,500 psi). Successive displacements of the D-sections were the input, while stress and strain distributions were the outputs.

Prior to plastic yielding, significant bending stresses were noted near the plane of D-section separation, with the highest stresses occurring on the inside of the ring where the load tends to straighten the curved ring. Plastic yielding was imminent at the D-separation of 0.000718 in. Figure B-1 shows the growth of the plastic zone with increasing D-separation and load. At a separation of 0.00161 in., the entire ring had yielded. As the plastic zone spread through the ring, the bending stresses at the mid-plane decreased, becoming negligible at the small strain corresponding to general yielding of the ring. Hence, this finite-element analysis shows that for rings having a diameter-to-thickness ratio near those analyzed, bending stresses may be ignored, and reliable results obtained from a simple membrane analysis.

FIGURE B-1. GROWTH OF THE PLASTIC ZONE WITH INCREASING SEPARATION (δ) OF THE D-SECTIONS

The simple membrane analysis followed reasoning similar to that of Spotts⁽¹⁾. If bending stresses are neglected the forces in the ring are as shown in Figure B-2. On each element of length $Rd\theta$, there acts a tension, T , a normal force,

$$P = Td\theta, \quad (B-1)$$

and a friction force,

$$F = \mu P = \mu Td\theta, \quad (B-2)$$

where μ is the coefficient of friction.

Summation of forces in the tangential direction and subsequent solution of the resultant differential equation shows that T decreases from T_0 to $T_0 \exp(-\mu\pi/2)$ at the top of the ring.

The engineering stress, σ , in the ring was found by dividing T_0 by the initial cross-sectional area:

$$\sigma(\theta) = \sigma_o \exp(-\mu\theta) , \quad (B-3)$$

which is independent of the stress-strain behavior of the material. This result shows that the stress in the ring is nonuniform and that its distribution is dependent on the coefficient of friction, μ , between the ring and the D-section.

If the material shows a definite yield strength, σ_y , followed by work hardening to a strength level $\sigma_o > \sigma_y$, the plastic zone from $\theta = 0$ to $\theta = \theta_p$ is determined by the ratio σ_o/σ_y and the friction coefficient, μ , according to the relation

$$\mu \theta_p = \ln(\sigma_o/\sigma_y) . \quad (B-4)$$

The growth of the plastic zone with increasing σ_o is shown in Figure B-3.

Closed-form solutions for the load-elongation curve of this ring test are obtainable for various assumed uniaxial stress-strain curves. For the stress-strain relation:

$$\sigma = B\epsilon^n , \quad (B-5)$$

where B and n are constants characteristic of the material, it can be shown that the total elongation, δ , is given by

$$\delta = 2R \left(\frac{\sigma_o}{B} \right)^{1/n} \frac{n}{\mu} [1 - \exp(-\pi\mu/2n)] , \quad (B-6)$$

where R is the ring radius.

The load can be found as $2\sigma_o$ times the original cross-sectional area of the ring. This expression shows that the slope of the load versus elongation in a ring test conducted on this type of material plotted on log-log paper is the same as that of a uniaxial test, independent of the friction coefficient μ , and offers an unambiguous method for determination of the strain-hardening exponent.

The ring elongation result was compared with that of a uniaxial tensile test of the same material; this revealed the nonuniformity of the strain distribution to be expected around a ring, and the interdependence of the ring to D-section friction coefficient, μ , and the work-hardening exponent, n_1 , in determining the shape of the load-elongation curve.

Another factor analyzed was the strain-rate distribution in the ring for a constant elongation rate, r . Through use of the assumption of strain-rate-independent mechanical properties, it can be shown that the strain rate as a function of the angle θ in the ring test is given by

$$\dot{\epsilon}(\theta) = \frac{\dot{\epsilon}_1 L}{2R} \psi , \quad (B-7)$$

where $\dot{\epsilon}_1$ is uniaxial strain rate, and

$$\psi = \frac{\mu \exp(-\mu\theta/n)}{n[1 - \exp(-\pi\mu/2n)]} . \quad (B-8)$$

Hence, variations in strain rate similar to those in total strain exist for materials following this strain-hardening law. If the material properties are dependent upon strain rate, in the direction of increasing the flow stress with increasing strain rate, then the resultant strain distribution should be more uniform than predicted by this expression.

In summary, the preliminary analysis performed on the ring tensile test has shown the nature of the dependence of the stress and strain distribution in the ring on friction between the ring and the D-section grip for narrow ring specimens. It may be

FIGURE B-2. MEMBRANE ANALYSIS OF RING

FIGURE B-3. GROWTH OF PLASTIC ZONE WITH STRESS GREATER THAN THE ELASTIC LIMIT σ_y

concluded that unless the strain-hardening exponent, n , is very high, or a way is found to make the friction coefficient, μ , reproducibly very low (so that the ratio of μ/n is not more than a few tenths), significant variations in the load-elongation behavior obtained in this test from that obtained in a uniaxial tensile test will occur. In addition, a range of strain rates will exist in a single test. However, even though a difference in materials behavior in the ring tensile test as compared with a uniaxial test occurs, useful information regarding the strain-hardening exponent can still be obtained, but only if a standardized surface preparation and lubrication method can be shown to yield a reproducible friction condition on the D-surface would it be possible to obtain all the usual tensile data from this type of test. This would require calibration with materials of known tensile properties and the application of the type of analysis begun in this report period. Therefore, it was decided that this was not a good test for irradiated specimens and this effort was discontinued.

Microhardness tests on small samples provide a possible means of determining conventional stress-strain properties, if the deformation produced by the indenter can be related to the basic flow properties of the material.

Recent work by Atkins and Tabor⁽²⁾ has shown that plastic indentation of a metal with a cone-shaped indenter produces deformation which represents an effective strain of the metal. When a plastic indentation is produced in a material, the latter must clearly suffer some local permanent strain as a consequence of the deformation. The plastic strain field around such an indentation will generally be complicated and will vary from point to point, but can still be described in terms of a "representative strain", characteristic of the indenter and loading conditions. Where geometric similarity holds, this representative strain will be independent of the indentation size; this is true of wedge, cone, and pyramidal indentations.

For fully work-hardened materials, the indentation hardness is a certain multiple of the yield stress of the material. We may write $HN = cY$, where HN is the hardness, Y the yield stress, and c is the constant of proportionality which depends on the geometry of the indenter.

If it is assumed that the value of c remains the same for annealed as for partially work-hardened materials, we may write $HN = cS$, where S is the representative yield stress of the material as augmented by the indentation process itself, that is, the stress required to strain work-hardened material. Clearly, S is always greater than the yield stress of the specimen. Thus, if we locate on the stress-strain curve the yield stress, s_p , of a specimen strained a known amount, ϵ_p , beyond the fully annealed condition and then locate the other yield stress, S_h , obtained from the hardness relationship (i. e., HN/c), this will be found further along the curve, at some strain ϵ_h (i. e., $\epsilon_h > \epsilon_p$). Then, if the material starts in the ϵ_p condition, the strain associated with the indentation process will be approximately $(\epsilon_h - \epsilon_p) = \epsilon_\theta$.

These concepts may be applied to cone indentations so that by employing a series of cones of different angles, therefore imposing different strains on a material, it should be feasible to construct the stress-strain curve of the material by using the stress levels determined from the hardness values and the strain values appropriate to the cones.

For the analysis it is assumed that the variation in cone hardness number (CHN) with cold work can be represented ideally by

$$\log \text{CHN} = \log B + k \log \epsilon_p \quad . \quad (\text{B-9})$$

However, because of the finite deformation accompanying indentation,

$$\log \text{CHN} = \log B + m \log (\epsilon_p + \epsilon_\theta) \quad . \quad (\text{B-10})$$

As discussed earlier, it is assumed that the hardness can be expressed as some multiple of the flow stress, that is,

$$\text{CHN} = c_{\theta} S, \quad (\text{B-11})$$

where the true-flow curve can be represented by

$$S = A \epsilon^n. \quad (\text{B-12})$$

Then if $m = n$,

$$\log B = \log A + \log c_{\theta}, \quad (\text{B-13})$$

with $\epsilon = \epsilon_p + \epsilon_{\theta}$. These relationships are illustrated in Figure B-4.

In order to determine the constants c_{θ} and ϵ_{θ} , five cone indenters having apical angles of 60, 90, 120, 136, and 150 deg were employed. For the calibration, a series of cylindrical compression test specimens was prepared from annealed Type 347 stainless steel. These specimens were compressed to produce samples representing plastic strains (ϵ_p) of 22.0, 26.5, 19.1, 16.6, 10.4, 27.7, and 30.0 percent. The cone hardness of these samples was measured with each indenter. The cone hardness number (CHN) is defined as the ratio of the applied load to the projected area of the resultant indentation. A standard Rockwell hardness tester specially equipped for remote use in hot cell was employed to make the indentations with an applied load of 60 kg. The diameter of the projected area of each indentation was measured with the aid of the optics of a hot-cell metallograph. For each indenter the graphical procedure shown in Figure B-4 was used to find the constants c_{θ} and ϵ_{θ} utilizing the true-flow curve determined by conventional uniaxial tensile tests.

The results of this graphical trial-and-error calibration procedure are as follows:

	Value for Indicated Cone Angle				
	60 Deg	90 Deg	120 Deg	136 Deg	150 Deg
c_{θ}	3.28	3.32	3.48	3.65	3.56
ϵ_{θ} , percent	21.2	17.9	15.2	12.5	7.3

The values of ϵ_{θ} agree favorably with those evaluated previously by Tabor and Atkins. The c_{θ} values are somewhat higher than the expected approximate value of 2.6. However, this should not detract from the utility of the CHN technique.

The indenters were used to obtain CHN data for both the unirradiated, annealed stainless steel and irradiated stainless steel. The above constants c_{θ} and ϵ_{θ} were used to convert these data to equivalent stress-strain values. These results are compared to the uniaxial tensile determined stress-strain curves in Figure B-5. As indicated in the figure, the agreement for the unirradiated material is excellent. However, there is considerable disagreement for the irradiated material. The cause of the discrepancy is not known at this time.

Plans for Future Work

A final report summarizing the unusual test techniques will be issued.

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- (1) Spotts, M. F., *Design of Machine Elements*, Third Edition, Prentice-Hall, Inc., Englewood, New Jersey (1961), p 222.
 - (2) Atkins, A. G., and Tabor, D., "Plastic Indentation in Metals with Cones", *J. Mech. Phys. Solids*, 13, 149-164 (1965).

FIGURE B-4. SCHEMATIC DESCRIPTION OF CONE INDENTER CALIBRATION TECHNIQUE

FIGURE B-5. COMPARISON OF STRESS-STRAIN RELATIONSHIPS FOR UNIRRADIATED AND IRRADIATED TYPE 304 STAINLESS STEEL AS DETERMINED BY VARYING CONE-SHAPED INDENTERS AND BY UNIAXIAL TENSILE TESTS

Correlation of Radiation Effects on Mechanical Properties
as Caused by a Predominantly Fast Flux and a
Mixed Fast and Thermal Flux

M. Kangilaski, A. A. Bauer, and R. A. Wullaert

Objective of Program

To compare the effects of a predominantly fast neutron flux and a mixed fast and thermal neutron flux on the mechanical properties of cladding materials.

Approach Being Used

Tensile and fatigue specimens of the same lot of Type 347 stainless steel irradiated in the ETR to fast fluences of up to 3×10^{22} nvt (>1 Mev) will be irradiated in the EBR-II to equivalent fast fluences. 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).

Tubular specimens of Type 347 stainless steel and tensile specimens of Tenelon, UMC0-50, and Haynes Stellite 25 will also be irradiated in the EBR-II. Tubular specimens of Type 347 stainless steel better simulate the cladding in nuclear applications. A few tensile specimens of Tenelon, UMC0-50, and Haynes Stellite 25 were included to determine the effects of a predominantly fast neutron spectrum on their mechanical properties and to relate these effects to results obtained previously in a mixed neutron flux spectrum.

The irradiated specimens of Type 347 stainless steel will be tested at room temperature and at 315 and 750 C to correlate the effects of predominantly fast neutrons and mixed thermal and fast neutrons on the mechanical properties. Tensile tests on specimens of Type 348 stainless steel will be carried out at elevated temperatures to determine their susceptibility to radiation-induced embrittlement at elevated temperatures.

Since the EBR-II irradiation will take place at elevated temperatures (400 and 650 C) and the original ETR irradiation was conducted at 50 C, it is probable that the different irradiation temperatures will not allow a direct comparison of the postirradiation mechanical properties of Type 347 stainless steel. To permit a direct correlation, a capsule will be irradiated in the ETR with specimen temperatures of 400 and 650 C. The ETR specimens will be tested at room temperature and at 315 and 750 C after irradiation. In addition, some tensile specimens of Type 348 stainless steel and tubular specimens of Type 347 stainless steel will be included in the ETR irradiation capsule.

Significant Accomplishments During Fiscal Year 1970

A relationship was established between the irradiation-induced elevated-temperature embrittlement and the microstructure of Type 347 stainless steel that was irradiated in the ETR at 50 C to a fast fluence (>1 Mev) of 2.1×10^{22} nvt. Transmission electron microscopy (TEM) did not show the presence of helium bubbles in the specimens that were tested as irradiated. However, a 1-hr anneal at 980 C caused the appearance of large (200 to 500 Å) helium bubbles which were located in the matrix as well as grain boundaries. The ductility at 500 C of the irradiated and annealed

material was not significantly influenced by the presence of helium bubbles. It was actually observed by TEM that some of the bubbles at the grain boundaries were sheared without fracture occurring at the grain boundaries. At 600 C the irradiated and annealed stainless steel exhibited considerable embrittlement and no sheared helium bubbles were found on the grain boundaries. It was also observed that embrittlement was more severe at 600 C in the as-irradiated stainless steel (no observable bubbles) than in the irradiated and annealed stainless steel (helium bubbles). Thus, it was concluded that as the helium bubbles grow they achieve a distribution where their effectiveness in causing embrittlement is reduced. The relationship between the microstructure and irradiation-induced embrittlement for the different testing conditions is summarized in Figure B-6.

Specimens of Type 347 and 348 stainless steel were irradiated in the ETR at 400 and 650 C to fast fluences ranging from 3.3×10^{21} to 4.6×10^{21} nvt. Mechanical property test results from these specimens were compared with those from earlier studies where specimens from the same heat of Type 347 stainless steel were irradiated at 50 C in the ETR to a fast fluence of 5.5×10^{21} nvt⁽¹⁾. The comparisons indicated that irradiation-induced changes in tensile properties are dependent on irradiation temperature. For low-temperature tests (<450 C) the changes in tensile properties are at the maximum for irradiation at 400 C, intermediate for irradiation at 50 C, and smallest for irradiation at 650 C. The elevated-temperature embrittlement was found to start at testing temperatures above 450 C, while at 750 C embrittlement was found to be independent of irradiation temperature.

Helium analyses were obtained on Type 347 and Type 348 stainless steel specimens irradiated in the ETR. The analyses indicated much higher helium contents than could be calculated on the basis of available cross-sectional data. A possible explanation for the discrepancy has been advanced by Weitman⁽²⁾, who suggests that thermal neutrons transmute nickel to an isotope which in turn has a high (n, α) cross section to neutrons. Extrapolation of Weitman's data by two orders of magnitude produced remarkable agreement with helium analyses on irradiated specimens.

Summary of Research Carried Out

Transmission-electron-microscopy (TEM) studies were performed to characterize the structure of Type 347 stainless steel specimens that had been irradiated to a fast fluence of 2.1×10^{22} nvt at 50 C in the ETR. These specimens have been tested in tension at temperatures ranging from 500 to 600 C and exhibited various degrees of ductility, as summarized in Table B-1.

TABLE B-1. PROPERTIES AND TESTING CONDITIONS OF TYPE 347 STAINLESS STEEL SPECIMENS EXAMINED BY TEM

Fast Fluence, 10^{22} nvt	Specimen Condition	Test Temperature, C	Uniform Elongation, percent	0.2 Percent Offset Yield Strength, psi
0	Solution annealed	600	17.8	23,000
2.1	Annealed for 1 hr at 980 C	500	20.0	28,000
2.1	As irradiated	525	16.0	51,500
2.1	Annealed for 1 hr at 980 C	600	5.2	22,300
2.1	As irradiated	600	0.7	34,600

FIGURE B-6. RESPONSE OF DIFFERENT UNIRRADIATED AND IRRADIATED TYPE 347 STAINLESS STEEL MICROSTRUCTURES TO TENSILE TESTING AT ELEVATED TEMPERATURES

The unirradiated specimen that was tested at 600 C had a tensile yield strength of 23,000 psi and a uniform elongation of 17.8 percent. A representative transmission electron photomicrograph of this specimen is presented in Figure B-7. The predominant feature of this microstructure is the dislocation network or cell structure which is approximately 0.5μ in size. The examination also revealed twins, stacking faults, slip traces, and inclusions.

30,000X

301

FIGURE B-7. MICROSTRUCTURE OF UNIRRADIATED TYPE 347 STAINLESS STEEL TESTED IN TENSION AT 600 C

Note the cell structure, which is 5μ in size.

Specimens that were tested at 525 and 600 C in the as-irradiated condition were also examined by TEM. The tensile elongation for the as-irradiated Type 347 stainless steel tested at 525 C was slightly less than that of the unirradiated material, but at 600 C it was significantly less. TEM studies of these two specimens 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 observed. The occurrence of twins, inclusions, slip traces, and stacking faults was similar to that observed in the unirradiated specimens.

After the Type 347 stainless steel had been irradiated and then annealed at 980 C for 1 hr, it exhibited tensile properties similar to those of the unirradiated material when tested at temperatures up to 500 C. However, examination of this material by TEM, as illustrated in Figure B-8a, revealed a significant difference in structure when compared to the unirradiated or as-irradiated specimens. This was the presence of a general distribution of 200 to 500-A-diameter bubbles. It was concluded that these 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. The bubbles were observed to be fairly uniformly distributed throughout the material, but with a tendency to be located in grain and twin boundaries. The bubbles were also observed at triple points and were not generally found to be associated with the inclusions. A very interesting observation is the shearing of some of the bubbles by grain-boundary and twin-boundary shear. The

occurrence of twins, slip traces, stacking faults, and inclusions was not significantly different from that observed in the unirradiated material while the dislocation cell structure seen in the annealed specimen tested at 600 C, as described earlier, was not observed. However, point defects produced by irradiation were visible.

Testing of identical irradiated and annealed material at higher temperatures resulted in a substantial decrease in the tensile elongation, while the yield strength remained similar to that of unirradiated material. TEM of a tensile specimen tested at 600 C revealed a dislocation cell structure similar to but less prevalent than that in the unirradiated material, but with bubbles concentrated in the cell boundaries (Figure B-8b). General observations of the bubbles in this material were similar to those for the material tested at 500 C. The occurrence of twins, substructure, and inclusions was similar to that observed with the unirradiated material.

Figure B-6 provides a summary of the finding by incorporating the elongation values as well as the typical TEM observations into a single figure.

The dislocation cell structure that was observed in the unirradiated specimen was not observed at all in the as-irradiated specimens tested at 525 or 600 C. The cell structure was present in only the irradiated specimen which was annealed for 1 hr at 980 C and then tested at 600 C. The 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 is increased. Cross-slip, and, consequently, cell formation, also is hampered by precipitates⁽³⁾. Thus, it might be expected that irradiation-induced loops, fine 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 transmutation products that are produced during long-time irradiations (see BMI-1845, an earlier report in this series) may reduce the stacking-fault energy and thus minimize the tendency toward the cell-structure formation. Reduction of the tendency toward cell formation by irradiation could cause significant changes in the deformation mechanisms in Type 347 stainless steel.

The shearing of bubbles at grain boundaries was noticed only in the irradiated specimen that was annealed at 980 C for 1 hr and then tested at 500 C. No shearing of bubbles was observed in the specimen that was annealed for 1 hr and tested at 600 C. It is possible that sheared bubbles were not observed in the specimen tested at 600 C because of the small amount of thin foil that was examined or because of the lower deformation experienced by this specimen. However, it may also be related to the fact that at 500 C the bubbles, when sheared, do not promote intergranular fracture, while at 600 C they do. This behavior is consistent with the tensile data which show high ductility and transgranular fracture at 500 C associated with the presence of sheared bubbles, while at 600 C, where no bubble shearing is observed, the fracture mode is intergranular and the ductility is considerably less.

Since the degree of embrittlement at 600 C was less when helium bubbles were observed, and the presence of helium bubbles did not seriously impair ductility at 500 C, it appears that once helium bubble growth occurs, irradiation-induced elevated-temperature embrittlement is reduced. Thus helium bubbles, perhaps smaller than 50 Å and therefore not observable, while perhaps responsible for embrittlement, lose their effectiveness in promoting embrittlement as they become larger, producing an overaging effect. This behavior can be explained in terms of an irradiation-induced aggravation of the ductility minimum, as was discussed in a previous report (BMI-1845). A ductility minimum is observed in unirradiated stressed materials when grain-boundary shearing occurs and fissures form in the grain boundaries; these grain-boundary fissures promote intergranular fracture. Grain-boundary migration tends to minimize the

150,000X

455

a. Specimen Tested at 500 C (Uniform Elongation = 20 Percent)

Bubbles in the grain boundaries sheared due to grain-boundary shear.
Note the bubble at the triple point.

100,000X

486

b. Specimen Tested at 600 C (Uniform Elongation = 5.2 Percent)

A dislocation network is shown with the bubbles pinning the network.

FIGURE B-8. IRRADIATED TYPE 304 STAINLESS STEEL SPECIMENS WHICH WERE ANNEALED AT 980 C FOR 1 HR AND THEN TESTED IN TENSION AT VARIOUS TEMPERATURES

effect of these fissures by continuously healing them. The presence of small helium bubbles in the grain boundaries will tend to pin the boundaries and prevent their movement and thus promote intergranular fracture. However, as the bubbles increase in size, they will become less effective in pinning grain boundaries and, with boundary migration easier, reduce the tendency for intergranular fracture. The experimental findings are in accord with this hypothesis since the ductility of the irradiated material is improved when large, visible helium bubbles are formed.

Alternatively, it has been suggested that helium may cause intergranular fracture in stainless steel because of small bubbles formed within the grains which impede dislocation movement and consequently strengthen the grains⁽⁴⁾. Although the grain boundaries are not directly affected, they become weaker relative to the grain interior, so that failure occurs in the grain boundary. The experimental findings of reduced embrittlement and yield strength with increased helium bubble size can also be explained by this mechanism. As bubbles coalesce and their size increases (concentration decrease), the bubbles become less effective in pinning dislocations and the dislocations move at lower stresses. Consequently, the irradiated specimens have higher ductility and less strength after growth of helium bubbles has occurred.

Tensile tests were performed on irradiated Type 347 and 348 stainless steel specimens at temperatures ranging from room temperature to 750 C. These specimens had been irradiated at either 400 or 650 C in the ETR to fast fluences (>0.1 Mev) ranging from 3.3×10^{21} to 4.6×10^{21} nvt. The tensile properties of unirradiated and irradiated Type 347 stainless steel specimens are given in Table B-2, while Table B-3 gives the tensile properties of Type 348 stainless steel specimens. Table B-2 also includes previously reported test results on Type 347 stainless steel specimens that were irradiated at 50 C to a fast fluence of 5×10^{21} nvt⁽¹⁾. 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-9 through B-13.

The tensile properties of irradiated Type 347 and 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 elongation and total elongation when testing is done above about 500 C. Reduction-in-area values are slightly reduced by irradiation when testing is done at 315 C. However, at higher testing temperatures the reduction-in-area values are drastically reduced by irradiation.

Selected specimens were annealed after irradiation for 1 hr at 980 C to see if the anneal would restore the preirradiation 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 was carried out on selected unirradiated and irradiated specimens that were tested in tension. Of special interest in this examination was the

TABLE B-2. TENSILE PROPERTIES OF UNIRRADIATED AND IRRADIATED TYPE 347 STAINLESS STEEL

Test Temperature, C	0.2% Offset Yield Strength, 1000 psi	Ultimate Strength, 1000 psi	Uniform Elongation, percent	Total Elongation, percent	Reduction in Area, percent
<u>Unirradiated Specimens</u>					
25	37.0	90.0	52.0	64.0	79.0
315	26.8	64.2	31.9	36	72
500	24.0	47.5	22.4	26.6	64
600	23.0	40.5	17.8	22.5	55
750	22.0	33.0	16.0	26	45
<u>Specimens Irradiated at 50 C</u>					
25	88.7	111.5	24.7	32.7	70
315	80.0	84.0	13.2	16.4	60
<u>Specimens 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>Specimens 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>Specimens Irradiated at 650 C and Then Annealed for 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-3. TENSILE PROPERTIES OF UNIRRADIATED AND IRRADIATED TYPE 348 STAINLESS STEEL

Test Temperature, C	0.2% Offset Yield Strength, 1000 psi	Ultimate Strength 1000 psi	Uniform Elongation, percent	Total Elongation, percent	Reduction in Area, percent
<u>Unirradiated Specimens</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>Specimens 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>Specimens 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>Specimens Irradiated at 650 C and Then Annealed for 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

FIGURE B-9. EFFECT OF IRRADIATION ON THE YIELD STRENGTH OF TYPE 347 AND TYPE 348 STAINLESS STEEL

FIGURE B-10. EFFECT OF IRRADIATION ON THE UNIFORM ELONGATION OF TYPE 347 AND TYPE 348 STAINLESS STEEL

FIGURE B-11. REDUCTION IN AREA OF TYPE 347 AND TYPE 348 STAINLESS STEEL

FIGURE B-12. EFFECT OF IRRADIATION ON THE ULTIMATE STRENGTH OF TYPE 347 AND TYPE 348 STAINLESS STEEL

FIGURE B-13. EFFECT OF IRRADIATION ON THE TOTAL ELONGATION OF TYPE 347 AND TYPE 348 STAINLESS STEEL

250X

3C310

FIGURE B-14. FRACTURE APPEARANCE OF UNIRRADIATED TYPE 348 STAINLESS STEEL TESTED IN TENSION AT 500 C

The total elongation of this specimen was 27 percent.

250X

HC23669

FIGURE B-15. 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

The total elongation of this specimen was 4.9 percent.

250X

3C675

FIGURE B-16. FRACTURE APPEARANCE OF UNIRRADIATED TYPE 347 STAINLESS STEEL TESTED IN TENSION AT 750 C

The total elongation of this specimen was 26 percent.

250X

HC23679

FIGURE B-17. 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

This specimen underwent a total elongation of 3.7 percent.

appearance of the fracture area. Figure B-14 shows that the unirradiated specimen that 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-15 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 a large number of voids are visible at grain boundaries.

Figures B-16 and B-17 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 intergranular fracture but without elongation of the grains. There are also a large number of cracks near the fracture surface and at the side of the specimen. These cracks in the irradiated specimens are considerably different from the voids in the unirradiated specimen that was tested at 750 C. In the irradiated specimens the cracks are grain-boundary separations perpendicular to the stress direction while the voids in the unirradiated specimen are rodlike in appearance and aligned parallel with the direction of stress.

Replicas were obtained from the fracture surfaces of selected specimens. For comparison, replicas were also obtained from unirradiated stainless steel specimens that were tested in tension at 500 and 750 C. A typical fracture surface of the unirradiated specimens is illustrated in Figure B-18. The unirradiated specimens fractured transgranularly with considerable plastic flow, as indicated by the presence of dimples.

7,500X

900

FIGURE B-18. APPEARANCE OF FRACTURE SURFACE FOR UNIRRADIATED TYPE 348 STAINLESS STEEL TESTED IN TENSION AT 750 C

The total elongation of this specimen was 35 percent.

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-19 and B-20. 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 large number of dimples on the grain boundaries. It is hypothesized that helium bubbles, enlarged by the annealing at 980 C, may have formed these dimples when they were locally deformed.

The specimen that was irradiated at 400 C and tested at 500 C shows complete intergranular fracture, as shown in Figure B-21a. A large number of small bubbles are seen at the grain boundary. Figure B-21b shows these bubbles at grain boundaries at high magnification.

The relatively greater yield strength and reduced ductility at test temperatures below 750 C for specimens irradiated at 400 C, as compared with those irradiated at 50 and 650 C, are attributed to the combined effects of irradiation-produced helium bubbles and lattice defects. Stable lattice defects are produced both at 50 and 400 C while these anneal out as they are produced during irradiation at 650 C. Conversely, helium bubbles are observed only after irradiation at 400 and 650 C because 50 C is too low a temperature for the formation of bubbles by helium atom diffusion; however, it is suspected that irradiation may accelerate the thermally induced diffusion of helium at 400 C.

The lattice defects and clusters strengthen by interacting with dislocations, and while it is expected that cluster growth at 400 C would lessen their effectiveness relative to 50 C, it is presumed that fine helium bubbles in the matrix also interact with dislocations to supplement the effect of the lattice defects. This strengthening effect is lost with the larger helium bubbles produced during irradiation at 650 C. The low ductility of the material irradiated at 400 C apparently results from the extensive decoration of grain boundaries with helium bubbles which provide a ready path for fracture, even at temperatures (i. e., 500 C) where transgranular fracture normally is observed. When tests are conducted at 750 C, annealing of the lattice defects occurs and the strength is recovered, but the characteristic high-temperature embrittlement phenomenon, resulting from helium bubbles at the grain boundaries which prevent healing of grain-boundary cracks by preventing grain-boundary motion, is encountered for all the irradiated material, regardless of irradiation temperature.

Results of helium analyses performed at Atomics International are given in Table B-4. In addition to the analyzed results, the calculated helium contents are presented. These calculations were based on (n, α) cross-sectional data for neutrons with energies > 1 Mev, as published by Birrs⁽⁵⁾. The results show that the predicted helium content is considerably lower than the analyzed helium content.

TABLE B-4. HELIUM CONTENT OF IRRADIATED TYPE 347 AND TYPE 348 STAINLESS STEEL SPECIMENS

Stainless Steel	Fast Fluence, 10^{21} nvt	Irradiation Temperature, C	Helium Content, ppm (atomic)	
			Calculated	Analyzed
Type 348	3.3	400	10.8	23.5
Type 348	4.4	650	11.7	37.2
Type 347	21.0	50	18.0	917
Type 347	30.0	50	24.1	2016

15,000X

852

FIGURE B-19. APPEARANCE OF FRACTURE SURFACE FOR TYPE 347 STAINLESS STEEL IRRADIATED AT 650 C AND TESTED IN TENSION AT 750 C

The total elongation of this specimen was 3.7 percent.

15,000X

862

FIGURE B-20. APPEARANCE OF FRACTURE SURFACE FOR TYPE 347 STAINLESS STEEL IRRADIATED AT 650 C, ANNEALED FOR 1 HR AT 980 C, AND TESTED IN TENSION AT 750 C

The total elongation of this specimen was 3.6 percent.

7,500X

832

a. Low Magnification

45,000X

833

b. High Magnification

FIGURE B-21. APPEARANCE OF FRACTURE SURFACE FOR TYPE 348 STAINLESS STEEL IRRADIATED AT 400 C AND TESTED AT 500 C

The total elongation of this specimen was 4.9 percent.

However, it has been recently suggested by Weitman⁽²⁾ of Sweden that thermal neutrons make a large contribution to helium production from nickel. It was found that if a cadmium shield is used around nickel specimens (to eliminate thermal neutrons) a considerably smaller amount of helium is produced than when no cadmium shield is utilized. The results of this experiment are plotted in Figure B-22. If the data reported in Table B-4, normalized for nickel content and with the contribution of helium due to (n,α) reactions with boron subtracted, are plotted on this figure, remarkable agreement is obtained with an extrapolation of Weitman's results to higher fluences. Weitman surmises that a nickel isotope is transmuted by thermal neutrons to a new isotope which has a high (n,α) cross section for either fast or thermal neutrons. While proof of this suggestion is lacking, the helium data obtained provide support for such an explanation.

Irradiation was continued on the EBR-II capsule. The number of specimens and the designed irradiation temperatures are enumerated in Table B-5. The capsule will be removed when the EBR-II is shut down for its power increase to 62.5 Mw. The estimated total fluence on the specimens will be about 2×10^{22} nvt.

TABLE B-5. SPECIMENS BEING IRRADIATED IN THE EBR-II

Type	Material	Quantity	Irradiation Temperature, C
Tensile	Type 347 stainless	16	400
Tensile	Type 347 stainless	4	650
Tensile	Type 348 stainless	4	400
Tensile	Type 348 stainless	4	650
Tensile	UMCo-50	6	400
Tensile	Haynes Stellite 25	2	400
Tensile	Tenelon	8	400
Tensile	Telelon	4	650
Fatigue	Type 347 stainless	4	400
Tubes	Type 347 stainless	12	400

Plans for Future Work

The EBR-II capsule containing Type 347 and 348 stainless steel specimens will be returned to the Battelle-Columbus Hot-Cell Facility. Selected specimens will be tested in tension and fatigue. The structural characteristics of these specimens will be examined by metallography, fractography, and transmission electron microscopy. Helium analyses will also be performed on selected specimens. The test results from the specimens irradiated in the EBR-II will be correlated with the test results that were obtained from the specimens irradiated in the ETR to establish the influence of neutron spectrum on irradiation-induced mechanical property changes.

- (1) Kangilaski, M., and Shober, F. R., "The effect of Neutron Irradiation on the Mechanical Properties of AISI Type 347 Stainless Steel", *The Effects of Radiation on Structural Metals*, ASTM-STP-426 (1967), pp 487-511.
- (2) Weitman, J., Aktiebolaget Atomenergi, Sweden, private communication.
- (3) Swann, P. R., "Dislocation Arrangement in Face Centered Cubic Metals and Alloys", in *Electron Microscopy and Strength of Crystals*, edited by G. Thomas and J. Washburn, Interscience Publishers, Inc., New York (1963).
- (4) Woodford, D. A., Smith, J. P., and Moteff, J., "Observation of Helium Bubbles in an Irradiated and Annealed Austenitic Steel", *J. Iron Steel Inst.*, **208**, 70-76 (January, 1969).
- (5) Birrs, I. R., "Helium Production in Reactor Materials", *J. Nucl. Mater.*, **34**, 241-259 (1970).

FIGURE B-22. HELIUM PRODUCTION IN NICKEL DUE TO NEUTRONS

Response of Different Microstructures to Irradiation-Induced
Embrittlement at Elevated Temperatures

M. Kangilaski, A. A. Bauer, and R. A. Wullaert

Objective of Program

To determine the role of the microstructure of cladding and structural materials on irradiation-induced embrittlement at elevated temperatures.

Approach Being Used

Since the elevated-temperature embrittlement of an irradiated stainless steel results in intergranular fracture, a better understanding of the phenomenon might be obtained by investigating grain-boundary effects at elevated temperature after irradiation. Tensile specimens of single crystals (no grain boundaries) of pure nickel will be irradiated and tested at elevated temperatures. Polycrystalline specimens of Type 316 stainless steel with elongated grains oriented parallel, perpendicular, and at 45 deg to the tensile axis will be irradiated and tested at elevated temperatures.

Irradiation at elevated temperatures in a high instantaneous flux has been found to cause the formation of voids, resulting in a reduction in density of various materials. It is believed that this void formation may be minimized by using a structure containing a large number of dislocations, interstitial clusters, subgrain boundaries, etc. This type of high-defect structure is desirable so that vacancies that are formed by irradiation are annihilated before they can agglomerate into voids. To test this hypothesis, specimens of Type 316 stainless steel will be shock hardened (to obtain a high-defect structure) and irradiated.

Significant Accomplishments During Fiscal Year 1970

A capsule containing various tensile specimens was prepared and inserted into the EBR-II for irradiation. This capsule contained nickel single-crystal specimens, specimens of Type 316 stainless steel that had directionally oriented grains, and specimens of shock-hardened Type 316 stainless steel.

It was found that explosive shock strengthening of stainless steel resulted in large increases of yield strength, moderate increases of ultimate strength, and reductions in ductility. The shock hardening caused a reduction in steady-state creep rate by a factor of 1000 at 650 C and a stress of 25,000 psi.

Summary of Research Carried Out

Tensile specimens were prepared from three castings of Type 316 stainless steel that were directionally solidified to obtain elongated grains. X-ray diffraction studies were performed on the three different castings of Type 316 stainless steel. It was established that the axes of the elongated grains were approximately in the $\langle 100 \rangle$ direction for all three castings. Chemical analyses indicated that all three castings had similar chemical compositions. Earlier observations had shown coring in the as-cast microstructure, but this coring was removed by a 24-hr anneal at 1100 C. Electron-beam microprobe analysis of the cast material after the 24-hr heat treatment indicated that the distribution of iron, nickel, chromium, and molybdenum was uniform between the grains and grain boundaries.

The sheet specimens were machined to have the grains either parallel, perpendicular, or 45-deg inclined to the tensile axis. Figure B-23 illustrates the appearance of the specimens with the directional grains.

FIGURE B-23. TYPICAL APPEARANCE OF TYPE 316 STAINLESS STEEL SPECIMENS WITH ORIENTED GRAINS

All specimens were etched with 80 cm³ HCl, 20 cm³ HNO₃, and 5 g CuCl₂. The grains in the respective specimens were oriented:

- A. Perpendicular to the tensile axis
- B. 45-deg inclined to the tensile axis
- C. Parallel to the tensile axis.

Tensile tests were performed on the specimens with the oriented grains; the test results are given in Table B-6. These test results show that there is essentially no difference among the tensile properties of specimens with different grain orientations.

TABLE B-6. TENSILE PROPERTIES OF CAST TYPE 316 STAINLESS STEEL SPECIMENS WITH ORIENTED GRAINS

Orientation of Grains with Respect to Tensile Axis	Test Temperature, C	0.2% Offset Yield Strength, psi	Ultimate Strength, psi	Uniform Elongation, percent	Total Elongation, percent
Parallel	600	8,700	32,400	47	56
	675	8,000	28,400	48	61
	750	6,540	21,500	24	46
Perpendicular	600	9,230	35,600	56	60
	675	8,100	24,800	50	62
	750	7,300	20,300	23	51
	850	5,720	11,600	9.5	46
Inclined 45 deg	600	8,530	35,000	47	54
	675	8,140	25,600	39	45

Specimens of nickel single crystals, Type 316 stainless steel specimens with oriented grains, and shock-hardened Type 316 stainless steel specimens were encapsulated for irradiation. Table B-7 enumerates the number and types of specimens as well as the proposed irradiation temperatures. The capsule was shipped to the EBR-II and will be inserted when the EBR-II power level is changed to 625 Mw.

TABLE B-7. PROPOSED IRRADIATION CONDITIONS FOR SPECIMENS TO BE IRRADIATED IN THE EBR-II

Material	Condition	Quantity of Specimens to be Irradiated at Indicated Temperature		
		450 C	550 C	650 C
Nickel	Single crystals	4	4	4
Type 316 stainless steel	Longitudinal grains	4	4	4
	Transverse grains	4	4	4
	45-deg-inclined grains	4	4	4
	Unshocked	4	4	4
	Shocked at room temperature	2	2	2
	Shocked at 665 C	3	3	3
	Shocked at room temperature ^(a)	3	3	3

(a) Annealed for 1 hr at 750 C.

Type 304 and Type 316 stainless steel were shock strengthened by an explosive pressure of 320 kbar. The microstructure of the shock-strengthened stainless steel was studied by light metallography and transmission electron microscopy. Examples of the microstructure are illustrated in Figure B-24. The microstructure consisted of an unresolved dislocation network and fine striations. By means of electron diffraction examination these striations were identified as mechanical twins in the (111) plane.

Tensile tests were performed on as-shocked specimens and shocked specimens that were annealed before testing. The tensile properties at room temperature, 500 C, and 650 C are summarized in Figures B-25, B-26, and B-27, respectively. It is apparent that shocking causes a large increase in yield strength, a moderate increase in ultimate strength, and a reduction in ductility. These trends are similar for both room-temperature tests and elevated-temperature tensile tests. By annealing the shock-hardened specimens before testing the strength is reduced while the ductility is increased. Thus it appears that different combinations of strength and ductility can be obtained by appropriate heat treatment after shock hardening. It is interesting to note that a 1-hr anneal at 875 C does not remove the effects of shock hardening on tensile properties when the testing temperature is 650 C, but a 1-hr anneal at 900 C does. Metallographic examination of these tensile specimens indicated that recrystallization had occurred in the specimen that was annealed at 900 C before testing.

Limited creep tests were performed at 650 C and a stress of 25,000 psi on shocked and unshocked stainless steel specimens. Comparison of the creep curves for the shocked and unshocked material is given in Figure B-28. The unshocked stainless steel specimen failed in 54 hr after an elongation of 42 percent, while the shocked stainless steel specimen elongated only 1.1 percent after 820 hr. At that time the specimen had not failed and the test was discontinued. The steady-state creep rate of Type 304 stainless steel was reduced by a factor of 1000 by shock hardening.

750X

4E271

a. Light Metallography

30,000X

TEM

b. Transmission Electron Microscopy

FIGURE B-24. MICROSTRUCTURE OF SHOCK-HARDENED TYPE 316 STAINLESS STEEL

The lines were identified as mechanical twins in the (111) plane.

FIGURE B-25. ROOM-TEMPERATURE TENSILE PROPERTIES OF TYPE 304 STAINLESS STEEL

FIGURE B-26. TENSILE PROPERTIES OF TYPE 304 STAINLESS STEEL AT 500 C

FIGURE B-27. TENSILE PROPERTIES OF TYPE 304 STAINLESS STEEL AT 650 C

FIGURE B-28. CREEP CURVES FOR TYPE 304 STAINLESS STEEL
AT 650 C WITH A STRESS OF 25,000 PSI

To establish the stability of a shock-hardening-induced defect structure, hardness measurements were performed on specimens after annealing. Figure B-29 illustrates the effect on the microhardness of 1-hr anneals at different temperatures. Annealing at temperatures of 600 C and below for 1 hr causes a slight increase in the microhardness of the shock-hardened material. However, at higher annealing temperatures the microhardness is progressively decreased, the decrease being most pronounced at 850 C and above.

Long-time annealing treatments were also performed at 450, 700, 750, and 800 C with the results shown in Figure B-30. The anneal at 450 C caused slight increases in hardness and was discontinued after 1750 hr. Annealing at 800 C for 200 hr resulted in almost complete removal of the shock-hardening-induced hardness increase. The anneal at 700 C resulted in some hardness decreases, but after 1500 hr the hardness was still considerably higher than that of the unshocked material. The anneal at 750 C has not been in progress sufficiently long to draw any significant conclusions. Thus, it appears that the shock-hardened stainless steel is capable of retaining the defect structure, which contributes to its strength, after extended times at temperatures at least to 700 C.

Plans for Future Work

After the target fluence of 1×10^{22} nvt has been reached, the capsule will be removed from the EBR-II and the specimens will be tested.

Shocking pressures of 80 and 160 kbar will be utilized to shock harden Type 316 stainless steel. After shock hardening, the tensile properties and stability of the defect structure at elevated temperatures will be evaluated. If the properties appear to be promising, specimens of the shock-harden material will be prepared for irradiation in the EBR-II.

Evaluation of Low-Carbon Ferritic 2-1/4 Cr-1 Mo Steel

M. Kangilaski and A. A. Bauer

Objective of Program

To investigate 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.

Approach Being Used

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 program.

FIGURE B-29. EFFECT OF 1-HR ANNEALS ON THE MICROHARDNESS OF SHOCK-HARDENED TYPE 304 STAINLESS STEEL

FIGURE B-30. EFFECT OF LONG-TIME HEAT TREATMENTS ON THE HARDNESS OF SHOCK-HARDENED TYPE 316 STAINLESS STEEL

Material from two experimental heats of 2-1/4 Cr-1 Mo alloy containing 0.012 and 0.046 w/o carbon will be obtained from Climax-Molybdenum Company. These are to be creep-rupture tested at temperatures ranging from 950 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. Material is also to be obtained from three large heats of 2-1/4 Cr-1 Mo alloy material, cast and fabricated by the Bureau of Mines, containing 0.015 to 0.08 w/o carbon. Specimens will be prepared; tensile, creep rupture, and other tests will then be performed as are required to evaluate these alloys for LMFBR use. The results will be evaluated against data for standard commercial and niobium-stabilized materials.

Significant Accomplishments During Fiscal Year 1970

Creep tests were initiated on low-carbon ferritic steel, 2-1/4 Cr-1 Mo alloy received from Climax-Molybdenum. Initial minimum creep-rate and stress-rupture data have been obtained.

Summary of Research Carried Out

Bars of 2-1/4 Cr-1 Mo alloy material containing 0.012 or 0.046 w/o carbon were received from Climax-Molybdenum. Creep-rupture specimens were machined from the bars.

Experimental tensile and creep-rupture data for these materials, supplied by the Climax-Molybdenum Company Research Laboratory, were 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.

TABLE B-8. STATUS OF CREEP TESTS ON LOW-CARBON 2-1/4 Cr-1 Mo FERRITIC ALLOYS

Carbon Content, w/o	Test Temperature, F	Stress, 1000 psi	Time in Progress ^(a) , hr	Minimum Creep Rate, hr ⁻¹
0.012	900	20	2450 (10,000)	0.00002
0.012	950	19.5	1080 (2,000)	0.0001
0.012	950	16.3	2500 (10,000)	0.000065
0.012	1000	18	973.3 ^(b) (800)	0.00075
0.012	1000	15.5	-- (2000)	--
0.012	1000	12	2570 (10,000)	0.00007
0.012	1100	8.7	1340 (2,000)	0.008
0.046	900	26	2520 (10,000)	0.000025
0.046	950	25	700 (2,000)	--
0.046	950	20	2470 (10,000)	0.000025
0.046	1000	22.7	1271.1 ^(b) (800)	0.00033
0.046	1000	19.7	1170 (2,000)	0.00009
0.046	1000	15.5	2575 (10,000)	0.00006
0.046	1100	11.2	1320 (2,000)	0.0031

(a) Expected rupture life in parentheses.

(b) Ruptured.

Test temperatures and stresses were selected on the basis of a parametric analysis of the Climax-Molybdenum data and creep-rupture tests were initiated. The status of the tests and initial rupture and minimum creep-rate data are shown in Table B-8 along with creep-rupture life estimates which resulted from the parametric analysis. The rupture life of the two failed specimens is somewhat greater than estimated while the creep-rate data are in general accord with values determined by the Climax-Molybdenum Company.

Plans for Future Work

The creep tests on the alloys containing 0.012 and 0.046 w/o carbon will be completed. Material from three large heats of 2-1/4 Cr-1 Mo ferritic alloy containing 0.015 to 0.08 w/o carbon will be obtained. Both tensile and creep-rupture tests will be performed on specimens from these alloys.

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