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FAST NEUTRON DAMAGE IN TYPE-304 STAINLESS STEEL AND HIGH-NICKEL ALLOYS

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**FAST NEUTRON DAMAGE IN TYPE-304
STAINLESS STEEL AND HIGH - NICKEL ALLOYS**

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TABLE OF CONTENTS

1. INTRODUCTION	1
2. DESCRIPTION OF EXPERIMENT	1
2.1 Capsule Design	1
2.2 Exposure History	1
2.3 Examination and Disassembly	2
3. RADIATION-INDUCED VOID FORMATION	8
3.1 Immersion Density Measurement	8
3.2 Examination by Transmission Electron Microscopy	9
4. TENSILE TESTING	26
4.1 Specimens and Procedure	26
4.2 Tensile Test Results	26
5. DISCUSSION	37
5.1 Temperature and Fluence Dependence of Void Formation	37
5.2 Effect of Microstructure on Void Formation	42
5.3 Effect of Irradiation and Microstructure on Mechanical Properties	43
ACKNOWLEDGMENTS	45
REFERENCES	45

APPENDICES

A MATERIALS INVENTORY AND EXPOSURE CONDITIONS FOR MT-3 CAPSULE MATERIALS	47
B ANALYSIS OF ERRORS INCURRED IN THE TEM EXAMINATION	55
C RELATION OF VOID VOLUME FRACTION TO VOLUME INCREASE	63
D MT-3 TENSILE TEST RESULTS	64
DISTRIBUTION	67

LIST OF ILLUSTRATIONS

Figure	Title	Page
2-1	MT-3 Capsule As Fabricated	3
2-2	Subassembly XA08 Loading Pattern	4
2-3	Neutron Radiograph of MT-3	5
2-4	Gamma Scan and Diametral Profilometry	6
2-5	Comparison of Capsule Tube and Type-304 Stainless Steel Gamma Scans	6
2-6	Temperature Sentinel Positioning	7
3-1	Comparison of Volume Increase of Capsule Tube As Measured by Density Decrease and Diameter Increase	10
3-2	Location of Transmission Electron Microscopy Specimens with Respect to Temperature Profile	12
3-3	Specimen 04	14
3-4	Specimen A1	15
3-5	Specimen M3	16
3-6	Specimen K1	17
3-7	Specimen I1	18
3-8	Specimen J1	19
3-9	Specimen H1 - Voids in $M_{23}C_6$ Surrounded by Void-Free Matrix	20
3-10	Frequency Distribution of Void Sizes - Specimen A1	21
3-11	Voids and Interstitial Loops in Type-304 Stainless Steel Irradiated at 400°C	22
3-12	Incoloy-800	23
3-13	Hastelloy-X	24
3-14	Inconel-625	25
4-1	Tensile Coupon Used in Fast Neutron Damage Experiments	27
4-2	The Effect of Test Temperature on the Mechanical Properties of Incoloy-800	28
4-3	The Effect of Test Temperature on the Mechanical Properties of Inconel-625	29
4-4	The Effect of Test Temperature on the Mechanical Properties of Hastelloy-X	30
4-5	The Effect of Strain Rate on the Mechanical Properties of Incoloy-800 Tested at 1300°F	31

LIST OF ILLUSTRATIONS (Continued)

4-6	The Effect of Strain Rate on the Mechanical Properties of Inconel-625 Tested at 1300°F	32
4-7	The Effect of Strain Rate on the Mechanical Properties of Hastelloy-X Tested at 1300°F	33
4-8	The Effect of Strain Rate on the Mechanical Properties of Incoloy-800 Tested at 1100°F	34
4-9	The Effect of Strain Rate on the Mechanical Properties of Inconel-625 Tested at 1100°F	35
4-10	The Effect of Strain Rate on the Mechanical Properties of Hastelloy-X Tested at 1100°F	36
5-1	Comparison of Voids Observed by Thin Foil Transmission and Replication Electron Microscopy	38
5-2	Effect of Irradiation Temperature on Void Diameter in Type-304 Stainless Steel	40
5-3	Effect of Irradiation Temperature on Minimum Observed Void Diameter	40
5-4	Effect of Irradiation Temperature on Void Number Density	41
5-5	Volume Increase Related to Irradiation Temperature As Determined by TEM in MT-3	41
A-1	Fluence Profile of MT-3 Materials Capsule	49
A-2	Axial Temperature Distribution of MT-3 Capsule Tube	52
A-3	Axial Temperature Profile, MT-3 Capsule Type-304 Stainless Steel Strip and Tensile Specimens	52
A-4	Temperature Profile of the Tensile Specimens in Region 2 for Reactor Operation at 45 MW-Thermal	54

LIST OF TABLES

Table	Title	Page
2-1	Chemical Composition of Tensile Specimens and Electron Microscopy Disks	2
3-1	Volume Increase of the MT-3 Capsule Tube	9
3-2	Immersion Density Results from Nickel Alloy Tensile Specimens	9
3-3	Location and Disposition of Transmission Electron Microscopy Disks from MT-3	11
3-4	Quantitative Results of Void Measurements in Type-304 Stainless Steel	13
A-1	Materials in MT-3 Capsule	48
A-2	Position of Tensile Specimens in MT-3	48
A-3	Reactor History for MT-3 Materials Capsule - Subassembly XA08	50
A-4	Summary of Irradiation and Thermal Exposure	53
B-1	Standard Deviations	60
D-1	Tensile Test Properties (Unirradiated) Group MT-3	65
D-2	Tensile Test Properties (Irradiated) Group MT-3	66

ABSTRACT

A materials capsule containing Incoloy-800, Hastelloy-X, Inconel-625 tensile specimens, electron microscopy disks and a Type-304 stainless steel supporting strip was irradiated in EBR-II to a peak fluence of 6.4×10^{22} n/cm² over a temperature range of 750 to 1275°F (400 to 690°C).

No voids formed in the Type-304 stainless steel irradiated at temperatures greater than $650 \pm 15^\circ\text{C}$ ($1200 \pm 25^\circ\text{F}$), but void size increased and number density decreased with increasing temperature of irradiation. No voids were found (zero swelling) in Inconel-625.

Precipitation strengthened alloys had higher ultimate strength and 0.2% yield strength after irradiation. All high nickel alloys exhibited reduced ductility after irradiation.

SUMMARY

A capsule designated MT-3 has been irradiated in the Experimental Breeder Reactor-II for 19,280 megawatt-days. Peak total neutron fluence was 6.4×10^{22} n/cm². The capsule, constructed of Type-304 stainless steel, contained tensile specimens and electron microscopy disks of Incoloy-800, Hastelloy-X, and Inconel-625. Temperatures in the capsule ranged from 400 to 690°C (750 to 1275°F) during reactor operation at 45 megawatts-thermal (MW_t).

Disks of the high-nickel alloys and the Type-304 stainless steel specimen support strip were examined by transmission electron microscopy.

Tensile specimens irradiated in the core were tested to failure and compared to tests on unirradiated material. Test conditions included test temperatures of 900, 1100 and 1300°F (482, 593, and 704°C) and strain rates of 2×10^{-4} , 2×10^{-2} , and 2 min^{-1} .

The test results indicate that:

1. In Type-304 stainless steel: No void formation occurs when it is irradiated in a fast neutron flux at temperatures greater than $1200 \pm 27^\circ\text{F}$ ($650 \pm 15^\circ\text{C}$).
2. Void size increases with irradiation temperature and void number density decreases.
3. Very large voids with average diameter greater than 1500Å occur in unstressed material irradiated to fluences greater than 4.0×10^{22} n/cm², $E > 0.1 \text{ MeV}$ at temperatures greater than 600°C (1112°F).
4. Void growth rather than void nucleation is the major contributor to volume increase for Type-304 stainless steel irradiated to fluences greater than 4.0×10^{22} , $E > 0.1 \text{ MeV}$ at temperatures greater than 450°C (842°F).

Regarding High-Nickel Alloys:

5. No voids formed in Inconel-625.
6. Small finely divided precipitates can suppress void formation while large widely spaced precipitates may enhance it.

7. Both Inconel-625 and Hastelloy-X showed higher ultimate strength and 0.2% yield strength after irradiation to $\sim 5 \times 10^{22}$ n/cm², E > 0.1 MeV at temperatures > 1150°F (621°C). Incoloy-800 showed reduced ultimate strength and unchanged 0.2% yield strength.
8. Inconel-625, Hastelloy-X, and Incoloy-800 showed substantially reduced ductility after irradiation.
9. All mechanical properties of irradiated Inconel-625, Hastelloy-X, and Incoloy-800 decreased with increased test temperature. Response of unirradiated material was mixed.
10. At a test temperature of 1300°F (704°C) all mechanical properties of the three alloys (except Incoloy-800 total elongation) increased with increasing strain rate after irradiation. At a test temperature of 1100°F (593°C) all mechanical properties of the irradiated alloys increased with increasing strain rate except the 0.2% yield strength. Response of unirradiated material to variation in strain rate was mixed.

1. INTRODUCTION

The MT-3 experiment was designed in 1966 to evaluate the general effects of exposure to fast neutron flux on the physical and mechanical properties of three high-nickel alloys. At the time the experiment was designed, void formation was not anticipated nor was the merit of the stainless steels in competition with the high-nickel alloys.

The swelling resulting from void formation induced by neutron exposure has now been recognized to be a major potential limiting factor in the design of the core of an LMFBR. The evaluation of the results of the MT-3 experiment has therefore been directed toward void formation and the resulting changes in physical and mechanical properties. Since the stainless steels have now become more important, the analysis of the MT-3 experiment has been broadened to include the Type-304 stainless steel capsule and supporting strip which were originally only incidental component parts of the experiment.

This final report on the MT-3 experiment is confined to the presentation of the data. Some of the implications regarding theories of swelling are included in a paper scheduled for publication in *The Journal of Nuclear Materials*.¹ A companion capsule MT-4 is undergoing further irradiation in EBR-11 toward a goal of 2×10^{23} n/cm².

2. DESCRIPTION OF EXPERIMENT

2.1 CAPSULE DESIGN

The MT-3 capsule was designed primarily as an irradiation vehicle for high-nickel alloy tensile specimens of Inconel-625, Hastelloy-X and Incoloy-800. The unirradiated chemical analysis of these alloys is given in Table 2-1. The tensile specimens were attached to a Type-304 stainless steel* strip which was loaded into a Type 304 capsule tube. The assembly was then filled with argon to provide an inert atmosphere. Figure 2-1 shows a schematic of the capsule and inserts and a photograph of the components prior to final assembly. The stainless strip was attached to the bottom end plug. Also included in the capsule were melting alloy temperature sentinels and pre-cut transmission electron microscopy (TEM) disks. An inventory of components and specimens within the capsule is given in Appendix A.

2.2 EXPOSURE HISTORY

The MT-3 capsule was irradiated in EBR-II at temperatures between 400 and 690°C (750 to 1275°F) in a fast neutron flux. Specimens in the MT-3 capsule were subjected to concurrent accumulation of irradiation and thermal exposure.

2.2.1 Irradiation History

The MT-3 capsule was irradiated in the EBR-II as part of the XA08 subassembly. The accumulated exposure was 19,278 MWd in Row 4, which is equivalent to 6.4×10^{22} n/cm² total fluence.** The exposure time was approximately 428 effective full power days at 45 MW (~10,280 hours). The specific reactor location of the XA08 subassembly was position 4F2. The flux distribution through the XA08 subassembly which contained the MT-3 capsule was dependent on the fuel or driver subassemblies adjacent to XA08 as shown in Figure 2-2.²

Reactor operation history indicated that the MT-3 capsule received an accumulated exposure of 19,278 MWd; the total was comprised of 19% at 50 MW, 54% at 45 MW, and 27% at power levels below 45 MW. These exposures at each power level correspond to a length of time at each temperature. More detail on exposure and neutron fluence are included in Appendix A.

2.2.2 Thermal Exposure

Temperature profile of the MT-3 capsule was analyzed at reactor power levels of 45 MW and 50 MW. There was a temperature gradient along each tensile test specimen length. This was similar for all the regions, but the magnitude and

* A post-irradiation chemical analysis of the strip is given in Table 2-1.

** All fluence values are based on EBR-II calculations modified to correspond to experimental dosimetry results.³ The fluence value reported is for the specific position of the MT-3 capsule.

Table 2-1

**CHEMICAL COMPOSITION OF TENSILE SPECIMENS
AND ELECTRON MICROSCOPY DISKS**

Elements	Incoloy-800 (HH-9696A) ^(a)	Inconel-625 (Y8668) ^(a)	Hastelloy-X (X1-4370) ^(a)	Type-304 Strip ^(b)
C	0.06	0.03	0.10	0.054
Mn	0.89	0.12	0.60	1.5
Fe	45.70	1.86	18.7	69.9
S	0.007	0.007	0.006	—
Si	0.41	0.25	0.85	0.5
Cu	0.13	0.03	—	Trace
Ni	32.03	62.31	Bal	8.95
Cr	20.75	21.98	21.68	18.6
Al	0.10	0.17	—	Trace
Ti	0.21	0.23	—	Trace
Mo	—	8.75	9.17	Trace
Nb + Ta	—	4.24	—	—
B	0.0030	0.0065	0.0040	Trace
W	—	—	0.44	—
Co	—	—	1.87	Trace
N	—	—	—	0.056

(a) Heat numbers; not available for strip

(b) Analysis performed on samples from the strip after irradiation

specific values of each varied. A discussion of the temperature calculations and temperature profiles are given in Appendix A.

2.3 EXAMINATION AND DISASSEMBLY

2.3.1 Post-Irradiation Examination

Post-irradiation examination of the MT-3 capsule employed destructive as well as non-destructive examination techniques. Since the position of each specimen in the capsule was related directly to its accumulated exposure and temperature, extreme care was exercised to ensure accurate evaluation of that position. The procedures and results of this examination are included as follows.

2.3.2 Preliminary Examination

The MT-3 materials capsule was visually examined to note any unusual conditions; none were found. The capsule measured 38.0 inches long. It was observed through the cell window that the capsule was slightly bowed.

2.3.3 Neutron Radiograph

The capsule was neutron radiographed to determine the as-irradiated position of each test section. Although the post-irradiation configuration was similar to the as-assembled design, the insert assembly had moved upward about 5/8 inch. The shift of materials was caused by the tensile failure of the Type 304 strip as indicated in Figure 2-3.

2.3.4 Profilometry

The outside diameter of the capsule tube was measured by rotational profilometry and longitudinal profiles at 0 degrees and 90 degrees. The results of these measurements showed diametral swelling of approximately 0.001 inch in

Figure 2-1. MT-3 Capsule As Fabricated

Figure 2-2. Subassembly XA08 Loading Pattern

the core region (Figure 2-4). The accuracy of this measuring system was checked by profilometry of a known standard of comparable length. As can be seen in the figure, the peak diametral increase occurs about eleven inches above the bottom of the core while the flux peak (as indicated by the capsule gamma scan) occurs about six inches above the bottom of the core.

2.3.5 Gamma Scan of Entire Capsule

The complete capsule was gamma scanned for $E > 0.5$ MeV. It was possible to note the position of each tensile specimen region by the variation in activity levels. The position of each specimen region in the insert assembly corresponded directly with the positions determined by neutron radiography.

2.3.6 Capsule Disassembly and Gamma Scan of Capsule Components

The capsule tube was sectioned at the weld between the top end plug and the capsule tube; multiple cuts were required to remove weld penetration. The insert assembly was then easily removed and the appearance of the assembly revealed no unusual conditions.

As the insert assembly was removed, a portion of temperature sentinel No. 4 fell from its affixed position suggesting that the low-melting temperature alloy had interacted with the Type 304 stainless steel tube (containment tube for the sentinel). There was a powdery reaction product around the broken region.

After removal of the insert assembly, the MT-3 capsule tube was scanned for gamma activity $E > 0.5$ MeV.

The type 304 stainless steel strip which supported the tensile specimens during irradiation was also gamma scanned for $E > 0.5$ MeV. The normalized gamma scans are shown in Figure 2-5. From this comparison it was possible to see the concurrence of the capsule tube gamma scan and the Type 304 strip gamma scan, which indicated that the strip broke early in the irradiation period, and that it was irradiated in the position noted on the neutron radiograph.

Figure 2-3. Neutron Radiograph of MT-3

Figure 2-4. Gamma Scan and Diametral Profilometry

Figure 2-5. Comparison of Capsule Tube and Type-304 Stainless Steel Gamma Scans

Figure 2-6. Temperature Sentinel Positioning

2.3.7 Temperature Sentinels

Low-melting temperature alloys were placed in the MT-3 capsule to provide an indication of the peak temperature at selected locations during irradiation. Temperature sentinels, containing the low melting-temperature alloys were examined to determine the condition of each alloy. The position and composition of each alloy is noted in Figure 2-6; melting of an alloy was determined by metallographic examination. The temperature sentinel examination showed that region 3 reached the melting point of the aluminum which was ~ 1190 to 1210°F (~ 643 to 654°C). The aluminum in region 1 showed signs of reaction, but it did not melt. Therefore, conclusion that the temperature in region 1 was at least 1070°F (577°C) (since the Al-Si melted), but probably closer to 1190°F (643°C) is consistent with the examination.

These results confirm the temperature distributions predicted by heat transfer calculations. The temperatures for regions 1 and 3 were 1191°F and 1214°F respectively for reactor operation at 50 MW-thermal.

3. RADIATION-INDUCED VOID FORMATION

Considerable effort has been expended recently to study irradiation induced void formation in metals and the resulting swelling because of its implications on the design of a Fast Breeder Reactor Core. The MT-3 capsule and its contents offered the opportunity to extend the knowledge of the swelling phenomenon into a range of materials composition and temperature and neutron fluence where little data currently exist. The Type-304 stainless steel strip used to support tensile specimens was ideally suited for preparation of specimens for transmission electron microscopy (TEM) examination. Pre-cut TEM disks of Incoloy-800, Hastelloy-X and Inconel-625 were examined for void occurrence and morphology.

3.1 IMMERSION DENSITY MEASUREMENT

The diametral profile of the MT-3 capsule tube indicated a diameter increase near the top of the EBR-II core. This was the first GE observation of clad swelling in a materials capsule tube. It had been assumed that capsules running at temperatures as low as $<720^{\circ}\text{F}$ (382°C), would not appreciably swell.

To determine if the observed diametral increase was actually the result of fluence-induced swelling, a limited number of immersion density measurements were taken. Samples one-half inch long of the 0.375 inch outside surface tube were taken in the region where the diameter was observed to increase.

The results are summarized in Table 3-1. In Figure 3-1 it can be seen that although the immersion density measurements do not exactly correspond with the diametral measurements, the same trend exists. The greatest swelling occurred about 12 inches above the bottom of the core while the maximum flux (Figure A-1) occurred at a height of 6 inches. The temperature increased with height as shown in Figure A-2. This implies that swelling is more sensitive to temperature than to fluence in the vicinity of these values of the variables.

From this observation it may be anticipated that reactor hardware will increase in volume even at temperatures only slightly above (20°F [11°C]) the EBR-II inlet temperature provided it receives sufficient exposure.

Immersion density measurements were also performed on some of the high-nickel alloy tensile specimens. The measurements are reliable, but their interpretation is somewhat uncertain because of the large temperature gradients found to exist along the two-inch specimen length. However, the values do show the relative dimensional stability of the three alloys as shown in Table 3-2. The density of the Hastelloy-X decreased approximately 2% in two cases and the density of Incoloy-800 decreased less than 0.5%. Inconel-625 densified as much as 0.5% during irradiation. The irradiation temperatures shown are the temperatures at the center of the gage lengths. Each specimen experienced higher temperatures than those shown in the table in the grip region due to the higher mass and the resulting higher gamma heating.

Table 3-1

VOLUME INCREASE OF THE MT-3 CAPSULE TUBE

Sample	IABC ^(a)	Temperature (°C)	Temperature (°F)	Fluence, 10 ²² E>0.1 Mev	% $\frac{\Delta\rho}{\rho}$	% $\frac{\Delta v}{v}$
MT-3B	6.25	375	707	5.41	-0.33	0.33
MT-3D	8.25	377	711	5.17	-0.90	0.91
MT-3F	11.75	381	718	4.07	-0.96	0.97
MT-3H	14.25	382	720	3.09	-0.61	0.61

(a) Inches Above Bottom of Core (EBR-II)

Table 3-2

IMMERSION DENSITY RESULTS FROM NICKEL ALLOY TENSILE SPECIMENS

Alloy	Fluence E>0.1 MeV-GE (x10 ²²)	Irradiation (a) Temperature (°C) (°F)		% $\frac{\Delta\rho}{\rho}$	% $\frac{\Delta V}{V}$
Incoloy-800	5.4	643	1190	-0.46	0.46
	5.4	643	1190	-0.14	0.14
	5.4	643	1190	-0.05	0.05
Hastelloy-X	4.5	603	1115	-0.13	0.13
	5.4	643	1190	-0.56	0.56
	5.4	643	1190	-1.8	1.8
Inconel-625	4.5	603	1115	-2.1	2.1
	5.4	643	1190	0.56	-0.56
	5.4	643	1190	0.23	-0.23
	4.5	603	1115	0.40	-0.40

(a) At center of gage length; temperature range over entire specimen was 1190 to 1280 for 1190°F specimens, and 1115 to 1225 for 1115°F specimens.

3.2 EXAMINATION BY TRANSMISSION ELECTRON MICROSCOPY

The Type 304 stainless steel strip had been included in the MT-3 capsule as a structural member to locate the nickel alloy tensile specimens. The strip offered a valuable opportunity to study void formation in Type-304 stainless steel at relatively high temperatures and neutron fluences in addition to the observations on the pre-cut high-nickel alloy TEM disks.

3.2.1 Experimental Procedure

Specimens for examination were chosen from various locations in the Type-304 strip to include as wide a range of temperature and fluence as possible.

Specimens were mechanically punched from the strip. Both 3 mm and 2.3 mm diameter punchings were made from the strip. The 2.3 mm specimens were sent to the General Electric Research and Development Center in Schenectady and the 3 mm specimens were retained for examination at the Vallecitos Nuclear Center. The locations of all specimens punched from the strip are listed in Table 3-3. Some difficulty was experienced in punching the 3 mm diameter specimens in that some deformation occurred making some of the earlier disks unusable. Additional disks

Figure 3-1. Comparison of Volume Increase of Capsule Tube As Measured by Density Decrease and Diameter Increase

were punched out with a new close-tolerance punch to fill in gaps in the temperature distribution. Figure 3-2 shows the location of all the specimens examined with respect to the temperature profile of the strip. The second group of specimens was rubbed on 600 grit abrasive paper to remove a thin oxide layer that was interfering with the thinning of the disks.^{4, 5}

In addition to the stainless steel specimens, the pre-cut high-nickel alloy disks were also included in the examination. Since the exact location of each disk within the container during irradiation was unknown, a temperature range is reported for these samples, 1155 to 1177°F (624 to 636°C). There is an additional uncertainty in temperature because of uncertainty in the heat transfer characteristics between the tensile specimens and the TEM disk container. This could make the temperatures as much as 30°F (17°C) below that of the adjacent tensile specimens. With this factor included, the temperature range for the high-nickel alloys excluding uncertainty factors for reactor power was 1124 to 1177°F (607 to 636°C) for 45 MW operation.

Disks were prepared by electrolytic thinning⁴ in a twin jet polisher⁶ using 10% perchloric acid in glacial acetic acid. A potential of 20V was used with a current density of 30 to 50 A/dm². All stainless steel disks were thinned successfully with the exception of those noted in Table 3-3.

Prepared specimens were viewed with 100 kilovolt electrons in a JEM 6A electron microscope. Photographs of voids were taken over as much of each foil as possible to give statistical weight to void counting and size measurements. Void counting and diameter measurement was performed on a Zeiss Particle Size Analyzer. This allowed a much larger number of voids to be counted than would have been possible by manual counting and sizing.

The foil thickness of each specimen was estimated in one of three ways:

- 1) By counting the voids intersected by one of the foil surfaces and using the equations of Sellers and Smith^{5, 7};
- 2) By determining the foil orientation from electron diffraction patterns and by stereographic projection for geometric analysis of slip traces;
- 3) By rough estimation of foil thickness from comparison with results from method (1) or (2).

Table 3-3

LOCATION AND DISPOSITION OF TRANSMISSION ELECTRON MICROSCOPY DISKS FROM MT-3

Sample	Location IABC ^(a)	Disposition	Sample	Location IABC ^(a)	Disposition
A1	1.77	E	M1	12.47	N-D
A2	1.88	E	M2	12.56	E
A3	1.55	N-UT	M3	12.66	E
B1	2.36	N-D	N1	13.44	E
B2	2.66	E	N2	13.75	E
B3	2.78	E	O1	15.52	N-UT
C1	3.38	N	O2	15.23	E
C2	3.64	E	O3	15.36	E
C3	3.16	N	O4	15.66	E
D1	4.47	N	P1	23.56	N-D
D2	4.31	E	P2	23.31	E
E1	5.20	E	Q1	24.00	N
E2	4.90	E	R1	24.70	N-D
F1	5.70	E	R2	24.47	E
F2	5.85	E	Incoloy-800	8.78-9.34	1E, 3N.
G1	6.94	N-D	Hastelloy-X	8.78-9.34	2E, 2N.
G2	6.72	E	Inconel-625	8.78-9.34	1E, 1N-UT, 2N
H1	7.80	E			
H2	7.63	E			
I1	8.69	E			
I2	8.47	E			
I3	8.92	N			
J1	9.66	E			
J2	9.55	E			
J3	9.22	N			
K1	10.81	E			
K2	10.94	E			
L1	11.63	N-D			
L2	11.41	E			

(a) - Inches Above Bottom of Core

D - Deformed

E - Examined

N - Not Examined

UT - Unsuccessful Thinning

A computer program was written which used the following input data for each micrograph: area, magnification, number of surface voids (or foil thickness), and number of voids in each class (size range) of the particle size analyzer. The computer output listed for each micrograph and for the whole specimen the void volume fraction, number of voids per cubic centimeter, average void diameter, foil thickness and area, foil volume, as well as the standard deviation of the void diameter measurements. In addition, a frequency distribution of void sizes was printed out in both tabular and histogram form.

3.2.2 Experimental Results

A total of thirty-three disks were examined. Twenty-nine of these were stainless steel specimens from the Type-304 strip irradiated in the temperature range of 766 to 1267°F (408 to 686°C). Fluence greater than 0.1 MeV

Figure 3-2. Location of Transmission Electron Microscopy Specimens with Respect to Temperature Profile

ranged from 0.7 to 5.41×10^{22} n/cm^2 $E > 0.1$ MeV-GE. Table 3-4 gives a detailed summary of the quantitative results of the examination. Four of the high-nickel alloy disks were also examined. The fluence ranged from 4.85 to 5.05×10^{22} n/cm^2 $E > 0.1$ MeV-GE. Only a qualitative examination was made of these specimens due to difficulty in interpreting results. It should be emphasized that in this type of examination the uncertainties are quite large, particularly for those MT-3 specimens in which the void sizes are comparable with or greater than the foil thickness. For the void volume fraction the uncertainty is estimated to be $\pm 100\%/50\%$. A detailed discussion of the uncertainty incurred in the analysis is given in Appendix B.

3.2.2.1 Type 304 Stainless Steel

In addition to the quantitative data, qualitative descriptions provide further details. Figures 3-3 to 3-9 show electron micrographs of the stainless steel specimens which are representative of the various microstructural details observed. Much of the dislocation structure observed may have been introduced after irradiation during specimen preparation. The figures are presented in order of increasing temperature.

Figure 3-3 shows sample O4 which was an upper blanket specimen irradiated at 786°F (419°C). This specimen contained a large number of small voids polyhedral in shape. In comparing Figure 3-3 to the other micrographs, note the much higher magnification of Figure 3-3. Figure 3-4 shows the Disk A1 [irradiation temperature of 864°F (462°C)] in which the largest volume fraction* of voids was found. Also of interest is the stratification of voids, apparently along some of the (111) crystallographic planes. Disk M3 [irradiation temperature of 1033°F (556°C)] in Figure 3-5 shows a considerable increase in void diameter and a corresponding decrease in number density. The average void size in M3 is ~ 1000 Å diameter. Again noting the variation in magnification, the void size has increased and the number density decreased going to Sample K1 [irradiation temperature of 1117°F (603°C)] Figure 3-6. In this specimen the voids appear quite rounded suggesting that a number of them had been etched through the foil. The average void size in Sample I1 [irradiation temperature of 1191°F (644°C)] Figure 3-7, is somewhat smaller than in K1 and the voids are

* See Appendix C for a discussion of the relation of void volume fraction to bulk volume increase.

Table 3-4

QUANTITATIVE RESULTS OF VOID MEASUREMENTS IN TYPE-304 STAINLESS STEEL

Void										
Disk	Temperature		Neutron Fluence 10 ²² n/cm ² (E > 0.1 MeV)	Volume Fraction (%)	Diameter (Å)		Size Range (Å)	Number Density (cm ⁻³)	Total Volume Examined (mm ³)	Total Voids Counted
	(°F)	(°C)			Mean	Mode				
F2	1278	692	5.42	0	NA	NA	NA	0	ND	0
F1	1267	686	5.41	0	NA	NA	NA	0	ND	0
H1	1260	682	5.26	0	NA	NA	NA	0	ND	0
D2	1252	678	5.28	0	NA	NA	NA	0	ND	0
H2	1247	675	5.29	0	NA	NA	NA	0	ND	0
C2	1243	673	5.16	0	NA	NA	NA	0	ND	0
I2	1240	671	5.13	0	NA	NA	NA	0	ND	0
G2	1209	654	5.39	0	NA	NA	NA	0	ND	0
J1	1213	656	4.81	0	110	S	80–160	<10 ¹⁰	ND	5
E1	1195	646	5.39	1.4	2080	1850	700–3800	2.2x10 ¹²	48x 10 ⁻⁹	106
I1	1191	644	4.08	0.1	1450	S	500–2350	6.5x 10 ¹¹	180x 10 ⁻⁹	117
B3	1171	633	4.94	1.5	1830	1700	600–3600	3.4x 10 ¹²	21x 10 ⁻⁹	70
K1	1117	603	4.42	2.0	1870	1450	400–3700	4.0x 10 ¹²	46x 10 ⁻⁹	183
M1	1078	581	3.79	1.4	1110	S	200–2600	1.3x 10 ¹³	16x 10 ⁻⁹	211
M3	1033	556	3.71	1.8	1000	S	200–2200	2.1x 10 ¹³	11x 10 ⁻⁹	222
N1	939	504	3.40	1.1	660	450	80–2000	2.9x 10 ¹³	16x 10 ⁻⁹	457
A1	864	462	4.62	2.4	460	415	115–1000	3.9x 10 ¹⁴	5x 10 ⁻⁹	1969
O4	786	419	2.58	0.1	120	S	20–310	1.2x 10 ¹⁵	0.9x 10 ⁻⁹	1058
R2	752	400	0.70	ND	ND	ND	ND	ND	ND	ND

NA = Not Applicable

ND = Not Determined

S = Same as the Mean

NEG No. 5381A

Figure 3-3. Specimen 04

Figure 3-4. Specimen A1

widely and non-uniformly spaced. This suggests that the temperature for this specimen has reached a point where either voids are unable to grow to very large sizes or they have started to anneal out during reactor operation at 50 MWt. Another 18°F (10°C) increase in temperature produced the structure in Figure 3-8, Disk J1, [irradiation temperature of 1213°F (656°C)] in which no voids were observed with the exception of one cluster of 5 ranging from 80 to 160 Å.

It has been suggested that such small isolated cavities produced in high temperature irradiation are helium bubbles rather than voids.⁸ In the specimens irradiated at temperatures >1211°F (655°C) no voids were observed in the stainless steel matrix. There were a few instances of what appeared to be voids in the grain boundary carbides in Sample H1. This is best illustrated in Figure 3-9 in which the voids range in size from 100 to 300 Å diameter. The precipitate containing them was confirmed to be $M_{23}C_6$ by selected area electron diffraction. In addition to the specimens noted above that contained no voids, a number of the foils examined at the GE Research and Development Center also were void free. Table 3-4 is a listing of all specimens which contained no voids.

In specimens where a large number of voids were counted (>1500), the distribution of sizes was approximately normal but the peak was skewed toward the lower sizes. The best example is Sample A1 in which ~2000 voids were measured. Their size distribution, taken from the computer output, is shown in Figure 3-10. Although the peak frequency of voids occurs at ~410 Å, there is a tail of large size voids which extends almost to 1000 Å. If this distribution is representative of all specimens, then for the high temperature specimens in which very large voids were observed, there is a strong probability that some voids had diameters which were substantially greater than the foil thickness. These voids would then be enlarged by electroetching so that quantitative values would become strongly biased toward large sizes. Considerable effort was made at the GE Research and Development Center in Schenectady to find some distinguishing feature of voids etched through the foil. It was concluded that there was no consistent visual difference between a large void near the thin edge of the foil which was etched through and a similar size void in a thicker area.

NEG No. 5351A

Figure 3-5. Specimen M3

NEG No. 5278

Figure 3-6. Specimen K1

NEG No. 5175

Figure 3-7. Specimen 11

↑
0.1 μ
↓

NEG No. 5230

Figure 3-8. Specimen J1

0.1 μ

NEG No. 5148

Figure 3-9. Specimen H1 - Voids in $M_{23}C_6$ Surrounded by Void-Free Matrix

Figure 3-10. Frequency Distribution of Void Sizes - Specimen A1

The set of foils examined at the GE Research and Development Center showed no features significantly different from the specimens examined at VNC. At any given temperature the foils were comparable. In addition to the core samples, two specimens were examined which were approximately 10 and 11 inches above the top of the EBR-II core. From comparison it was noted that there was a marked absence of interstitial loops in the specimens taken from the core region of the reactor, presumably because of the high operating temperature (460 to 690°C). Some loops were present in Disk 04 [786°F (419°C)] and many were present in Disk R2 irradiated at 752°F (400°C) as can be seen in Figure 3-11. Disk R2, located 11 inches above the top of the EBR-II core, had been irradiated to a neutron fluence of 0.7×10^{22} n/cm², $E > 0.1$ MeV. The small polyhedral voids (≈ 30 to 200 Å diameter) in Disk R2 were not measured quantitatively. However, despite the lower neutron flux and spectrum, the void size and number density of R2 appear similar to those of other specimens from the EBR-II core irradiated at a similar temperature and neutron fluence.⁹

3.2.2.2 High-Nickel Alloys

The Incoloy-800 disk contained voids with an average diameter of about 850 Å and a number density of less than 10^{13} per cm³. Figure 3-12 shows a typical area. Some very small pores were detected, especially at twin boundaries. These pores are probably helium bubbles. The microstructure is generally clean, apart from a few carbide particles ($M_{23}C_6$) of similar size to the voids which frequently have voids associated with them. The general microstructure is very similar to solution annealed Type-304 stainless steel irradiated at the same temperature, i.e., a normal austenitic structure containing little precipitation.

In contrast the Hastelloy-X disks contain very large voids and a considerable quantity of large rod-like precipitates as seen in Figure 3-13. The voids are so large (2500 to 10,000 Å diameter on the micrographs) that it is obvious that most of them have been enlarged during thinning. However, examination of very thick areas of the foil which were otherwise opaque showed clearly defined voids (with diameters up to 3000 Å) without extinction contours. Also, the void number density is very small (approximately 5×10^{12} voids/cm³) and an average void size of 2000 to 3000 Å is required to achieve the volumetric swellings of up to 2.1% measured by liquid immersion in the tensile specimens. Thus

0.1 μ

NEG No. 6695

Figure 3-11. Voids and Interstitial Loops in Type-304 Stainless Steel Irradiated at 400°C

NEG No. 5436

Figure 3-12. Incoloy-800

NEG No. 5450A

Figure 3-13. Hastelloy-X

→ | ← 0.1 μ

NEG No. 6564A

→ | ← 0.1 μ

NEG No. 6563A

Figure 3-14. Inconel-625

it is evident that the swelling measured in the Hastelloy-X is due to a small number of very large voids. Comparison with unirradiated Hastelloy-X samples shows that the rod-shaped precipitates seen in Figure 3-13 have appeared during irradiation. From the work of Comprelli and Lewis¹⁰, they are most probably M_6C .

Micrographs of the Inconel-625 are shown in Figure 3-14; it may be seen that no voids have been generated. Instead the microstructure shows a very high density of finely divided precipitate which is Ni_3Nb , a gamma-prime precipitate. The Ni_3Nb has obviously appeared during in-reactor aging and has inhibited void formation as well as causing densification of the alloy. Ni_3Nb has a measured density of 8.8 g/cm^3 (8.99 g/cm^3 theoretical) compared to the measured unirradiated value of 8.452 g/cm^3 for Inconel-625. The precipitate is in the form of semi-coherent disks 1000 to 2000 Å in diameter and 100 to 200 Å thick. The habit planes of the disks are (100) matrix planes, and the interlamellar spacing is $\sim 500 \text{ Å}$ as shown in Figure 3-14. The matrix precipitate is not the stable orthorhombic Ni_3Nb which had been identified in an earlier study¹⁰ of unirradiated Inconel-625, heat treated for ~ 1000 hours at 1200°F . Electron diffraction patterns obtained from the irradiated Inconel-625 were indicative of the metastable tetragonal Ni_3Nb phase described by Royer and Gantois¹¹ with unit cell dimensions of $a = 3.57$, $c = 7.39$. With this unit cell, there is nearly perfect coherency in the 100 and 010 directions of the precipitate phase but a 3% deviation in the 001 direction. These facts account for the platelet shape and for the coherency strains observed. Coherency strains are invisible when the platelets are seen edge-on.

The structure of the unirradiated solution-annealed material contained no Ni_3Nb but only larger equiaxed carbide precipitates, which are also present in the post-irradiation structure (Figure 3-14).

4. TENSILE TESTING

4.1 SPECIMENS AND PROCEDURE

The high-nickel alloy tensile coupons which had been irradiated to the highest fluence were tested in air at temperatures of 900, 1100, and 1300°F (482 , 594 and 704°C), using an Instron testing machine. Details of the shape and size of the specimens are shown in Figure 4-1. The effect of strain rate on tensile properties was investigated by performing the 1100 and 1300°F (594 and 704°C) tests at three strain rates: 2 min^{-1} , $2 \times 10^{-2} \text{ min}^{-1}$ and $2 \times 10^{-4} \text{ min}^{-1}$. A total of thirty-six tests were performed on twelve specimens each of Incoloy-800, Inconel-625 and Hastelloy X. None of the coupons from the reactor blanket region were tested. Some difficulty with grip slippage was experienced during testing; those tests are not reported. All stress-strain values were obtained from the load-deflection measurements. Tests on unirradiated controls were carried out on samples from the same material heats.

4.2 TENSILE TEST RESULTS*

4.2.1 General Description

Several of the specimens, both irradiated and unirradiated, exhibited load-deflection curves typical of the discontinuous yielding attributed to precipitate or solute locking of dislocations (Portevin-le Chatelier effect).

This effect was observed in tests on unirradiated Incoloy-800, Hastelloy-X and Inconel-625 at temperatures of 900°F (482°C) and 1100°F (594°C) at the $2 \times 10^{-2} \text{ min}^{-1}$ strain rate and also in tests at the $2 \times 10^{-4} \text{ min}^{-1}$ strain rate at both 1100°F and 1300°F (594 and 704°C). Only Inconel-625 displayed the effect at the faster $2 \times 10^{-2} \text{ min}^{-1}$ strain rate at 1300°F (704°C). Test results on the irradiated alloys, with the exception of the 900°F (482°C) tests, indicated that the effect had apparently been suppressed at the $2 \times 10^{-2} \text{ min}^{-1}$ strain rate but was still present at the slowest strain rate $2 \times 10^{-4} \text{ min}^{-1}$ at both 1100°F and 1300°F (594 and 704°C).

4.2.2 Effect of Irradiation on Tensile Properties

The temperature during irradiation for all these alloys was well above 1100°F (594°C) and structural hardening by vacancy clustering and dislocation loop interaction would be expected to be minimal because thermal annealing was concurrent with defect generation. The similarity of yield stress values for irradiated and unirradiated Incoloy-800 substantiates this hypothesis. Both the Hastelloy-X and Inconel-625, however, showed hardening manifested by

* Results for individual irradiated and unirradiated specimens are given in Appendix D.

Figure 4-1. Tensile Coupon Used in Fast Neutron Damage Experiments

increased ultimate tensile strength and 0.2% yield stress values. Total and uniform elongation values for all the alloys were substantially reduced as seen in Figures 4-2, 4-3, and 4-4.

4.2.3 Effect of Test Temperature Increase

All mechanical property values resulting from the irradiated alloy tensile tests decreased with increasing test temperature (Figures 4-2, 4-3, and 4-4).

For the unirradiated material, all alloys showed a decreasing ultimate tensile strength and uniform elongation with increasing test temperature. The response of total elongation and 0.2% yield stress values to a test temperature increase, however, varied with each alloy. Incoloy-800 displayed a reduction in the 0.2% yield stress with temperature increase and an increase in total elongation. For Hastelloy-X, neither 0.2% yield stress nor total elongation values changed substantially with test temperature increase. Inconel-625 showed reducing total elongation values and an unchanged 0.2% yield stress (Figures 4-2, 4-3, and 4-4).

4.2.4 Effect of a Strain Rate Increase

At a test temperature of 1300°F (704°C), with the exception of the Incoloy-800 total elongation values, all mechanical property values for the irradiated alloys increased with increasing strain rate. Incoloy-800 showed a ductility minimum at the $2 \times 10^{-2} \text{ min}^{-1}$ strain rate. This is shown in Figures 4-5(b), 4-6(b) and 4-7(b).

At the 1100°F (594°C) test temperature, all mechanical property values of the irradiated material, with the exception of the 0.2% yield stress, increased with increasing strain rate. The yield stress values tended to remain comparatively unaffected although Hastelloy-X displayed a considerable increase at the fastest (2 min^{-1}) strain rate (Figures 4-8(b), 4-9(b) and 4-10(b)).

Figure 4-2. The Effect of Test Temperature on the Mechanical Properties of Incoloy-800
 Strain Rate 0.02 min^{-1}

Figure 4-3. The Effect of Test Temperature on the Mechanical Properties of Inconel-625
 Strain Rate 0.02 min^{-1}

Figure 4-4. The Effect of Test Temperature on the Mechanical Properties of Hastelloy-X
 Strain Rate 0.02 min^{-1}

Figure 4-5. The Effect of Strain Rate on the Mechanical Properties of Incoloy-800 Tested at 1300°F (704.4°C)

Figure 4-6. The Effect of Strain Rate on the Mechanical Properties of Inconel-625 Tested at 1300°F (704.4°C)

(a) UNIRRADIATED

(b) IRRADIATED

Figure 4-7. The Effect of Strain Rate on the Mechanical Properties of Hastelloy-X Tested at 1300°F (704.4°C)

(a) UNIRRADIATED

(b) IRRADIATED

Figure 4-8. The Effect of Strain Rate on the Mechanical Properties of Incoloy-800 Tested at 1100°F (593.3°C)

(a) UNIRRADIATED

(b) IRRADIATED

Figure 4-9. The Effect of Strain Rate on the Mechanical Properties of Inconel-625 Tested at 1100°F (593.3°C).

(a) UNIRRADIATED

(b) IRRADIATED

Figure 4-10. The Effect of Strain Rate on the Mechanical Properties of Hastelloy-X Tested at 1100°F (593.3°C)

All unirradiated alloys tested at 1300°F (704°C) showed increased ultimate tensile strength and uniform elongation values. The response of other properties varied considerably; however, Inconel-625 and Hastelloy-X displayed the same lack of yield strength sensitivity to strain rate they had previously shown to test temperature. Incoloy-800 showed an increase in yield strength. Total elongation values decreased with increasing strain rate for Incoloy-800 and Hastelloy-X but increased for Inconel-625 (Figures 4-5(a), and 4-7(a).

Little positive similarity in behavior was observed for the unirradiated alloys tested at 1100°F (593°C). With respect to general trends, Incoloy-800 and Hastelloy-X showed increasing values for ultimate tensile strength and uniform elongation, decreasing values for total elongation and comparative insensitivity of yield strength values to strain rate (Figures 4-8(a) and 4-10(a).

Inconel-625 showed effectively no change in ultimate tensile strength (although a slight increase was observed at the $2 \times 10^{-2} \text{ min}^{-1}$ strain rate). The 0.2% yield stress showed a pronounced dip at the intermediate strain rate, $2 \times 10^{-2} \text{ min}^{-1}$, and maximum elongation values, total and uniform, at the intermediate $2 \times 10^{-2} \text{ min}^{-1}$ strain rate, Figure 4-9(a).

5. DISCUSSION

5.1 TEMPERATURE AND FLUENCE DEPENDENCE OF VOID FORMATION

An unexpected result of the examination of the Type-304 stainless steel strip was the large void size observed in many of the foils. The only previous observation of material containing many voids greater than 1000 Å was reported by Cawthorne and Fulton¹² in their initial work on highly irradiated fuel cladding. It had been assumed these large voids were the result of stress in the cladding.

The maximum calculated stress in the Type-304 strip in the MT-3 capsule at the beginning of life was 2500 psi. The post-irradiation examination results lead to the conclusion that the strip broke early in life. Therefore, for most of the irradiation time the stress in the strip was essentially zero. It would appear then that the only prerequisite for large diameter voids in Type-304 stainless steel is high temperature 932 to 1202°F (500 to 650°C) and long exposure times (i.e., high neutron fluence, $\sim 5 \times 10^{22} \text{ n/cm}^2$, $E > 0.1 \text{ MeV}$).

It became apparent during this examination that void diameters can be measured meaningfully by thin foil transmission electron microscopy only when they are smaller than approximately 2000 Å. If a normal distribution of sizes is assumed, it is evident that many of the voids will have diameters larger than the foil thickness. Such large voids inevitably intersect the surface of the foil. Smaller voids also occasionally intersect the surface since the nucleation points are random. In preparing the specimen, all these voids which intersect the surface will be enlarged by the electrolyte. Consequently the large voids are likely to form holes completely through the foil. When this occurs, the original size of the voids cannot be determined. If the hole sizes are taken as the original void sizes, an erroneous average diameter results which in turn affects the calculated volume fraction.

It is reasonable to assume that the situation described above occurred in samples in which the average void diameter approached the foil thickness (~ 2000 to 3000 Å), i.e., Samples E1, B3 and K1. For these samples the mode (i.e., the peak in the frequency distribution of void size) was considered to be more closely related to the actual mean size before thinning than the average measured diameter. However, it must be emphasized that electropolishing through the foil cannot begin to explain the range of void sizes observed. To help resolve the question of actual void size, a plane section was cut through a specimen with large voids. The surface was polished by ion sputtering.¹³ Electron micrographs of two-stage chromium-shadowed carbon replicas of this surface were compared with thin foil transmission micrographs of a specimen irradiated at a similar temperature and neutron fluence (see Figure 5-1). From stereo pictures and from the shape of the shadows, it was ascertained that the intersections of the surface with the voids were undistorted. By this technique, many voids were observed to be greater than 2000 Å in diameter.

IRRADIATION TEMP 630°C
 FLUENCE 5×10^{22} n/cm²
 E > 0.1 MeV

SPECIMEN C3 REPLICATION

NEG No. 5779

Figure 5-1. Comparison of Voids Observed by Thin Foil Transmission and Replication Electron Microscopy

The relation of average void diameter to temperature during irradiation found in MT-3 is shown in Figure 5-2 along with data obtained by others.^{14,15,18} As can be seen, the data generated from MT-3 are self-consistent, but are considerably higher than data from other laboratories. The main difference between these results and the other sets of specimens reported was significantly longer exposure time and higher fluence for MT-3. Most of the data from other experimenters had neutron fluences of about 3×10^{22} neutrons/cm² ($E > 0.1$ MeV) or less. The difference observed between void diameters from the MT-3 experiment and other data could be the result of minor variations in chemistry or microstructure. The chemical analysis of the strip showed it to conform to an ASTM Type-304 stainless steel and the microstructure was typical of an annealed austenitic. All the other data in Figure 5-2 were also obtained from annealed Type 304.

Another interesting feature of the void size measurements was the increase in minimum size with increasing temperature. The largest minimum diameter observed was 700 Å in Disk E1. For comparison, the minimum size observed in Disk 04 was 20 Å at the limit of resolution of the electron microscope. Figure 5-3 shows that the minimum observed void diameter is directly related to temperature.

The void number density also exhibited a definite relationship with temperature during irradiation. A plot of the number density versus temperature during irradiation (Table 3-4) is shown in Figure 5-4. The data show no apparent trend with respect to neutron fluence over the limited range of 2.6 to 5.4×10^{22} n/cm², $E > 0.1$ MeV.

Disk I1 deviated from the trend established by the other data points in both average diameter and void number density. Although the calculated temperature for I1 was 4°F (2°C) below that of E1, it is possible the temperature was actually a few degrees higher. When the reactor power was raised to 50 MW, voids already formed at lower power (temperature) may have started to anneal. Considered that no voids were found in specimens irradiated at an 18°F (10°C) higher temperature, it would appear that the temperature dependence of void size and number has a discontinuity at about 1202°F (650°C). This is clearly demonstrated by a plot of void volume fraction versus irradiation temperature* (Figure 5-5). Above 1202°F (650°C) no voids occur in Type-304 stainless steel irradiated to a neutron fluence of 5.0×10^{22} n/cm², $E > 0.1$ MeV. As that temperature is approached, the void volume fraction decreases by more than 1 order of magnitude within a few degrees (compare Disks E1 and I1 in Table 3-4). All specimens were cut from the same piece of material, hence no variations in microstructure and chemical composition are to be expected.

One possible explanation for the decrease in void production with increasing irradiation temperature may be surmised from a relation of vacancy concentration and temperature, illustrated by Harkness and Li.¹⁶ The concentration of vacancies produced by irradiation decreases with increasing temperature because of increased number of recombinations of vacancies and interstitials, while the concentration of vacancies produced by thermal excitation increases. The difference between the number of vacancies produced by radiation and the number produced by thermal excitation at a given temperature is a measure of the supersaturation of vacancies produced by irradiation. At some temperature the two curves intersect such that the contribution of radiation to the total vacancy population above the temperature of intersection is negligible. Therefore, the vacancy supersaturation is essentially zero for all temperatures above the intersection. If, at the point of intersection, (apparently about 650°C) both curves have large slopes (one positive and one negative) then an appreciable vacancy supersaturation will extend to within a few degrees of the intersection; and above the temperature of intersection void formation will abruptly cease rather than taper off gradually with increasing temperature.

There has been some disagreement between experimental observations of voids increasing in number density but not in size with continued irradiation,^{18,19} and the quantitative theoretical approach of Harkness and Li,¹⁶ which predicts increases in void diameters with continued irradiation.

Because the material in the experiment reported here covered nearly the whole temperature range in which void formation occurs, some insight into this problem may be gained. After nucleation, each void acts as a vacancy sink. Vacancies in close proximity to a void have a high probability of colliding with the void and being absorbed by it. Thus

* Volume fraction has been normalized to a neutron fluence of 5×10^{22} neutrons/cm², $E > 0.1$ MeV, by multiplying each value by a factor of $(\frac{5.0}{F})^{1.81}$ where F is fluence $\times 10^{-22}$ neutrons/cm², $E > 0.1$ MeV. The fluence exponent 1.81 is the result of empirically fitting swelling data to an equation model relating swelling to neutron fluence and irradiation temperature.¹⁷

Figure 5-2. Effect of Irradiation Temperature on Void Diameter In Type-304 Stainless Steel

Figure 5-3. Effect of Irradiation Temperature on Minimum Observed Void Diameter

Figure 5-4. Effect of Irradiation Temperature on Void Number Density

Figure 5-5. Volume Increase Related to Irradiation Temperature As Determined by TEM in MT-3

a vacancy gradient is produced and results in a net flow of vacancies into the void. The volume containing the vacancy gradient may be thought of as the "sphere of influence" of the void.¹ Since the size of the sphere of influence is related to vacancy mobility, it will increase with increasing irradiation temperature. As irradiation continues, more and more voids are nucleated until their spheres of influence impinge upon each other. At that point, void nucleation will in effect stop since vacancies created thereafter will have a much higher probability of contributing to growth of existing voids. If this analysis of the sequence of events is correct, the conflicting data can be explained: Early during irradiation, i.e., at low neutron fluence, nucleation will be the dominant mechanism of swelling. After the impingement of the spheres of influence, void growth only will take place. Since the size of the spheres of influence increases with temperature, it follows that at higher irradiation temperature, void nucleation will stop sooner (i.e., at lower neutron fluence) than at lower temperature. Therefore, an observation of continuing nucleation with increasing neutron fluence need not be inconsistent with an observation of continuing growth at a higher irradiation temperature. Similar conclusions may also be derived from the quantitative treatment by Harkness and Li.

This argument can be applied qualitatively to the data from this experiment. The strongest supporting evidence is the consistent increase in minimum observed void diameter with increasing irradiation temperature. The only disks which had voids as small as the resolution limit of the microscope ($\sim 20 \text{ \AA}$) were 04 and R2; both had been irradiated at relatively low temperatures ($< 410^\circ \text{C}$) for which the spheres of influence around the voids would be small. Therefore, nucleation was still occurring when irradiation was terminated. In all other disks, some minimum void size substantially greater than $20 \text{ \AA}$ was observed. In these disks, the spheres of influence around existing voids had overlapped and caused nucleation to cease. The higher the irradiation temperature, the earlier in life nucleation apparently had stopped as evidenced by the ever-increasing minimum void diameters (Figure 5-3). There have been other recent observations of swelling from which the experimenters have drawn the conclusion that void nucleation ceases relatively early during irradiation while void growth continues. These observations include Type-304 stainless steel irradiated to fluences greater than $1 \times 10^{23} \text{ n/cm}^2$,²⁰ Types-316 and-347 irradiated at constant temperature to different fluences²¹ and-Type M316 irradiated in Dounreay Fast Reactor.²²

Another explanation for the apparent absence of small voids is connected with a growth rate phenomenon. In the initial stages of growth, the increase in diameter will be rapid. However, the relative increase in diameter will drop off as growth continues since the diameter increases as the cube root of the number of vacancies added. Thus a large number of vacancies will have to be added to the void to make a visual increase in the diameter of large voids. In this case the chronological age of two voids may be very different even though under a microscope they appear to be quite similar in size. However, even if it was assumed that nucleation was a relatively rare event, it seems likely that out of all the foils examined there should have been some small voids found if nucleation was still occurring.

For long-term, high temperature irradiations, these observations may be significant because growth of voids nucleated early in life rather than nucleation of new voids will make the major contribution to volume increase. This consideration is particularly important when designing alloys for nuclear applications. An alloy designed to reduce the number of nucleation sites may initially swell less. However, reducing nucleation by this method will result in high vacancy supersaturation since there will be fewer voids to act as vacancy sinks. The subsequent growth rate of the existing voids may be enhanced. Even relatively low void number densities, such as those observed in Disks E1, B3, and K, (10^{12} to 10^{13} voids/cm³) will eventually cause appreciable swelling after long-term exposure.

5.2 EFFECT OF MICROSTRUCTURE ON VOID FORMATION

It is apparent from examination of the electron micrographs and from the immersion density measurements that the MT-3 materials irradiated and examined did not experience similar amounts of swelling when exposed to the same neutron fluence at the same temperature. Hastelloy-X increased in volume at least* 2% and Type-304 stainless steel 1.5% while Incoloy-800 swelled at least* 0.5% and Inconel-625 densified about 0.5%.

* It must be remembered that the density measurements of these alloys were performed on tensile specimens which experienced severe temperature gradients (see Figure A-4). If the temperature dependence of void formation in Incoloy-800 and Hastelloy-X is similar to that found in Type-304 stainless steel, then most of the observed swelling occurred in the low temperature part of the specimens. Therefore, the swelling in the localized areas which relate to the electron micrographs may be considerably higher than the average value indicated by density measurements.

The vast differences in swelling behavior of these alloys is attributed to their different aging characteristics. Incoloy-800 with Ti content below 0.4 wt% is a fully austenitic solid solution alloy with no purposeful additions for second-phase strengthening and thus behaves similarly to the Type-304 stainless steel. The Hastelloy-X formed a relatively coarse rod-shaped precipitate 7000 to 10,000 Å in length by about 1000 Å thick. The rods were incoherent and spaced far enough apart for dislocations to move between them.

In contrast the Ni_3Nb precipitate in the Inconel-625 is on a much finer scale. A detailed examination of the micrographs also showed no visible dislocations. It is possible that the density and spacing of these precipitates is such that the random motion of interstitial atoms is effectively blocked. Interstitials are more mobile than vacancies and thus have a higher probability of encountering a sink. If, however, in its random migration an interstitial atom encounters the strain field of a semi-coherent precipitate, it will be attracted and occupy the interface which represents a lower energy state than an interstitial site in the metal matrix. The interstitial then is trapped on the precipitate surface until the random migration of a vacancy brings it within range of the interstitial for annihilation. With a high density of fine precipitates, this trapping mechanism does several things. It keeps large numbers of interstitials from reaching sinks thereby leaving them available for recombination with vacancies. This maintains both a balance in the two species and depresses the concentration of both species. At the irradiation temperature experienced by the specimens reported herein, the interstitial trapping apparently acted to limit the formation of line dislocations. Since dislocation motion is a strong interstitial sink, the absence of dislocations further reduces the possibility of the occurrence of an imbalance in the concentration of vacancies and interstitials. With no appreciable excess of vacancies with respect to interstitials there will be little tendency to form stable voids.

The preceding arguments apply to a finely divided precipitate such as found in the irradiated Inconel-625. With respect to the Hastelloy-X, the large precipitates were too widely spaced to trap a sufficient number of interstitial atoms. Line dislocations formed (which swept out more interstitials) and left an excess of vacancies. The energy required to nucleate voids will be lower if one side of the nucleus is on a free surface. The large precipitates provide free surface area and thereby enhance void nucleation in a system with excess vacancies.

Swelling resistance in Nimonic PE-16²³ has been ascribed to the presence of coherent gamma-prime precipitate $\text{Ni}_3(\text{Al}, \text{Ti})$. The coherence of inter-particle spacing was considered essential to the limitation of swelling. In the case of the Inconel-625, precipitate coherency was not completely maintained. From the present information it can not be determined if coherency is definitely a prerequisite to inhibit void formation. A companion capsule (MT-4) is continuing irradiation to high neutron fluence. It may then be possible to determine if the precipitates maintain at least partial coherency and continue to inhibit swelling.

5.3 EFFECT OF IRRADIATION AND MICROSTRUCTURE ON MECHANICAL PROPERTIES

Despite the high irradiation temperature $\sim 1150^\circ\text{F}$ ($\sim 620^\circ\text{C}$), the increased ultimate tensile strength and 0.2% yield stress values of Inconel-625 and Hastelloy-X after irradiation would at first be attributed to irradiation hardening defined as the interaction between dislocations and irradiation-produced obstacles. There is evidence to suggest²⁴ that impediments to screw dislocation movement are prime factors in determining the yield stress. Such impediments would necessarily require large strain fields such as those generated by vacancy clusters or dumbbell shaped interstitials (two atoms sharing one lattice site). However, this type of hardening should be minimal at these irradiation temperatures. It has been shown²⁵ that at high irradiation temperatures the mechanism of hardening is by voids according to the Coulomb expression:

$$\Delta\sigma = 2Gb/L$$

where

- G = shear modulus
- b = Burgers Vector
- L = average spacing along a dislocation = $(Nd)^{-1/2}$
- N = cavity density
- d = cavity diameter

However, in the transmission electron microscopy examination (See Section 3), voids were not detected in Inconel-625 (a volume decrease was observed) and in the case of the Hastelloy-X, the voids were few and large (mean void diameter 2000 Å). Furthermore, Incoloy-800 irradiated at the same temperature showed a decrease in strength. Comprelli²⁶ has reported strength increases from 200 to 250% in tests at 1100 and 1200°F (594 and 704°C) on unirradiated Inconel-625 after soaking for 5000 hours at 1200°F (649°C). The 122% and 78% yield strength increases noted in the present series of tests are well within these limits. Similarly, exposure of Hastelloy-X for 1000 hours at 1100°F (594°C) almost doubled the 0.2% yield stress (39 ksi → 70 ksi) tested at 1100°F (594°C) which is very comparable to the 30 ksi to 55 ksi increase noted in the present studies.

However, Comprelli showed a minimum total elongation of 10 to 15% for Inconel-625 and 20% for Hastelloy-X compared to the 2.5% and 7% elongation observed in the irradiated material. This decreased ductility is taken to indicate that some helium embrittlement has occurred. The formation of helium bubbles at the grain boundaries and the corresponding reduction in cohesive strength and enhanced propagation of microcracks would not be expected to diminish with increasing irradiation temperature.

From a consideration of the above data and as a result of the TEM studies, the hardening of the Inconel-625 and the Hastelloy-X has been attributed to thermally induced precipitation: Ni_3Nb probably associated with TiN and NbC in the case of Inconel-625, and M_6C in the case of Hastelloy-X. The relative insensitivity of the unirradiated Hastelloy-X (0.2% yield stress) to increasing strain rate compared to the irradiated alloy would apparently be due to the more stable nature of the solid solution hardened unirradiated matrix.

The most significant features of these data are: (1) the similarity of response of all the irradiated alloys to a strain rate increase or a temperature decrease (all property values increased) compared to the unirradiated specimens whose mechanical property values show widely varying responses to changes in strain rate or test temperature; (2) the close relationship in the effect of a temperature decrease or a strain rate increase (all property values increase) for irradiated alloys over the temperature and strain rate range examined. Zener, Holloman, Sherby and Dorn²⁷ have shown that the ultimate tensile strength or creep rupture strength can be related to the test temperature and strain rate by a single expression

$$\sigma = f [\dot{\epsilon} e^{Q/RT}]$$

Q = activation energy for self diffusion.

Sumerling and Rhodes²⁸ attempted to carry this work one step further and relate uniform elongation to a Z parameter where

$$Z = \log_{10} [\dot{\epsilon} \cdot e^{Q/RT}]$$

Thus temperature and strain rate could be combined as one parameter. This analysis was to be applied to both irradiated and unirradiated material. It is generally accepted that for many unirradiated materials as temperature increases or strain rate decreases, i.e., for a low Z value, nonuniform elongation increases and uniform elongation decreases. The interrelationship between temperature and strain rate is evident. In a similar manner, the mode of deformation and failure can be related to the Z parameter with transgranular failure at high Z values and intergranular failure at low Z values. As has been stated earlier, the loss of ductility of irradiated material is largely associated with loss of grain boundary strength which is most important at high temperatures and low strain rates. The major reduction in ductility of irradiated material results from a reduction in this necking or nonuniform component.

In considering the present results on high-nickel alloys, and observing the uniformity of response of all irradiated alloys to both strain rate and temperature increases, it is very tempting to use a Z parameter. However, certain anomalies in the results, e.g., the ductility minimum for Incoloy-800 at the $2 \times 10^{-2} \text{ min}^{-1}$ strain rate, also observed by Comprelli,²⁹ and the increased ductility³⁰ noticed in irradiated PE-16 at low strain rates 10^{-7} sec^{-1} , combined with the wide variations in response of the current unirradiated material indicate that more experimental results are required covering a wider range of temperature and strain rate, before the parametric relationship is definitely established.

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APPENDIX A

A-1 MATERIALS INVENTORY AND EXPOSURE CONDITIONS FOR MT-3 CAPSULE MATERIALS

Various samples from MT-3 and the capsule tube itself were used to study the effects of fast neutron exposure on mechanical properties and void formation. A complete inventory of materials in the capsule is given in Table A-1. Table A-2 gives the location of individual tensile specimens in the capsule. The specimens are grouped according to their axial location in the capsule. Each group of 6 specimens at one axial location constitutes a region with Region 1 being the one nearest the bottom of the capsule. Regions 1 through 5 were in the core, while Region 6 overlapped the core and upper blanket. Regions 7 and 8 were located entirely in the upper blanket region of the EBR-II reactor.

A-2 FLUENCE

The neutron fluence experienced by MT-3 was determined by converting the number of megawatt days the capsule was in reactor to flux and then fluence. The calculated fluence at the reactor center was corrected for the radial location of the capsule (outside edge of Row 4). The total fluence and the fluence at energies >0.1 MeV and >1 MeV were determined for any axial location in the capsule using normalized profiles determined by dosimetry results. Table A-3 gives the operation history of MT-3. From this the fluence profiles shown in Figure A-1 were determined.

A-3 TEMPERATURE

The MT-3 capsule consisted of (from bottom to top) a bottom end plug, six sets of tensile specimens, a solid positioner, two additional sets of tensile specimens, and an upper end cap. A thin stainless steel strip (0.010 x 0.200 inch) ran the entire length of the assembly, and the tensile specimens were wired to the strip with a narrow argon gas gap separating the control assembly from the stainless steel capsule tube.

The capsule tube temperature was calculated to be only 27°F (15°C) above the core inlet temperature. The profile of this is shown in Figure A-2.

Temperature profiles of the capsule inserts were calculated using a computer code which employs a two-dimensional nodal model. The gamma heating rate axial profile (at 50 MW) for the EBR-II, Row 4 location, was taken from the *Guide for Irradiation Experiments in EBR-II*.^{A-1} In all regions, the capsule tubing adjacent to the sodium coolant was assumed to be at 700°F which is the core inlet temperature. The maximum coolant temperature rise across the core was estimated to be 20 to 30°F and was considered negligible. Both conduction and radiation occur across the gas gap between the central assembly and the capsule tube. For the radiative transfer, all surfaces were assumed to have an emissivity of 0.5, and an effective emissivity at each axial plane was calculated from the standard formula,

$$\epsilon_{\text{eff}} = \frac{1}{\frac{1}{\epsilon_1} + \frac{A_1}{A_o} \left(\frac{1}{\epsilon_o} - 1 \right)}$$

The resulting temperature distributions are noted in Figure A-3 and Table A-4. The corresponding maximum temperatures of the tensile specimens are 1278°F and 1324°F (692°C and 718°C).

A-4 AXIAL TEMPERATURE DISTRIBUTION ALONG TENSILE SPECIMEN

Each tensile specimen has the shape of a capital I, with wide sections (0.200 inch) at each end and a narrow section (0.100 inch) in the middle as shown in Figure 4-1. The 10-mil thick support strip is uniformly 0.200 inch wide and, therefore, extends as a fin on both sides of the central section of the specimen. The resulting difference in the surface area-to-volume ratio between the ends and the center produces a significant thermal gradient along each specimen. For example, at 50 MWt the maximum temperature difference along a specimen is 110°F (61°C).

The axial gradient along each tensile specimen occurred as a result of the small cross-sectional areas. The small cross-sectional areas for axial heat transfer along the specimen caused almost all (> 90%) of the heat flow to be radial. Because of the connector strip, the surface area for radial heat loss along the specimen is essentially constant while a

Table A-1

MATERIALS IN MT-3 CAPSULE

1. Capsule Tube: Type-304 stainless steel, 36-15/16 inches long x 0.375 inch diameter x 0.020 inch wall.
2. Stainless steel strip (Type-304) 24-5/8 inches long x 0.200 x 0.010 inch.
3. Temperature Sentinels:
 - No. 1 Al; Al-Si
 - No. 2 Al; Al-Si
 - No. 3 Al-Cu; Ag-Sb
 - No. 4 Al-Cu; Ag-Sb
4. Tensile Specimens: 0.030 inch thick x 0.200 inch wide x 2 inches long.
 - 36 from core region
 - 12 from blanket region
5. Electron microscopy disks
 - 4 Inconel-625
 - 4 Incoloy-800
 - 4 Hastelloy-X

Table A-2

POSITION OF TENSILE SPECIMENS IN MT-3

Region ^(a)	1	2	3	4	5	6	7	8
	X1-36	X1-34	X1-40	X1-4	X1-37	X1-70	X1-44	X1-32
	L1-12	L1-16	L1-55	L1-67	L1-5	L1-45	L1-19	L1-24
	V1-50	V1-4	V1-42	V1-10	V1-79	V1-28	V1-45	V1-11

304 STAINLESS STEEL STRIP

V1-13	V1-70	V1-9	V1-33	V1-60	V1-22	V1-49	V1-56
L1-29	L1-71	L1-18	L1-65	L1-7	L1-52	L1-28	L1-51
X1-67	X1-50	X1-31	X1-13	X1-3	X1-69	X1-7	X1-64

← CORE →
 ← UPPER BLANKET →

MATERIAL SYMBOL

Hastelloy-X	X1
Incoloy-800	L1
Inconel-625	V1

(a) See Figure A3

Figure A-1. Fluence Profile of MT-3 Materials Capsule

significant difference in volume and internal heat generation rate exists between the ends and the center. With only a relatively small amount of axial heat transfer possible, the ends run considerably hotter than the center.

To specify this temperature variation more accurately, a tensile specimen near the core centerline was isolated and similar calculations rerun. The resulting temperature distribution is shown in Figure A-4.

A-5 COMPARISON WITH PREVIOUS THERMAL CALCULATIONS

A one-dimensional thermal calculation was made by D. Miller in 1966¹ which predicted an average specimen temperature of 1120°F (604°C) at the core midplane for 45 MW operation. This is significantly lower than the 1225 to 1230°F (663-666°C) average temperature calculated in this study. However, several errors were found in the original calculations, the most significant being the use of 0.5 surface emissivity for radiation heat transfer rather than the combined effective emissivity of approximately 0.39. Corrected one-dimensional calculations indicate an average specimen temperature of 1225°F (663°C), almost exactly that predicted by this analysis.

A-6 ESTIMATE OF THERMAL UNCERTAINTY

The largest uncertainty in the thermal calculations is that due to the local heating rate. This includes overall reactor power level, local rod variations due to adjacent assemblies, and the axial profile shape. The uncertainty is estimated to be $\pm 20\%$. The heating rate uncertainty dominates the thermal calculations to such an extent that all other uncertainties can be neglected. Since going from 45 to 50 MW is a 10% increase in power, thermal uncertainties can be estimated by doubling the temperature difference in each node between these power levels. Therefore, the maximum temperature uncertainty for the tensile specimens is approximately $\pm 90^\circ\text{F}$ (50°C) at the core midplane. This estimate is based only upon the calculated values. When the observed behavior of the melting alloy temperature sentinels are taken into account, the estimate of uncertainty may be lowered to $\pm 25^\circ\text{F}$ (14°C).

Table A-3

**REACTOR HISTORY FOR MT-3 MATERIALS CAPSULE -
SUBASSEMBLY XA08**

Run	Reactor Exposure (MWd)	Peak Power (MW)	Starting Date	Number of Days	
1	343		8- 1-64	22	
2	362		10- 2-64	17	
3	575		3-15-65	26	
4	351		4-24-65	10	
5	372	45	5-10-65	11	
6	499	45	5-24-65	32	
7	425	45	7-29-65	17	
8	393	45	9- 7-65	12	
9	345	45	10-13-65	8	
10	315	45	10-28-65	10	
11	300	45	11-18-65	9	
12	470	45	11-30-65	11	
13	300	45	12-17-65	7	
14	520	45	1-10-66	12	
15	795	45	3-28-66	17	
16	775	45	3-17-66	21	
17	475	45	5-11-66	12	
18	867	45	5-26-66	20	
19	89	45	6-27-66	2	XA08
20	699	45	7-19-66	19	
21	610	45	8-12-66	15	
22	1045	45	10-20-66	25	
23	690	45	11-16-66	17	
24	630	45	12- 6-66	25	
25A	641	45	4-17-67	63	
25B	150	45	6-21-67	6	
25C	723	45	6-29-67	21	
25D	34	45	7-21-67	1	
25E	13	45	7-23-67	26	
26A	87	45	9-22-67	7	XA08
26B	1029	45	10-11-67	40	
26C	619	45	11-22-67	21	
27A	283	45	2- 2-68	27	
27B	177	45	3- 1-68	4	
27C	62	45	3- 7-68	4	XA08

Table A-3 (Continued)

Run	Reactor Exposure (MWd)	Peak Power (MW)	Starting Date	Number of Days	
27D	286	45	4- 6-68	7	
27E	90	45	4-11-68	4	
27F	68	45	4-16-68	3	
27G	49	45	4-19-68	2	
27H	206	45	5- 2-68	7	
27I	96	45	5- 6-68	3	
28A	154	45	5-13-68	4	
28B	303	45	5-27-68	12	
28C	669	45	6-15-68	17	
29A	188	45	7- 5-68	9	
29B	24	45	7-11-68	2	
29C	205	45	7-22-68	7	
29D	710	45	8-16-68	22	
30A	652	50	9- 5-68	15	
30B	166	50	9-11-68	4	
30C	52	30	9-15-68	4	
30D	79	30	9-21-68	4	
30E	79	30	9-22-68	3	
31A	89	30	9-30-68	4	
31B	159	30	10-11-68	7	
31C	84	30	10-19-68	3	
31D	372	50	10-26-68	8	
31E	0	0.08	11- 4-68	1h	
31F	413	50	11- 5-68	9	
31G	111	11	11-15-68	2	
32A	852	50	11-27-68	20	
32B	249	50	12-18-68	6	
32C	72	50	12-25-68	2	
32D	244	50	12-28-68	5	
33A	599	50	2-19-69	17	
33B	577	50	4- 7-69	14	
34A	822	50	4-25-69	18	
34B	310	50	5-14-69	7	
35	1202	50	5-29-69	28	
36A	800	50	7-22-69	9	
37	1196	50	8-28-69	28	
38A	770	62.5	9-10-69	19	
38B	600	50	10-3-69	12	

Total: 19278 Megawatt-days

Figure A-2. Axial Temperature Distribution of MT-3 Capsule Tube

Figure A-3. Axial Temperature Profile, MT-3 Capsule Type-304 Stainless Steel Strip and Tensile Specimens

Table A-4

SUMMARY OF IRRADIATION AND THERMAL EXPOSURE

Region	Reactor Power Level (MW)	Exposure Time (h)	Temperature ^(a) (°F)
1	50	1,760	1191
	45	5,550	1156
	<45	4,160	<1156
2	50	1,760	1214
	45	5,550	1179
	<45	4,160	<1179
3	50	1,760	1214
	45	5,550	1179
	<45	4,160	<1179
4	50	1,760	1192
	45	5,550	1157
	<45	4,160	<1157
5	50	1,760	1137
	45	5,550	1103
	<45	4,160	<1103
6	50	1,760	972
	45	5,550	948
	<45	4,160	< 948
7	50	1,760	736
	45	5,550	732
	<45	4,160	< 732
8	50	1,760	733
	45	5,550	730
	<45	4,160	< 730

(a) Temperature at the center of each tensile specimen.

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Figure A-4. Temperature Profile of the Tensile Specimens in Region 2 for Reactor Operation at 45 MW-Thermal

APPENDIX B

ANALYSIS OF ERRORS INCURRED IN THE
TEM EXAMINATION

U. E. Wolff

In the following analysis the magnitude of the errors and uncertainties associated with the different measurements is estimated and their effect on the quantitative results is assessed. For the purpose of this discussion, the error is defined as two times the standard deviation, σ .

The total variance σ^2 of a function $F = F(a, b, c, \dots)$ can be written^{B-1} as:

$$\sigma_F^2 = \left(\frac{\partial F}{\partial a}\right)^2 \sigma_a^2 + \left(\frac{\partial F}{\partial b}\right)^2 \sigma_b^2 + \dots$$

where the σ_a , σ_b , etc. are the standard deviations of the individual parameters which define F . It is assumed that the parameters a , b , c , ... are independent variables.

In the case of void measurements on electron micrographs of thin foils, the calculated quantities are different functions of the following measured values:

M = magnification of each micrograph

A' = area of each micrograph (with the corresponding foil area $A = A'/M^2$)

d' = diameter (or diagonal) of each void measured on the micrograph (with the corresponding real diameter $d = d'/M$)

N_t = number of voids with their centers within the foil

N_s = number of voids with their centers outside the foil.

From these measured and counted values, the following quantities were calculated by a computer program written for the purpose:

$\bar{d}$ = mean void diameter (or diagonal)

N_v = number of voids per unit volume

f = volume fraction of voids.

The somewhat simplified functions relating the calculated to the measured values are as follows:

$$\bar{d} = \frac{1}{MN_t} \sum_{i=1}^{N_t} d'_i = F(M, d') \quad (B-1)$$

$$N_v = N_s / (A\bar{d}) = F(N_s, \bar{d}, A', M) \quad (B-2a)$$

or

$$N_v = N_t / tA = F(N_t, t, A', M) \quad (B-2b)$$

$f = \bar{v} N_v$ where $\bar{v}$ is the average volume per void. Thus

$$f = \frac{CN_s \bar{d}^3}{A' \bar{d}} = F(N_s, d', A') \quad (B-3a)$$

or

$$f = \frac{CN_t \bar{d}^3 M}{A' t} = F(N_t, d', A', t, M) \quad (B-3b)$$

C is a factor relating the linear void dimension to the void volume. For spheres $C = \pi/6 = 0.554$; for octahedra $C = \sqrt{2}/3 = 0.578$.

Where the foil thickness was determined from the number of surface and internal voids* the following equation was used:

$$t = \bar{d} N_t / N_s$$

For derivation of the equations (B-1) to (B-4), see references (B-2, B-3).

The standard deviations of the individual quantities M , A' , d' , $\bar{d}$, t , N_t and N_s must now be estimated and other sources of error assessed.

1. The magnification M of electron micrographs is determined from the intermediate lens current via a calibration curve obtained with a grating replica. The accuracy of the magnification determination depends on the accuracy of the current setting, on the effect of the permanent magnetism in the microscope lenses, and on the distance of the specimen plane from the objective lens focal plane. The latter distance is a function of the total thickness of the specimen disk; it was found that the magnification decreased about 1% for each 25 μm (0.001 inch) of additional distance beyond the normal location of a specimen screen. The approximate location of the foil plane was 75 μm higher than normal; this was taken into account for the magnification determinations. After all known corrections have been made, an unknown uncertainty remains, and σ_M is estimated to be 0.025 M .
2. The area A' of an electron micrograph can be measured to about $\pm 10 \text{ mm}^2$ (i.e., $\sigma_{A'} = 5 \text{ mm}^2$). Since A' is on the order of 20,000 to 30,000 mm^2 , $\sigma_{A'}/A' < 2.5 \times 10^{-4}$.
3. The measurement of the individual Void diameters d' on enlarged prints is affected by imperfectly focused micrographs, by odd shapes of voids, by poor contrast in thicker foils and by the width of the size classes into which the Zeiss particle size analyzer groups the diameter measurements. The first three of these effects introduce an uncertainty which is different for different micrographs and might be as large as $2 \sigma = 1 \text{ mm}$. The class width of the analyzer is 0.55 mm or 0.18 mm depending on the range setting used. These combined uncertainties result in a maximum estimated measuring deviation of $\sigma_{d'} = 0.8 \text{ mm}$. For voids of 2 to 20 mm diameter (on the micrographs) the range for $\sigma_{d'}/d'$ would be 0.4 to 0.04, i.e., for the smallest voids the error is largest.
4. For the mean, d' , σ is inversely proportional to the square root of the number N_t of voids measured. $\sigma_{d'} = \sigma_{d'}/\sqrt{N_t}$. The number N_t is different for each specimen; it is largest (i.e., the error smallest) for specimen A1 and smallest for specimen B3.
5. A major source of uncertainty is the foil thickness t . The accuracy of the foil thickness determination does not affect the void size determination, but it does affect the result of all determinations which depend on the volume examined, e.g., the void volume fraction and the number of voids per unit volume. Where the voids are small compared with the foil thickness (i.e., $< 500 \text{ \AA}$), the method of Sellars and Smith can be used for foil thickness determination.⁷ This method depends on the ability of the investigator to recognize surface voids (i.e., N_s) as contrasted to voids lying within the foil (i.e., N_t). This is not always possible and in any case is certainly not reliable. The only justification for using this method is that it gives not unreasonable results. It has been used (with the modification of the definition of surface voids previously noted only for the foils A1 and O4. For foils with larger voids, the foil thickness in most cases has been estimated without any help except the knowledge that 100 kV electrons can penetrate steel of, at most, about 3000 $\text{\AA}$ thickness, and that foils of less than 1000 $\text{\AA}$ thickness are rare. Consequently, for most of the foils the thickness was assumed to be around 1500 to 2000 $\text{\AA}$. One can intuitively estimate the uncertainty to be about $\pm 1500 \text{ \AA}$ or $\sigma_t = 750 \text{ \AA}$ and $\sigma_t/t = 0.25$ to > 1.00 .

* Surface voids should be defined as those intersected by the surfaces of the foil; and interior voids should be defined as those lying totally within the foil. However, this treatment gave thickness results far beyond the capability of the 100 kV electron microscope. Therefore, in this investigation "surface voids" were believed to be those with their centers outside the foil and interior voids those with their centers within the foil.

The most reliable thickness determination is that using trace analysis. This method can be used only when suitable traces (slip bands or twin boundaries) are in the field of the micrograph. It also is time consuming. The error associated with trace analysis depends on the accuracy with which the foil orientation can be determined. This is about $\pm 5^\circ$ for ordinary diffraction spot analyses. To this must be added any possible foil tilt around a usually unknown axis, making the estimated angular error about $\pm 10^\circ$. This results in a possible thickness error with σ_t/t of about 0.10.^{B-2} Such trace analyses were made for about half a dozen electron micrographs. The results were consistent with the other thickness estimates made.

6. The errors associated with N_t and N_s depend mainly on the quality of the electropolish, on the quality of the micrograph (focusing, contrast, etc.), and on the foil thickness. The error is approximately independent of the number of voids.

The surface voids are much more difficult to detect than most of the internal voids. The errors are estimated as follows:

$$\sigma_{N_t}/N_t = 0.05$$

$$\sigma_{N_s}/N_s = 0.25$$

Calculation of the total variance for each of the functions (equations B-1 through B-3) follows:

(B-1)

$$\bar{d} = \frac{1}{MN_t} \sum_{i=1}^{N_t} d'_i$$

$$\frac{\partial \bar{d}}{\partial M} = -\frac{\bar{d}}{M}; \sigma_M = 0.025M$$

$$\frac{\partial \bar{d}}{\partial d'_i} = \frac{1}{MN_t}; \sigma_{d'} = 8 \times 10^6 [\text{\AA}]$$

$$\sigma_{\bar{d}}^2 = \frac{\bar{d}^2}{M^2} \sigma_M^2 + \frac{1}{M^2 N_t} \sigma_{d'}^2$$

(B-5)

$$\sigma_{\bar{d}}^2 = 6.25 \times 10^{-4} \bar{d}^2 + \frac{1}{M^2 N_t} \times 64 \times 10^{12} [\text{\AA}^2]$$

For example, A1 with $\bar{d} = 460 \text{ \AA}$, $M = 1.5 \times 10^5$, and $N_t \approx 2000$,

$$\sigma_{\bar{d}}^2 (A1) = 132 + 1.4; \sigma_{\bar{d}} (A1) \approx 12 \text{ \AA}.$$

For sample M3 with $\bar{d} = 1000 \text{ \AA}$, $M = 60,000$, and $N_t = 222$,

$$\sigma_{\bar{d}}^2 (M3) = 625 + 80; \sigma_{\bar{d}} (M3) \approx 27 \text{ \AA}.$$

(B-2a)

$$N_v = \frac{N_s M^2}{\bar{d} A'}$$

$$\frac{\partial N_v}{\partial N_s} = \frac{M^2}{\bar{d} A'}; \sigma_{N_s} = 0.25 N_s$$

$$\frac{\partial N_v}{\partial \bar{d}} = -\frac{N_s M^2}{\bar{d}^2 A'}; \sigma_{\bar{d}}^2 = \frac{1}{M^2} \left(\bar{d}^2 \sigma_M^2 + \frac{1}{N_t} \sigma_{\bar{d}}^2 \right)$$

$$\frac{\partial N_v}{\partial A'} = -\frac{N_s M^2}{\bar{d} A'^2}; \sigma_{A'} = 2.5 \times 10^{-4} A'$$

$$\frac{\partial N_v}{\partial M} = \frac{2 N_s M}{\bar{d} A'}; \sigma_M = 2.5 \times 10^{-2} M$$

$$\sigma_{N_v}^2 = \frac{M^4 N_s^2}{\bar{d}^2 A'^2} [6.25 \times 10^{-2} + \frac{1}{\bar{d}^2} \sigma_{\bar{d}}^2 + 6.25 \times 10^{-8} + 25 \times 10^{-4}] \quad (B-6)$$

It was shown above that the second term in $\sigma_{\bar{d}}^2$ is small compared with the first. In the first approximation, therefore,

$$\sigma_{\bar{d}}^2 \approx 7 \times 10^{-4} \bar{d}^2 + [A^2].$$

Substituting this value into equation (B-6) one obtains,

$$\sigma_{N_v}^2 \approx N_v^2 (6.25 \times 10^{-2} + 7 \times 10^{-4} + 6.25 \times 10^{-8} + 25 \times 10^{-4}). \quad (B-6a)$$

Again, the second, third and fourth term are negligible. Therefore,

$$\sigma_{N_v}^2 \approx 6.5 \times 10^{-2} N_v^2; \sigma_{N_v} \approx 0.26 N_v.$$

The reason for this large value of σ_{N_v} is the unreliability of the N_s count, i.e., the difficulty of identifying surface voids.

(B-2b) The alternate equation for N_v is,

$$N_v = \frac{N_t M^2}{t A'}$$

$$\frac{\partial N_v}{\partial N_t} = \frac{M^2}{t A'}; \sigma_{N_t} = 0.05 N_t$$

$$\frac{\partial N_v}{\partial t} = -\frac{N_t M^2}{t^2 A'}; \sigma_t = 0.5 t$$

$$\frac{\partial N_v}{\partial A'} = - \frac{N_t M^2}{t A'^2}; \sigma_{A'} = 2.5 \times 10^{-4} A'$$

$$\frac{\partial N_v}{\partial M} = \frac{2 N_t M}{t A'}; \sigma_M = 2.5 \times 10^{-2} M$$

$$\sigma_{N_v}^2 = \frac{N_t^2 M^4}{t^2 A'^2} [25 \times 10^{-4} + 0.25 + 6.25 \times 10^{-8} + 25 \times 10^{-4}] \quad (B-6b)$$

The second term is overriding all others, and one obtains,

$$\sigma_{N_v}^2 \approx 0.25 N_v^2; \sigma_{N_v} \approx 0.5 N_v$$

Here, again, the uncertainty in the foil thickness has the main effect on σ_{N_v} .

(B-3a) The volume fraction is independent of magnification,

$$f = \frac{C N_s \bar{d}^{1/3}}{A' \bar{d}'}$$

$$\frac{\partial f}{\partial N_s} = \frac{C \bar{d}^{1/3}}{A' \bar{d}'}; \sigma_{N_s} = 0.25 N_s$$

$$\frac{\partial f}{\partial A'} = - \frac{C N_s \bar{d}^{1/3}}{A'^2 \bar{d}'}; \sigma_{A'} = 2.5 \times 10^{-4} A'$$

$$\frac{\partial f}{\partial \bar{d}'} \approx \frac{2 C N_s \bar{d}^{1/3}}{A'}; \sigma_{\bar{d}'} = \sigma_{\bar{d}'} / \sqrt{N_t} < 0.4 \frac{\bar{d}'}{\sqrt{N_t}}$$

$$\sigma_f^2 \approx f^2 \left[6.25 \times 10^{-2} + 6.25 \times 10^{-8} + \frac{0.64}{N_t} \right] \quad (B-7a)$$

With the first term being the only significant one whenever $N_t > 100$:

$$\sigma_f^2 \approx 0.07 f^2 \quad \sigma_f \approx 0.27 f$$

(B-3b) For those cases where t has been estimated, the equation for the volume fraction is,

$$f = \frac{C N_t \bar{d}^{1/3} M}{A' t}$$

$$\frac{\partial f}{\partial N_t} = \frac{C \bar{d}^{7/3} M}{A' t} ; \sigma_{N_t} = 0.05 N_t$$

$$\frac{\partial f}{\partial A'} = - \frac{C N_t \bar{d}^{7/3} M}{A'^2 t} ; \sigma_{A'} = 2.5 \times 10^{-4} A'$$

$$\frac{\partial f}{\partial t} = - \frac{C N_t \bar{d}^{7/3} M}{A' t^2} ; \sigma_t = 0.5 t$$

$$\frac{\partial f}{\partial \bar{d}'} \approx \frac{3C \bar{d}'^{1/2} N_t M}{A' t} ; \sigma_{\bar{d}'} = \sigma_{\bar{d}'} / \sqrt{N_t} < 0.4 \frac{\bar{d}'}{\sqrt{N_t}}$$

$$\frac{\partial f}{\partial M} = \frac{C N_t \bar{d}'^{7/3}}{A' t} ; \sigma_M = 0.025 M$$

$$\sigma_f^2 \approx f^2 \left[25 \times 10^{-4} + 6.25 \times 10^{-8} + 0.25 + \frac{1.44}{N_t} + 6.25 \times 10^{-4} \right] \quad (B-7b)$$

The only significant term is the third one whenever $N_t > 60$:

$$\sigma_f^2 \approx 0.25 f^2 \quad \sigma_f \approx 0.5 f$$

The standard deviations calculated for the computed values are listed in the table below:

Table B-1 STANDARD DEVIATIONS

Value	Standard Deviation σ
Mean void diameter, $\bar{d}$	$\sqrt{6.25 \times 10^{-4} \bar{d}^2 + \frac{64 \times 10^{12}}{M^2 N_t}} [\text{\AA}]$
Number of voids per unit volume, N_v	$0.26 N_v^{(a)}$
	$0.5 N_v^{(b)}$
Void volume fraction, f	$0.3 f^{(a)}$
	$0.5 f^{(b)}$
(a) foil thickness calculated from number of surface voids. (b)	
(b) foil thickness estimated.	

7. In addition to these errors which can be assessed quantitatively, other sources of error have to be considered.

The mean void size most probably is biased toward larger voids. The reason for this is that measurements on the larger voids might be in error because they have been enlarged by the electropolishing procedure. Theoretically, only those voids should be measured which lie totally within the foil. However, in the practical case, voids might be thought of as internal when in fact they are intersected by a surface. This will happen with increasing frequency as the void size increases. When the void size is comparable to the foil thickness, all voids obviously have to be intersected by one or both foil surfaces. Therefore, any void measurement greater than about 2000 Å should be viewed with suspicion. It is possible that the mode of the void size (i.e., the peak of the distribution) would be a better measure of "average" void size than the mean. In those cases where a sufficient number of voids have been measured to obtain a good distribution curve, the peak of the distribution is about 10% lower than the mean void size.

A second kind of bias which is unaccounted for, is introduced by the operator during void measurement. In the few cases where duplicate measurements of the same voids have been made by different operators the void diameter results have differed by $\pm 16\%$. Any such bias is independent of the number of measurements and can shift all results in one direction or the other without destroying the self-consistency of the whole set.

A further source of uncertainty is the small volume examined and the possible **nonuniform void distribution**. The volume represented by all the micrographs from a given sample is on the order of 10^{-7} to 10^{-8} mm³. Within this volume only 100 to 2000 voids were counted. In addition, in some of the specimens the void density was quite nonuniform, with some micrographs containing no voids while others contained a considerable number. The statistical reliability of the counts is therefore low, especially for the specimens with few and large voids.

In Summary;

- (a) The mean void diameter has a probable error, 2σ , that depends on the void size and the number of voids measured, and on the quality of the micrographs. In most cases, the relative error is about ± 5 to 6%. However, a considerable additional uncertainty of at least 20% is introduced by operator bias.
- (b) The volume fraction and the number of voids per unit volume depend strongly on the foil thickness which cannot be measured accurately. The relative errors associated with those values are about 50 to 100%. However, nonuniform void distribution will further increase the uncertainty by an unknown amount.

ACKNOWLEDGMENT

A critical review of this Appendix and helpful suggestions by S. A. Wilson is gratefully acknowledged.

REFERENCES

- B-1 Young, H. D., *Statistical Treatment of Experimental Data*, McGraw-Hill, p. 96 ff.
- B-2 Hirsch, Howie, Nickolson, Pashley, and Wheelan, *Electron Microscopy of Thin Crystals*, New York; Plenum Press, London, Butterworth, 1965, pp. 418-419.

APPENDIX C

RELATION OF VOID VOLUME FRACTION
TO VOLUME INCREASE

The relation between void volume fraction, f , as measured by TEM, and volume increase as determined by dimensional measurements or immersion density, is as follows.

For the purpose of discussion let us choose an initial volume, V_o , which during irradiation has swelled by ΔV , so that its final volume, V_f , is the volume investigated in the TEM.

The two terms are defined as follows:

$$\text{VOID VOLUME FRACTION} \equiv f \equiv \frac{\Delta V}{V_f}$$

$$\text{VOLUME INCREASE} \equiv \frac{\Delta V}{V_o}$$

and the two terms are related by $V_f = V_o + \Delta V$

from which it can be seen that

$$\frac{\Delta V}{V_o} = \frac{\Delta V}{V_f - \Delta V} = \frac{f}{1 - f}$$

or

$$f = \frac{\Delta V}{V_f} = \frac{\Delta V}{V_o + \Delta V} = \frac{\Delta V}{V_o} \frac{1}{\left(1 + \frac{\Delta V}{V_o}\right)}$$

A similar analysis^{C-1} shows that density change and volume change are related in an analogous fashion, namely:

$$\frac{\Delta V}{V_o} = \frac{\left(\frac{-\Delta \rho}{\rho}\right)}{1 + \left(\frac{\Delta \rho}{\rho}\right)}$$

Thus void volume fraction and density change are identical except for opposite sign.

REFERENCES

- C-1 Comprelli, F.A., Oldberg, S., Sandusky, D., *Empirical Analysis of Swelling of Austenitic Stainless Steels in Fast Reactors*, September 1969 (GEAP-13517).

APPENDIX D

D-1 MT-3 Tensile Test Results

Tensile tests were run on unirradiated specimens fabricated from the same lot as the specimens irradiated in MT-3. Table D-1 gives the data on the unirradiated specimens and Table D-2 covers the irradiated ones. The tabulation includes test temperature, strain rate, yield stress, ultimate tensile stress and elongation; Table D-2 includes specimen location and neutron fluence.

Table D-1

**TENSILE TEST PROPERTIES (UNIRRADIATED)
GROUP MT3**

Specimen Number	Alloy Type	Heat Number	Test Temperature		Cross Head Speed Strain Rate in./in./min	Stress (ksi)		Elongation (%)	
			(°C)	(°F)		0.2% Yield Strength	Ultimate	Uniform	Total
V141	I625	Y8668	704	1300	2.0000	46.1	108.0	55.2	57.2
V120	I625	Y8668	704	1300	0.0200	42.5	82.4	18.4	35.1
V157	I625	Y8668	704	1300	0.0002	43.0	62.6	14.0	14.7
V182	I625	Y8668	593	1100	2.0000	71.6	109.0	31.6	36.8
V165	I625	Y8668	593	1100	0.0200	58.3	114.3	NA	NA
V151	I625	Y8668	593	1100	0.0200	45.2	118.0	NA	NA
V162	I625	Y8668	593	1100	0.0200	47.0	117.4	47.0	48.4
V133	I625	Y8668	593	1100	0.0002	46.6	96.6	16.6	17.0
V168	I625	Y8668	593	1100	0.0002	78.8	105.0	10.0	10.6
V138	I625	X8668	482	900	0.0200	43.5	113.0	47.0	47.8
V172	I625	Y8668	482	900	0.0200	45.3	110.0	44.6	45.6
V147	I625	Y8668	593	1100	2.0000	50.0	108.0	44.0	47.2
L136	I800	9696A	704	1300	2.0000	24.3	49.3	26.0	40.8
L162	I800	9696A	704	1300	0.0200	25.5	36.8	14.0	61.0
L16	I800	9696A	704	1300	0.0002	16.3	16.6	4.4	89.9
L130	I800	9696A	593	1100	2.0000	26.7	61.7	28.0	31.2
L117	I800	9696A	593	1100	0.0200	26.5	57.5	26.4	50.5
L115	I800	9696A	593	1100	0.0002	26.6	41.7	17.1	61.7
L11	I800	9696A	482	900	0.0200	28.7	74.2	32.2	35.0
L166	I800	9696A	482	900	0.0200	29.6	74.8	33.0	34.3
X162	Hastx	X14370	704	1300	2.0000	30.7	78.0	41.2	44.8
X161	Hastx	X14370	704	1300	0.0200	27.4	62.1	30.0	50.0
X184	Hastx	X14370	704	1300	0.0002	29.3	37.0	5.7	57.6
X127	Hastx	X14370	593	1100	2.0000	31.7	86.7	45.0	48.4
X186	Hastx	X14370	593	1100	2.0000	30.0	83.4	43.2	49.6
X152	Hastx	X14370	593	1100	0.0200	31.4	92.0	48.0	49.4
X178	Hastx	X14370	593	1100	0.0200	27.6	84.2	45.0	47.0
X11	Hastx	X14370	593	1100	0.0002	28.3	69.0	30.7	60.0
X151	Hastx	X14370	482	900	2.0000	30.0	84.0	47.6	50.8
X121	Hastx	X14370	482	900	0.0200	30.5	93.2	45.0	46.8
X123	Hastx	X14370	482	900	0.0200	26.7	86.6	45.4	47.6
X149	Hastx	X14370	482	900	0.0200	26.3	85.0	49.0	49.8
X153	Hastx	X14370	482	900	0.0200	28.4	85.0	48.8	52.0
X174	Hastx	X14370	482	900	0.0200	29.4	90.0	50.6	51.6
X187	Hastx	X14370	482	900	0.0200	22.7	78.4	52.6	54.4
X1555	Hastx	X14370	482	900	0.0200	22.7	78.5	53.6	56.2
X138	Hastx	X14370	593	1100	2.0000	24.0	71.7	52.8	55.2

NA = Not Available - Not Readable From Chart

Table D-2
TENSILE TEST PROPERTIES (IRRADIATED)
GROUP MT3

Specimen Number	Alloy Type	Heat Number	Test		Cross Head Speed	Stress (ksi)				Inches	Irradiation Conditions		
			Temperature C F	Strain Rate in. /in. /min	0.2% Yield Strength	Ultimate	Elongation (%)		Above Midplane	Fluence X 10 ²² > 0.1 MeV	Temperature		
							Uniform	Total			C	F	
V179	I625	Y8668	704	1300	2.0000	166.5	171.5	6.4	10.8	5.0	4.3	595	1103
V122	I625	Y8668	704	1300	0.0200	105.0	109.0	1.7	1.8	7.0	3.5	509	948
V133	I625	Y8668	704	1300	0.0200	86.0	98.4	2.8	2.9	3.0	5.0	625	1157
V142	I625	Y8668	704	1300	0.0200	79.0	81.7	1.3	1.4	1.0	5.4	637	1179
V1 9	I625	Y8668	704	1300	0.0002	58.0	58.0	1.3	1.4	1.0	5.4	637	1179
V113	I625	Y8668	593	1100	2.0000	46.7	46.7	1.2	2.4	-3.0	5.0	624	1155
V170	I625	Y8668	593	1100	2.0000	112.7	140.0	15.2	18.4	-1.0	5.4	637	1179
V110	I625	Y8668	593	1100	0.0200	102.3	135.0	5.3	5.4	3.0	5.0	625	1157
V128	I625	Y8668	593	1100	0.0200	129.0	132.0	1.9	2.0	7.0	3.5	509	948
V1 4	I625	Y8668	593	1100	0.0002	111.0	125.0	3.0	3.1	-1.0	5.4	637	1179
V150	I625	Y8668	482	900	0.0200	102.7	148.5	15.4	15.5	-3.0	5.0	624	1155
V160	I625	Y8668	NA	NA	NA	NA	NA	NA	NA	5.0	4.3	595	1103
L1 5	I800	9696A	704	1300	2.0000	28.4	48.4	17.6	19.2	5.0	4.3	595	1103
L1 7	I800	9696A	704	1300	0.0200	24.3	33.7	7.0	10.2	5.0	4.3	595	1103
L152	I800	9696A	704	1300	0.0200	26.3	34.6	2.8	3.3	7.0	3.5	509	948
L155	I800	9696A	704	1300	0.0200	23.3	23.6	0.7	1.6	1.0	5.4	637	1179
L165	I800	9696A	704	1300	0.0200	21.3	28.6	5.5	6.9	3.0	5.0	625	1157
L118	I800	9696A	704	1300	0.0002	17.0	17.3	0.5	15.4	1.0	5.4	637	1179
L112	I800	9696A	593	1100	2.0000	25.0	58.2	24.8	30.8	-3.0	5.0	624	1155
L171	I800	9696A	593	1100	2.0000	28.3	58.2	24.0	26.4	-1.0	5.4	637	1179
L145	I800	9696A	593	1100	0.0200	35.0	56.6	13.3	14.8	7.0	3.5	509	948
L167	I800	9696A	593	1100	0.0200	25.6	51.7	14.3	14.4	3.0	5.0	625	1157
L116	I800	9696A	593	1100	0.0002	27.0	32.3	4.5	5.2	-1.0	5.4	637	1179
L129	I800	9696A	482	900	0.0200	29.3	58.2	13.5	14.9	-3.0	5.0	624	1155
X131	Hastx	X14370	705	1302	0.0002	38.0	39.4	2.4	2.9	1.0	5.4	637	1179
X137	Hastx	X14370	704	1300	2.0000	93.4	98.4	6.8	9.2	5.0	4.3	595	1103
X113	Hastx	X14370	704	1300	0.0200	52.7	NA	NA	NA	3.0	5.0	625	1157
X140	Hastx	X14370	704	1300	0.0200	48.0	72.0	6.7	7.2	1.0	5.4	637	1179
X169	Hastx	X14370	704	1300	0.0200	62.6	68.4	3.1	3.2	7.0	3.5	509	948
X136	Hastx	X14370	593	1100	2.0000	79.0	107.8	8.4	10.8	-3.0	5.0	624	1155
X150	Hastx	X14370	593	1100	2.0000	78.3	95.5	7.8	10.0	-1.0	5.4	637	1179
X1 4	Hastx	X14370	593	1100	0.0200	53.3	101.2	8.5	8.6	3.0	5.0	625	1157
X170	Hastx	X14370	593	1100	0.0200	71.4	103.0	3.8	3.9	7.0	3.5	509	948
X134	Hastx	x14370	593	1100	0.0002	52.3	74.4	4.8	5.3	-1.0	5.4	637	1179
X1 3	Hastx	X14370	482	900	0.0200	84.3	131.0	11.6	11.7	5.0	4.3	595	1103
X167	Hastx	X14370	482	900	0.0200	52.7	62.0	1.4	1.5	-3.0	5.0	624	1155

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