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2.1 STATUS OF MFE-5 IN-REACTOR FATIGUE CRACK GROWTH EXPERIMENT -

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2.1.1 ADIP Task

ADIP Task I.B.1, "Fatigue Crack Growth in Austenitic Alloys"
(Path A).

2.1.2 Objectives

An apparatus has been developed to perform an in-reactor fatigue crack propagation test on the Path A Reference Alloy. Effects of dynamic irradiation on crack growth behavior will be evaluated by comparing the results with those of unirradiated and postirradiated tests.

2.1.3 Summary

The in-reactor fatigue machine has been inserted into the ORR and cycling on a chain of 20% cold-worked 316 stainless steel specimens has commenced. Because of the uncertainties in the γ -heating profile, the temperature profile across the specimen chain could not be optimized. However, this temperature profile was duplicated for the thermal control test, and cycling on this second chain of specimens has been initiated at HEDL.

2.1.4 Progress and Status

2.1.4.1 Introduction

Fatigue crack propagation (FCP) in the first wall of a magnetic fusion reactor may be a limiting quantity governing reactor lifetimes. Previous studies of irradiation effects on FCP have all be conducted out of reactor on materials preirradiated in the unstressed condition. The ORR-MFE-5 experiment will investigate FCP during irradiation, where dynamic irradiation may effect crack growth characteristics.

2.1.4.2 In-Reactor FCP Test at ORNL

The in-reactor fatigue machine^(1,2) was inserted into the ORR during the reactor shutdown period in November. After the reactor was brought up to full power, a large temperature gradient existed across the 58cm (23 in) specimen chain. After adjustments to the helium-neon mixtures through the three annular regions surrounding the capsule were made, the temperature profile did improve; but it was apparent that a flat temperature profile would not be attainable.

The temperature profile which was ultimately selected for MFE-5 is shown in Figure 2.1.1. The peak temperature of 460°C (860°F) can be easily controlled, while slight variations in the outermost temperatures occur during burnup and after refueling. The original target temperature was 425°C (797°F) over the entire length of the capsule. The large gradient can be explained in terms of uncertainties in the γ -heating profile which was used in the design of the capsule. In addition, a finite amount of mixing of the gas blends across the sliding seals which separate the three regions is believed to have prevented the good independent temperature control anticipated for the regions.

Since the test is being conducted in sodium, the direct effects of temperature on growth rate should be minimal since FCP in sodium is not strongly thermally activated up to about $0.5 T_m$ ⁽³⁾. Nevertheless, the temperature gradient observed for the in-reactor test was duplicated for the thermal control test.

Cycling of the in-reactor test was begun on December 4, 1980, and has achieved 58,000 cycles to date.

2.1.4.3 Thermal Control Test at HEDL

In order to duplicate the temperature profile of the in-reactor test, a furnace arrangement different from that which was previously described⁽⁴⁾ was used. A 1000 watt heating element was coiled around the central part of the assembly to provide the primary heating. The bottom of the assembly was inserted into a heated furnace, while the top was loosely wrapped with foil and exposed to the air. The resulting temperature profile is very similar to that of Figure 2.1.1.

FIGURE 2.1.1.1 Temperature Profile for the MFE-5 In-Reactor
FCP Experiment.

Cycling of the test has been initiated, with shutdowns scheduled to coincide with those of the in-reactor test.

2.1.5 Conclusions

(a) The MFE-5 in-reactor FCP test has been initiated in the ORR. The test has attained 58,000 cycles.

(b) The MFE-5 thermal control test has been initiated, duplicating the temperature and cycle history of the in-reactor test.

2.1.6 References

1. A. M. Ermi, "Status of an In-Reactor Fatigue Crack Growth Experiment", *ADIP Quarterly Progress Report, June 30, 1979*, DOE/ET-0058/6, pp. 28-34.
2. A. M. Ermi, "Results of Prototypic Testing for the MFE-5 In-Reactor Fatigue Crack Propagation Experiment", *ADIP Quarterly Progress Report, September 30, 1979*, DOE/ET-0058/7, pp. 50-65.
3. A. M. Ermi, "Fatigue Crack Growth Testing of 316SS for MFE-5", *ADIP Quarterly Progress Report, June 30, 1980*, DOE/ER-0045/3, pp. 42-47.
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5.2 TITANIUM ALLOY TENSILE PROPERTIES AFTER NEUTRON IRRADIATION —
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Development Laboratory)

5.2.1 ADIP Task

The Department of Energy DOE/Office of Fusion Energy (OFE) has cited the need for data under the following ADIP Program Plan Task⁽¹⁾:
I.B.15. Tensile Properties of Reactive/Refractory Alloys.

5.2.2 Objective

The objective of this work is to evaluate the effects of neutron irradiation on the tensile properties of titanium alloys. Assessment of mechanical property response to neutron irradiation will be a necessary step in choosing structural materials for fusion energy systems.

5.2.3 Summary

Irradiated specimens from three titanium alloys (6242S, 5621S, Ti-6Al-4V) were tested in uniaxial tension in air from room temperature to 550°C. Each alloy was in a duplex-annealed condition. The strain rate during testing was $4.2 \times 10^{-5} \text{ s}^{-1}$. The EBR-II irradiation temperature was 550°C; the maximum fluence was $5 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) or 24 dpa. The specimens were from the first of three capsules from the AD-1 experiment. The remaining two had irradiation temperatures of 394 and 450°C; tensile tests will be performed on material from these capsules in the near future.

Alloy 6242S increased in strength because of reactor exposure by approximately 20% at test temperatures from room to 400°C. Ductility losses of up to 75% were found.

The strength of 5621S seemed to be unaffected by reactor exposure while its ductility was reduced from that of unirradiated material by up to 80%.

Ti-6Al-4V displayed strength reductions at 550°C test temperature with nearly an 80% reduction in total elongation.

While large ductility losses were noted after reactor exposure, reasonable postirradiation ductility remained in the specimens. Total

elongations ranged from ~1-9%.

All of the specimens had some degree of corrosive attack, which probably occurred during cleaning after reactor discharge. The corrosion ranged from discoloration to loss of material. Although specimens with the least corrosion were chosen for testing, the mechanical properties could include corrosion effects as well as irradiation effects. Hence, the ductilities and ultimate strengths obtained in this study are minimum values.

5.2.4 Progress and Status

4.1 Introduction

Titanium alloys are potentially useful in fusion reactor structural applications because of favorable strength to weight ratios, low thermal stresses, and favorable coolant compatibility. In addition, the activity of titanium alloys decreases very rapidly, in comparison to stainless steels. On a strength to weight ratio and creep rupture basis elevated temperature alloys such as 6242S and 5621S are equal to or stronger than cold worked Type 316 stainless steel below 500°C. The effect of radiation to high neutron fluences on mechanical properties is unknown however.

4.2 Materials and Experimental Procedure

The alloys tested are given in Table 1. The alloys 5621S and 6242S were chosen because of their high strength, and Ti-6Al-4V because of its broad industrial usage base. The specimen dimensions are given in Figure 1. Specimens were fabricated from sheet stock supplied by the McDonnell-Douglas Astronautics Company of St. Louis. The alloys were originally manufactured by Reactive Metals Inc. (5621S and Ti-6Al-4V) and Titanium Metals Corporation (6242S).

Samples were irradiated in EBR-II in pin B286 of the AD-1 experiment. (2)
The irradiation temperature was 550°C, according to heat balance calculations. The uncertainty in temperature for uninstrumented capsules such as B286 is typically $\pm 30^\circ\text{C}$. The maximum fluence reached in B286 was $\sim 5 \times 10^{22} \text{ n/cm}^2 (E > 0.1 \text{ MeV})$ based on calculations from nominal reactor

TABLE 5.2.1
DESCRIPTION OF MATERIALS

ALLOY	COMPOSITION (%)				HEAT TREATMENT	
6242S (Heat N9882)	Al-5.8	Sn-1.9	Zr-4.0		900°C/½hr/Air Cool	} Duplex Anneal
	Mo-1.9	Si-0.08	C -0.018		785°C/¼hr/Air Cool	
	N -0.005	O -0.10	Fe-0.06			
5621S (Heat 802881)	Al-4.7	Sn-5.9	Zr-1.8		975°C/1hr/Air Cool	} Duplex Anneal
	Mo-0.78	Si-0.28	C -0.02	(vacuum)		
	N -0.009	O -0.09	Fe-0.04		595°C/2hr/Air Cool (vacuum)	
Ti-6Al-4V (Heat 891352)	Al-6.4	V -3.9	C -0.01		940°C/¼hr/Argon Cool	} Duplex Anneal
	N -0.01	O -0.12	Fe-0.21		675°C/¼hr/Argon Cool	

operating parameters. This is the highest neutron fluence obtained to date on any titanium alloy. The maximum dose was 24 dpa.

Tensile tests were performed in air on an Instron machine with a constant crosshead speed of 8.5×10^{-5} cm/s which yielded a strain rate of 4.2×10^{-5} s⁻¹ in the specimen gauge length. Several specimens were given an increase in crosshead speed subsequent to attaining the 0.2% yield load to decrease total test time. This procedure was not continued after a sample fractured during the crosshead speed change.

-5.2.4.3 Results

Postirradiation mechanical properties are given in Table 2. Two specimens, one Ti-6Al-4V and one 5621S, broke upon loading during testing at room temperature. This brittle behavior is attributed to corrosion.

Postirradiation strength and elongation results are presented in Figures 2 and 3 for all three alloys. The strongest alloy was 6242S. Its ultimate strength was nearly 40% stronger than 5621S and almost double that of Ti-6Al-4V. Elongation results were mixed, with 5621S and 6242S exhibiting fairly strong temperature dependencies. The minimum in total elongation for 5621S occurred at 400°C with values of 2.9 and 6.0%, while the minimum for 6242S occurred at 550°C at 3.3%. The elongation at failure of the single Ti-6Al-4V sample was 0.86%, at a test temperature of 550°C.

HEDL 8101-031.6

FIGURE 5.2.1 Postirradiation Tensile Specimen

TABLE 5.2.2
POSTIRRADIATION Ti ALLOY TENSILE PROPERTIES
($\dot{\epsilon} = 4.2 \times 10^{-5} s^{-1}$)

<u>Ti-6Al-4V</u>					
SPECIMEN ID	TEST TEMP. (°C)	0.2% YIELD STRENGTH (MPa)	ULTIMATE TENSILE STRENGTH (MPa)	UNIFORM ELONG. (%)	TOTAL ELONG. (%)
T2-52	550	394.4	417.8	0.74	0.86

<u>5621S</u>					
SPECIMEN ID	TEST TEMP. (°C)	0.2% YIELD STRENGTH (MPa)	ULTIMATE TENSILE STRENGTH (MPa)	UNIFORM ELONG. (%)	TOTAL ELONG. (%)
T5-70	200	659.1	788.8	7.0	8.7
T5-47	400	593.6	728.1	4.3	6.0
T5-58	400	547.4	628.1	1.8	2.9
T5-43	550	459.9	578.5	3.9	5.8

<u>6242S</u>					
SPECIMEN ID	TEST TEMP. (°C)	0.2% YIELD STRENGTH (MPa)	ULTIMATE TENSILE STRENGTH (MPa)	UNIFORM ELONG. (%)	TOTAL ELONG. (%)
T4-45	Room Temp.	926.7*	1252.8	5.9	5.9
T4-54	200	873.6*	**	2.4% at fracture	
T4-51	400	743.3	946.0	8.2	8.8
T4-44	550	620.5*	785.3	2.3	3.3

* Crosshead speed increased by 10X between 0.2% yield point and maximum load point.

** Broke during crosshead speed change.

HEDL 8101-031.4

FIGURE 5.2.2 Postirradiation Yield Strength and Ultimate Strength
[Irradiation Temp. = 550°C, $\phi t = 5.0 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$)

FIGURE 5.2.3 Postirradiation Uniform and Total Elongation
 [Irradiation Temp. = 550°C, $\phi t = 5.0 \times 10^{22} \text{ n/cm}^2 (E > 0.1 \text{ MeV})$]

Comparisons of postirradiation properties with properties of unirradiated material of similar composition and heat treatment are shown next in Figures 4-8. The ultimate strength of 6242S increased due to irradiation by approximately 20% at test temperatures of 400°C and below. The postirradiation yield strength was comparable to that of unirradiated material at room temperature but increased by approximately 20% at 200 and 400°C. Little effect of reactor exposure is apparent at the 550°C test temperature. Significant decreases in ductility were found in 6242S, as shown in Figure 5, although little ductility loss was noted in the test at 400°C. Unirradiated material results were from vendor certification reports and from Dotson.⁽³⁾ The trend line from Dotson represents an average of 20 tests.

A comparison of postirradiation and unirradiated material 5621S strength in Figure 6 indicates, somewhat surprisingly, no effect of reactor exposure. Yet a consistent trend of ductility loss is apparent in Figure 7. Unirradiated material results were from vendor certification reports and from single data points from Seagle and Bomberger.⁽⁴⁾

The single postirradiation test of Ti-6Al-4V resulted in strength decreases, principally in the ultimate strength, compared to averages from eight heats of unirradiated material, obtained from the Aerospace Structural Materials Handbook.⁽⁵⁾ A severe reduction in ductility occurred as a result of reactor exposure.

5.2.4.4 Discussion

Other investigators have irradiated titanium and its alloys with neutrons or heavy ions, but few mechanical property investigations have been undertaken. Hasse and Hartley⁽⁶⁾ irradiated Ti-6Al-4V to $\sim 2 \times 10^{21}$ n/cm² ($E > 0.1$ MeV) at 70°C. The room temperature ultimate strength increased by 40% and the elongation decreased by 70%. Sastry et al.⁽⁷⁾ tested the microhardness of Ti-6Al-4V and 5621S in the unirradiated condition and after irradiation to 3.4×10^{21} n/cm² ($E > 0.1$ MeV) at 450°C, and found hardness increases of ~ 20 -35%. Opperman⁽⁸⁾ and Nygren⁽⁹⁾ investigated the irradiation creep of titanium alloys under proton irradiation and neutron irradiation, respectively. Nygren's specimens had a fluence of 3.4×10^{21}

HEDL 8101-031.5

FIGURE 5.2.4 Effect of Neutron Irradiation on Strength of Alloy 6242S

HEDL 8101-031.2

FIGURE 5.2.5 Effect of Neutron Irradiation on the Total Elongation of Alloy 6242S

FIGURE 5.2.6 Effect of Neutron Irradiation on Strength of Alloy 5621S.

FIGURE 5.2.7 Effect of Neutron Irradiation on Total Elongation of Alloy 5621S.

FIGURE 5.2.8 Effect of Neutron Irradiation on Mechanical Properties of Ti-6Al-4V.

n/cm^2 ($E > 0.1$ MeV). The present work thus extends greatly the fluence and temperature range of the mechanical property data base.

Thermal exposure at 550°C, the irradiation temperature of the specimens tested in this work, can strengthen near- α alloys like 6242S and 5621S in subsequent lower temperature tests with little effect on strength at test temperatures near 550°C. An $\alpha + \beta$ alloy like Ti-6Al-4V will in general be softened by exposure at 550°C. Ductility of either type is not greatly affected by 550°C exposure. The strength increases of 6242S upon reactor exposure are thus at least partly due to aging effects. The net effect of irradiation and thermal exposure on 5621S was nil, while the softening observed in the Ti-6Al-4V specimen was undoubtedly a thermal effect. The ductility losses found with all of the alloys cannot be associated with thermal exposure, but must be irradiation or corrosion effects.

The corrosion experienced by the tensile specimens was discovered subsequent to cleaning after removal from the reactor. A significant proportion of the specimens of all alloys discharged from B286 were affected. The corrosion resulted in a dark mottled appearance, localized material removal, and in some cases separation of the specimen into two pieces. The postulated mechanism for the corrosive process was attacked by fused NaK, resulting from reaction with water during cleaning. In-reactor NaK attack was not ruled out, however. A more detailed discussion can be found in Reference 2. The specimens included in the tensile test matrix exhibited the least corrosion, but corrosion cannot be excluded as a possible contributing factor to the postirradiation mechanical properties.

5.2.5 Future Work

Tensile specimens from pins B284 and B285 from AD-1 will be tested next. These pins had irradiation temperatures of 394 and 450°C, respectively, with maximum fluences similar to B286. The corrosion encountered in the specimens from B286 is expected to be minimized. It is highly desirable to isolate the cause of any mechanical property changes to irradiation effects.

5.2.6 References

1. J. O. Stiegler et al., "Alloy Development for Irradiation Performance," *The Fusion Reactor Materials Program Plan*, July 1978.
2. R. J. Puigh and E. K. Opperman, "Examination of Titanium Alloy Specimens Irradiated in EBR-II," *ADIP Quarterly Report*, DOE/ER-0045/1, April 1980.
3. Clifford L. Dotson, "Mechanical and Thermal Properties of High Temperature Titanium Alloys," AFML-TR-67-41, April 1967.
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8. E. K. Opperman, "16 MeV Proton Creep in a Ti-6Al-4V Specimen," *Alloy Development for Irradiation Performance Progress Report*, DOE/ET-0058/1, 1978.
9. R. E. Nygren, "Irradiation Creep Studies of Titanium Alloys," *J. Nucl. Mat.*, 86: 861 (1979).
10. R. H. Jones et al., "Assessment of Titanium Alloys for Fusion Reactor First Wall and Blanket Applications," AP-1433, June 1980.

5.5 INITIAL OBSERVATION OF VOIDS IN NEUTRON IRRADIATED TITANIUM ALLOYS -

D. T. Peterson (Hanford Engineering Development Laboratory)

5.5.1 ADIP Task

Task Number I.C.4 - Microstructures and Swelling in Reactive/Refractory Alloys (Path C).

5.5.2 Objective

The objective of this work is to characterize the swelling and microstructural response of titanium alloys during neutron irradiation.

5.5.3 Summary

A preliminary transmission electron microscopy examination has been performed on Ti-6242S irradiated to 5.0×10^{22} n/cm² ($E > 0.2$ MeV) at 550°C. An inhomogeneous void distribution was observed in the specimen. The voids were observed only in the primary alpha grains and do not constitute a large volume fraction.

5.5.4 Progress and Status

5.5.4.1 Introduction

Very little is known about the effect of irradiation on the microstructures and mechanical properties of titanium alloys. The AD-1 experiment was designed to provide such information on a series of titanium alloys consisting of the near-alpha alloys Ti-6242S and Ti-5621S; the alpha-beta alloy Ti-64; and the beta alloy Ti-38644. These materials were irradiated in EBR-II at 394, 450 and 550°C. The 394°C capsule attained a fluence of 4.2×10^{22} n/cm² ($E > 0.1$ MeV) while the 450 and 550°C capsules attained a fluence of 5.0×10^{22} n/cm² ($E > 0.1$ MeV).⁽¹⁾ At present only the specimens from the 550°C capsule are available.

5.5.4.2 Results

The nominal composition of Ti-6242S is Ti-6%Al-2%Sn-4%Zr-2%Mo-0.09%Si. The specimens were given a duplex anneal consisting of 900°C/0.5 hr/AC + 788°C/0.25 hr/AC + 718°C/4 hr/Ang Cool. The last anneal was done in vacuum. This heat treatment produces a structure consisting predominately of equiaxed primary alpha with some small regions of transformed beta.

The microstructure of Ti-6242S following irradiation at 550°C is illustrated in Figure 5.5.1. Two microstructural features are readily discernible. The first is the presence of voids and the second is the presence of a high density of small defects that appear to be dislocation loops. Voids were observed only in the alpha grains and have an inhomogeneous distribution. Most grains contain only a few voids while a few grains contained a moderate number of voids dispersed throughout the grain. The voids are predominately well developed and are approximately 70 nm in size. The overall volume fraction of the voids is small and does not constitute a very large degree of void swelling.

5.5.4.3 Future Work

The electron microscopy examination will be continued on this material and will be initiated on the other alloys and irradiation conditions. The examination will be expanded to include EDAX analysis to determine if any microchemical segregation has occurred. Immersion density measurements will also be performed on the specimens.

5.5.5 Conclusions

Voids have been observed in Ti-6242S irradiated to 5.0×10^{22} n/cm² at 550°C. Although voids have been observed in ion-bombarded Ti-64⁽²⁾ this is the first observation of voids in neutron irradiated titanium alloys.

5.5.6 References

1. R. J. Puigh and E. K. Opperman, "Examination of Titanium Alloy Specimens Irradiated in EBR-II," ADIP Quarterly Report, October-December, 1979.
2. S. C. Agerwal et al, "Microstructure of Single and Duct-Ion Irradiated Fe-20Ni-15Cr and Ti-6Al-4V Alloys," J. Nuc. Mat., 85 and 86 (1979), 653.

Figure Caption

Fig. 5.5.1: The microstructure of Ti-6242S following irradiation at 550°C.

Figure S.5.1 (a.)

Figure S.5.1(b)

7.6 FABRICATION OF MATERIALS FOR THE RB-1 EXPERIMENT IN HFIR -

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(Hanford Engineering Development Laboratory).

7.6.1 ADIP Task

The Department of Energy (DOE), Office of Fusion Energy (OFE) has cited the need for data on these materials under the ADIP Program Task, Ferritic Material Development (Path E).

7.6.2 Objective

The objective of this work is to provide ferritic materials which are to be used in the fabrication of specimens to be irradiated in the RB-1 and AD-2 experiments.

7.6.3 Summary

Selected ferritic alloys have been mechanically processed for use in the fabrication of specimens for the MFE-RB-1 experiment in HFIR and for the reencapsulation of the AD-2 experiment in EBR-II. Weldments have been fabricated from HT-9, modified 9Cr-1Mo, and 2-1/4Cr-1Mo base metals.

7.6.4 Progress and Status

7.6.4.1 Introduction

The MFE-RB-1 experiment⁽¹⁾ will provide data on the effects of low energy neutron irradiation at 50°C on the mechanical properties of selected ferritic steels. Specifically, this experiment will contain specimens fabricated from the following alloys: HT-9 (2 heats), 9Cr-1Mo (2 heats), 2-1/4Cr-1Mo, AISI 316SS, weldments of HT-9 (one heat), 9Cr-1Mo (1 heat) and 2-1/4Cr-1Mo, and the nickel doped alloys fabricated by ORNL from HT-9 and 9Cr-1Mo. HEDL and ORNL share responsibility for the fabrication of materials and specimens for this experiment. This report will cover the materials for which HEDL has responsibility: HT-9 (one heat), 9Cr-1Mo (one heat), 2-1/4Cr-1Mo, all weldments, and AISI 316SS.

7.6.4.2 Material Fabrication

The vendor certified compositions for the Carpenter Technology heats of HT-9 and 9Cr-1Mo, for the Cameron Iron Works heat of 2-1/4Cr-1Mo, and for the MFE heat of AISI 316SS are given in Table 7.6.1. This heat of HT-9 is the same material as was used in the fabrication of specimens for the AD-2 experiment.⁽²⁾ Also, this particular heat of 9Cr-1Mo is from the same melt as the material used in the AD-2 experiment. Since a sufficient quantity of the heat of 2-1/4Cr-1Mo used in the AD-2 experiment was not available, another heat of 2-1/4Cr-1Mo was procured from ORNL. The major chemical difference between this heat and the heat used in the AD-2 experiment is the nickel content.

Figure 7.6.1 outlines the processing steps in the fabrication of the HT-9 specimen material stock from the 33.3 mm diameter bar stock. A decarburized layer as large as 0.25 mm thick forms on the material during the hot forging and rolling at 1038°C. After cold rolling the material approximately 29%, an anneal at 760°C for 2.5 hours was performed to relieve stresses incurred during cold rolling. This anneal and the following two heat treatments were performed in an argon atmosphere furnace to minimize further decarburization of the surfaces. The final heat treatment for the 6.35 and 3.18 mm plate stock was 1038°C/10 min/AC + 760°C/30 min/AC. The decarburized layer was removed from the 6.35 and 3.18mm material during specimen fabrication.

The mechanical processing steps used in the fabrication of the HT-9 sheet stock from which the fatigue crack growth, the flat tensile, and the Grodzinski Fatigue specimens are to be fabricated are shown in Figure 7.6.2. The starting stock was the 3.18 mm plate fabricated using the process outlined in Figure 7.6.1. The surfaces of this plate were ground to remove the decarburized layer. All subsequent heat treatments of this material were performed by encapsulating the material with a strip of Ta getter foil in a quartz tube which was then filled with a one-fourth atmosphere of argon. The final heat treatment for this thin material stock was 1038°C/4 min/AC + 760°C/30 min/AC. This heat treatment produced a microstructure for this material stock which was equivalent to the primary condition (TMT#1) of HT-9 used in the AD-2 experiment.⁽²⁾

TABLE 7.6.1
CHEMICAL COMPOSITIONS

Element	HT-9 (91353)		9Cr-1Mo (30176)		2-1/4Cr-1Mo (56447)	316SS (15893)	
	Vendor ¹	Overcheck ²	Vendor ³	Overcheck ⁴		Vendor ⁶	Overcheck ⁷
C	0.21	0.23	0.09	0.092	0.10	0.05	0.057
Mn	0.50	0.39	0.37	0.48	0.40	1.79	1.67
P	0.007		0.011	0.012	0.009	0.031	0.033
S	0.003		0.004	0.004	0.006	0.020	0.015
Si	0.22	0.23	0.16	0.15	0.23	0.65	0.66
Ni	0.58	0.46	0.11	0.09	0.14	12.38	11.84
Cr	11.80	12.34	8.43	8.32	2.16	17.33	16.56
Mo	1.02	0.99	0.89	0.86	1.03	2.14	2.44
V	0.32	0.45	0.24	0.20	0.01		
Nb			0.08	0.06			
Ti	0.003		<0.02	0.001	0.01		<.01
Co			0.02	0.017	0.01		
Cu	0.04	0.07	0.04	0.03	0.03		
Al	0.028		0.016	0.011			
B	<0.0010		<0.001	<0.001			
As	<0.01			<0.001			
Sn				0.002			
Zr				<0.001			
N	0.006			0.055			
O				0.010			
Pb				<0.001			
Sb				<0.001			
Ta	<0.01						
W	0.50			<0.01			
Fe	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.	Bal.

All numbers are weight percents.

TABLE 7.6.1 (continued)
CHEMICAL COMPOSITIONS

- ¹ Carpenter Technology Certificate of Composition dated July 22, 1975.
- ² Koon-Hall Test Report dated August 21, 1979.
- ³ Carpenter Technology (ORNL report, "Modified 9Cr-1Mo Steel Development Progress Report for Period Ending September 30, 1979").
- ⁴ Combustion Engineering (ORNL report, "Modified 9Cr-1Mo Steel Development Progress Report for Period Ending September 30, 1979").
- ⁵ Cameron Iron Works, Inc. Certificate of Composition dated July 21, 1975.
- ⁶ MSI Nuclear Material Certification dated November 4, 1975.
- ⁷ National Spectrographic Laboratories, Inc. report dated April 12, 1977.

**HT-9
HEAT NUMBER 91353**

HEDL 8101-203.1

FIGURE 7.6.1 HT-9 Mechanical Processing Steps for 0.25 and 0.125 inch plate stock.

HT-9
HEAT NUMBER 91353

FIGURE 7.6.2 HT-9 Mechanical Processing Steps for 0.030 and 0.024 inch sheet stock.

Figure 7.6.3 outlines the processing steps ORNL used in fabricating the 9Cr-1Mo specimen material stock from the 25 mm thick plate. The final heat treatment for this material was chosen to be 1038°C/30 min/AC + 760°C/30 min/AC. This heat treatment was performed in air with the material wrapped in stainless steel foil in order to minimize surface oxidation and decarburization.

Figure 7.6.4 outlines the steps performed at HEDL for processing the 2-1/4Cr-1Mo material into the desired form. The starting material was 51 mm thick plate from a vacuum arc remelt of a Cameron Iron Works, Inc. ingot, heat number 56447. As in the processing of HT-9, a decarburized layer as large as 0.15 mm thick was found on the material after the hot rolling. To minimize further decarbonization of the surface layers the heat treatments were performed in an argon atmosphere. The final heat treatment for this material was 900°C/30 min/AC + 700°C/1 hr/AC. This heat treatment is equivalent to the heat treatment used in the AD-2 experiment and the breeder program for 2-1/4Cr-1Mo. Again, the decarburized layer was removed during specimen fabrication.

The mechanical processing steps used in the fabrication of the 2-1/4Cr-1Mo sheet stock material for the fatigue crack growth, flat tensile, and Grodzinski fatigue specimens are shown on Figure 7.6.5. The surfaces of the starting 3.18 mm thick stock were ground to remove the decarburized layer. All heat treatments were performed with the material plus a tantalum getter foil encapsulated in a quartz tube filled with a one-fourth atmosphere of argon. The final heat treatment for this material stock was also 900°C/30 min/AC + 700°C/1 hr/AC.

The starting stock for the AISI 316SS material was 7.9 mm thick plate from MFE heat X-15893. This material was sandblasted, wire brushed and pickled, then annealed at 1050°C for one hour in an argon atmosphere. The material was cleaned again and then cold rolled (20% reduction) to a final thickness of 6.35 mm.

Some of the 6.35 mm plate stock from the Carpenter Technology heats of HT-9 and 9Cr-1Mo and the Cameron Iron Works heat of 2-1/4Cr-1Mo were shipped to General Atomic Corporation for the fabrication of welded plate stock from which specimens would be manufactured. The

FIGURE 7.6.3 9Cr-1Mo Mechanical Processing Steps for 0.25, 0.125, and 0.030 inch sheet stock.

2¼Cr - 1Mo
HEAT NUMBER 56447

HEDL 8101-203.4

FIGURE 7.6.4 2-1/4Cr-1Mo Mechanical Processing Steps for 0.25 and 0.125 inch plate stock.

**2¼Cr - 1Mo
HEAT NUMBER 56447**

*HEAT TREAT: 900°C/0.5hr/AC + 700°C/1hr/AC

HEDL 8101-203.5

FIGURE 7.6.5 2-1/4Cr-1Mo Mechanical Processing Steps for 0.030 and 0.024 inch Sheet Stock.

TABLE 7.6.2
CHEMICAL COMPOSITIONS OF WELD WIRES

ELEMENT	HT-9 ¹	9Cr-1Mo ²	2-1/4Cr-1Mo ³
C	0.26	0.086	0.108
Mn	0.50	0.389	0.65
P	0.011	0.011	0.011
S	0.005	0.015	0.018
Si	0.26	0.32	0.32
Ni	0.62	0.046	0.055
Cr	11.6	8.93	2.53
Mo	0.95	0.98	0.92
V	0.29		
Nb	<0.01		
Ti	<0.01		
Co	0.03	0.020	
Cu	0.04		
Al	0.011		
N	0.024		
Ta	<0.01		
W	0.49		
Fe	Bal.	Bal.	Bal.

All numbers are weight percents.

¹ Thyssen Specialty Steels, Inc.

² San Diego Welders Supply, Inc.

³ Arcos Corp., Chromenar 521.

chemical compositions of the weld wire are shown in Table 7.6.2. The welding procedure for the HT-9 and 9Cr-1Mo weldments were equivalent to those previously used for the AD-2 experiment.⁽³⁾ All the weldments were given a post weld temper of 780°C for one hour. Each plate was radiographed to insure the quality of the weld.

7.6.4.3 Metallographic Characterization of the Materials

Micrographs of the HT-9 material stock from which specimens are to be fabricated are shown in Figure 7.6.6. All thickness exhibited the martensitic structure typical of a 12Cr-1Mo steel. An estimate of the prior austenitic grain size for this HT-9 stock is an ASTM 7-8. Microhardness measurements were made on this material and its hardness was found to be 283 ± 5 DPH (500 gm).

Micrographs of the modified 9Cr-1Mo specimen material stock are shown in Figure 7.6.7. Although there was oxidation and some scaling on the surfaces due to the heat treatment, no evidence of any decarburized layer was found on any of the material stock. The estimated prior austenitic grain size for this material is ASTM 9-10 and the hardness is 218 ± 5 DPH (500 gm).

Micrographs of the 2-1/4Cr-1Mo specimen material stock are shown in Figure 7.6.8. An estimate of the prior austenitic grain size for this material stock is an ASTM 8-9. Microhardness for this material was measured to be 187 ± 1 DPH (500 gm).

The results of metallographic examination of the base materials are summarized in Table 7.6.3.

Micrographs of the weldments of HT-9 and 9Cr-1Mo are shown in Figures 7.6.9 and 7.6.10. Microhardness measurements were performed across both welds. For the HT-9 weldment the hardness varied from 226 ± 2 DPH(500 gm) in the base metal, to 227 ± 11 DPH(500 gm) in the heat affected (HAZ), to 262 ± 5 DPH(500 gm) in the weld metal. For the 9Cr-1Mo weldment the hardness varied from 197 ± 3 DPH(500 gm) in the base metal, to 224 ± 8 DPH(500 gm) in the HAZ, to 261 ± 8 DPH(500 gm) in the weld metal.

TABLE 7.6.3

METALLOGRAPHIC CHARACTERIZATION OF FERRITIC ALLOYS

ALLOY	PRIOR HISTORY	MICROHARDNESS 500gm DPH	GRAIN SIZE ASTM
HT-9 [†]	40%CW+1038°C/10m/AC+760°C/30m/AC	283±5	7-8
HT-9 ^{††}	40%CW+1038°C/4m/AC+760°C/30m/AC	283±5	7-8
9Cr-1Mo	1038°C/0.5hr/AC+760°C/0.5hr/AC	218±5	9-10
2-1/4Cr-1Mo	40%CW+900°C/0.5hr/AC+700°C/1hr/AC	187±1	7-9

† Thicknesses of 6.35 and 3.18mm

†† Thicknesses of 0.76 and 0.61mm

803307

C.S. 07

400x

Figure 7.6.6 (a)

Micrographs at 400x of HT-9 Material:
0.25 inch plate stock.

Figure 7.6.6 (b)

Micrographs at 400x of HT-9 Material:
0.030 inch Sheet Stock.

400x

803423

Figure 7.6.7(a)

Micrographs at 400x of 9Cr-1Mo Material:
0.25 inch Plate Stock

Figure 7.6.7(b)

Micrographs at 400x of 9Cr-1Mo Material:
0.030 inch Sheet Stock.

400x
0.25"
8/63

Figure 7.6.8(a)

Micrograph at 400x of 2-1/4Cr-1Mo Material:
0.25 inch Plate Stock.

5-1/2x

8/21

Figure 7.6.9

Macrograph at 5-1/2x of HT-9 Weldment.

1004 030 6000 b-2

FIGURE 7.6.8 Micrograph at 400x of 2-1/4Cr-1Mo Material
(b) 0.030 inch sheet stock.

184503

5/5

Figure 7.6.10

Macrograph at 5-1/2x of 9Cr-1Mo Weldment.

7.6.5 Conclusions and Future Work

The fabrication of three ferritic steels to be used in the MFE-RB-1 experiment has been successfully completed. Weldments of HT-9 and 9Cr-1Mo have been successfully fabricated. Weldments of 2-1/4Cr-1Mo are being fabricated now and should be completed by the first week in February, 1981. These materials have been shipped to manufacturers and ORNL for specimen preparation. A TEM examination of these materials is planned to complete the characterization of these materials.

7.6.6 References

1. J. M. Vitek, R. L. Klueh, M. L. Grossbeck and J. W. Woods, "HFIR-MFE-T1, -T2, and RB1: Experiments to Evaluate the Effects of Low-Temperature Irradiation on Ferritic Steels," *ADIP Quarterly Progress Report, September 30, 1980.*
2. R. J. Puigh and N. F. Panayouto, "Specimen Preparation and Loading for the AD-2 Ferritics Experiment," *ADIP Quarterly Progress Report, June 30, 1980.*
3. T. A. Lechtenburg, R. D. Stevenson, S. N. Rosenwasser, B. E. Thurgood, and L. D. Thompson, "Procurement of National 12Cr Heat and Evaluation of Welding Procedures for Irradiation Specimens," *ADIP Quarterly Progress Report, June 30, 1980.*

7.7 MICROSTRUCTURAL EXAMINATION OF A SERIES OF COMMERCIAL FERRITIC ALLOYS IRRADIATED TO VERY HIGH FLUENCE - D. S. Gelles (Hanford Engineering Development Laboratory)

7.7.1 ADIP Task

The Department of Energy (DOE)/Office of Fusion Energy (OFE) has cited the need to investigate ferritic alloys under the ADIP program task, Ferritic Steels Development (Path E). The tasks involved are akin to task number 1.C.2, Microstructures and Swelling in Austenitic Alloys and task number 1.C.1, Microstructural Stability.

7.7.2 Objective

The objective of this work is to provide guidance on the applicability of ferritic stainless steels for fusion reactor structural materials by examining a series of ferritic alloys (not including the Path E candidate alloy) after irradiation to very high fluence in a fast breeder reactor.

7.7.3 Summary

A series of five commercial ferritic alloys, 2 1/4Cr-1Mo, H-11, EM-12, 416, and 430F, covering the composition range 2.25 to 17% chromium and including a wide range of preirradiation microstructures has been examined by transmission electron microscopy following irradiation in the EBR-II over the temperature range 400-650°C and to a maximum fluence of 17.6×10^{22} n/cm² (E > 0.1 MeV) or 84 dpa. These materials were confined to be low void swelling with maximum swelling of 0.63% measured in EM-12 following irradiation at 400°C to 14.0×10^{22} n/cm². A wide range of precipitation response was found in these materials both as a function of alloy and irradiation temperature. Precipitates observed included M₆C, Mo₂C, Chi Laves, M₂₃C₆, α' and a low temperature phase as yet unidentified. It is predicted, based on these results,

that the major impact of irradiation on the ferritic alloy class will be changes in postirradiation mechanical properties due to precipitation.

7.7.4 Progress and Status

7.7.4.1 Introduction

As part of the National Cladding/Duct Materials Development Program (NCD) for LMFBR development, a series of ferritic alloys were included in the AA-I swelling test. As these materials are no longer being studied by the NCD program, they have been made available for fusion related studies. These materials have been irradiated in the EBR-II reactor at a series of temperatures covering the range 400 to 650°C. Irradiations have continued for over 6 years and the fluence levels achieved are now a maximum of 17.8×10^{22} n/cm² (E > 0.1 MeV) or 85 displacements per atom (dpa). As such, these materials provide an unsurpassed opportunity to examine the effects of fast neutron irradiation on the microstructures of the Path E alloy class.

The ferritic alloy series included in the AA-I swelling test consisted of commercial alloys covering a broad composition range. Although the series does not include HT-9, the candidate path E alloy, alloys similar in composition are included. The alloys chosen for study were 2 1/4Cr-1Mo, an alloy used for steam generator heat exchangers, H-11 a submarine steel, EM-12 a French ferritic stainless steel, 416 a low alloyed 12Cr ferritic stainless steel and 430F a high chromium alloy used for heat exchangers in the petrochemical industry. Alloy compositions may be compared with that for HT-9 in Table 1. The alloy series covers the broad range of chromium additions for commercial ferritic alloys from 2.25 to 17 percent chromium.

The ferritic alloys examined in the present study are among a series of alloys measured for density change after irradiation to a maximum fluence of 17.8×10^{22} n/cm² (E > 0.1 MeV).⁽¹⁾ Density change measurements demonstrated that these ferritic materials are highly resistant to irradiation induced

Table 7.7.1. Compositions (in weight percent) of the ferritic alloys under examination with the HT-9 composition provided for comparison.

<u>Alloy</u>	<u>Cr</u>	<u>Mo</u>	<u>C</u>	<u>Mn</u>	<u>Ni</u>	<u>Si</u>	<u>V</u>	<u>Nb</u>	<u>W</u>
2½Cr-1Mo	2.17	0.93	0.12	0.42	0.16	0.2	-	-	-
H-11	4.97	1.3	0.42	0.37	0.12	0.88	-	-	-
EM-12	9.42	2.0	0.085	1.0	-	0.28	0.27	0.40	-
416	13.5	0.19	0.125	0.45	0.22	0.48	-	0.28	-
430F	17.6	0.04	0.043	0.40	-	0.50	-	-	-
HT-9	12.0	1.03	0.20	0.50	0.47	0.41	0.3	-	0.5

swelling over the temperature range 400 to 650°C. The maximum swelling observed was 0.57 percent in EM-12 following irradiation at 400°C to 14×10^{22} n/cm² (E >0.1 MeV) or 67 dpa. But most of the alloys show a slight decrease in density of between 0.10 and 0.20% over the whole temperature range examined.

7.7.4.2 Experimental Procedure

Materials were received from various commercial sources. Results of chemistry overchecks for these materials are provided in Table 1. The materials were heat treated as prescribed in Table 2 XXXXXX producing the microstructures given in Table 2. Specimens 0.3 cm diameter by 1.3 cm were machined from the heat treated stock and stamped with a four digit alphanumeric identification code which defines both the material and irradiation history. These specimens were included in the NCD AA-I swelling test which consisted of five irradiation vehicles of B-7a capsule design with eight subcapsules in each constructed to run at eight different temperatures. The temperatures were controlled by gas gap regulation of gamma heat loss. In each subcapsule all specimens were immersed in static sodium to ensure uniform temperature.

Irradiations were performed in Row 2 of the Experimental Breeder Reactor II (EBR-II), Idaho Falls, ID. Maximum accumulated fluence was 17.8×10^{22} n/cm² (E >0.1 MeV) and fluences varied as a function of subcapsule position from core midplane.

Specimens selected for transmission electron microscopy as listed in Table 2 were prepared by slicing 0.03 cm thick wafers from rod specimens using a slow speed saw with diamond impregnated ganged blades and then by electro-jet polishing the wafers in a Metalthin electropolishing device. The polishing electrolyte was 5% perchloric acid in 95% butyl alcohol and the polishing conditions 90V., 250 to 300 ma. and a moderate pump speed of 2 to 3. Considerable difficulty was encountered with the lower chromium alloys due to oxide film development. The film was removed by subsequent flash polishing

Table 7.7.2. Preirradiation heat treatments and irradiation conditions of ferritic alloy specimens selected for microstructural examination.

<u>Alloy</u>	<u>Heat Treatment</u>	<u>Microstructure*</u>	<u>Selected Specimens**</u>			
			<u>400/14.0</u>	<u>425/15.8</u>	<u>510/17.2</u>	<u>650/17.6</u>
2½Cr-1Mo	1005°C/60 min/WQ + 840°C/120 min/WQ	α, M, tM		94L7	94K7	94H7
H-11	1005°C/60 min/WQ + 565°C/120 min/WQ	tM		93L7	93K7	
EM-12	1050°C/30 min/AC + 755°C/90 min/AC	α, tM	98M7	98L7	98K7	98H7
416	870°C/cool 13° per hour to 595°C/AC	tM		64L7	64K7	
430F	1065°C/60 min/WQ	α, M		91L7	91K7	

* α = ferrite
M = martensite
tM = tempered martensite

** x/y where x = irradiation temperature, (°C) and y = fluence, (10^{22} n/cm⁻²)
abcd where abcd = alphanumeric specimen identification code

in the same electrolyte. Microscopy was performed on a JEM-200A electron microscope equipped with a side-entry double-tilting goniometer stage and operating at 200 keV. Magnetic effects were overcome by use of the dark-field deflection coils and careful objective stigmator adjustment.

7.7.4.3 Results

7.7.4.3.1 2 1/4Cr-1Mo. The preirradiation heat treatment given to 2 1/4Cr-1Mo was non-standard consisting of 1) normalization, 2) water quench, 3) anneal in the two phase ($\alpha + \gamma$) region and 4) water quench. The structure therefore consisted of overtempered martensite, ferrite and untempered martensite. As a result, postirradiation microstructures were quite varied and large area to area differences were encountered which had the advantage of showing what effect these differences had on in-reactor behavior.

Following irradiation at 425°C (condition 94L7), significant effects of irradiation were apparent. Voids were found in both ferrite and tempered martensite, and were slightly truncated octahedra in shape. Swelling was greatest in ferrite regions which developed swelling of 0.2% consisting of voids of 24 nm mean diameter at a number density of $2.4 \times 10^{14} \text{ cm}^{-3}$. The dislocation structure consisted of a dislocation tangle typical of an irradiated metal undergoing significant dislocation climb. Precipitates examined in ferrite regions were of two types, equiaxed M_6C and rod shaped Mo_2C both precipitates distributed on a fine scale within ferrite grains. The orientation relationship of the M_6C with the matrix was the same as that identified in the 510°C condition.

Following irradiation at 510°C (condition 94K7) 2 1/4Cr-1Mo was found to have developed quite differently. No void swelling was found and the dislocation structure had relaxed to form subcells typical in martensite of a tempered martensite structure and in ferrite as a much coarser cell size. Most notable however, was the extensive precipitation observed. Isolated regions

were found containing Mo_2C but the predominant precipitate was M_6C . In ferrite grains, M_6C formed as a moderate density of globular 50 nm particles with denuded zones at grain boundaries of 500 nm. These particles were apparently oriented coincident with respect to the ferrite so that

$$\begin{array}{ccc} \langle 100 \rangle_{\text{M}_6\text{C}} & \parallel & \langle 100 \rangle_{\alpha} \\ \langle 110 \rangle_{\text{M}_6\text{C}} & \parallel & \langle 110 \rangle_{\alpha} \end{array}$$

However, due to the excessive double diffraction encountered and the non-equiaxed morphology of the particles, this orientation relationship must be considered speculative. In martensite grains, the M_6C formed instead as 200 nm particles associated with subgrain boundaries.

Following irradiation at 650°C (condition 94H7) 2 1/4Cr-1Mo resembled an overtempered structure. Subgrain cell walls were well developed, with a moderate dislocation density within cells. Precipitation consisted of large blocky precipitate particles at low number density and generally associated with subgrain and grain boundaries. The precipitate was identified by electron diffraction as M_6C .

Examples of these 2 1/4Cr-1Mo microstructure are provided for comparison in Figure 1. Figure 1a shows the 2 1/4Cr-1Mo microstructure at low magnification prior to irradiation. A ferrite region can be seen at the lower left, a tempered martensite region at the top and several untempered martensite lathes in the center. Figure 1b shows the 2 1/4Cr-1Mo microstructure in a ferrite grain following irradiation at 425°C to $15.8 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at much higher magnification. Voids can be seen as white faceted features fairly uniformly distributed. Precipitates appear as dark faint equiaxed particles in the case of M_6C and rod shaped particles in the case of Mo_2C . Figure 1c shows the 2 1/4Cr-1Mo microstructure at low magnification following irradiation at 510°C to $17.2 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$). A ferrite grain containing globular M_6C precipitates can be seen in the lower part of the micrograph

and a martensite region is shown at the top. Figure 1d provides an example of 2 1/4Cr-1Mo microstructure again at low magnification following irradiation at 650°C to $17.6 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$). Large blocky M_6C particles and subgrain development can be identified.

7.7.4.3.2 H-11. The preirradiation treatment given to H-11 produced a fully martensitic structure which was then very lightly tempered. As a result, the martensite lathes were well defined with some acicular precipitate present at lathe and grain boundaries. However, within lathes the dislocation structure was very dense and precipitation could not be resolved.

Following irradiation at 425°C (condition 93L7) the structure of H-11 was altered considerably. No evidence of voids swelling was found but extensive precipitation had occurred both within martensite lathes and on martensite lathe boundaries. Precipitate developed both as a globular particles tending to define martensite lathe boundaries and as rod shaped particles at high number density within lathes. The former is identified as M_6C based on analysis of the 510°C structure and the latter is identified as Mo_2C based on its morphology.

Following irradiation of H-11 at 510°C (condition 93K7) the structure was somewhat different. Again no void swelling was observed, but this time the dislocation structure had relaxed into a cell structure more characteristic of tempered martensite. The blocky precipitate was increased in size and positively identified as M_6C based on electron diffraction lattice parameter measurements. The Mo_2C was observed infrequently as small widely separated colonies of rod shaped precipitate. Therefore, the M_6C was determined to be the more stable precipitate phase.

Examples of the H-11 microstructures are provided in Figure 2. The preirradiation H-11 microstructure is shown in Figure 2a and may be compared with that following irradiation at 425°C in Figure 2b and that following irradiation at 510°C in Figure 2c all at the same magnification. Figure 2b provides a comparison of rod shaped Mo_2C precipitate and blocky M_6C . Figure 2c shows the typical subcell development pinned by M_6C particles following irradiation at 510°C. The region at the right of Figure 2c provides an example of the Mo_2C Structure in this condition.

7.7.4.3.3 EM-12. The heat treatment prescribed for EM-12 results in a duplex microstructure of tempered martensite and recovered ferrite. The tempered martensite constitutes a moderately small subgrain size decorated with blocky M_{23}C_6 carbide particles whereas the ferrite is precipitate-free with a low density of dislocations randomly distributed. Carbide precipitation is greatest on martensite/ferrite phase boundaries.

Following irradiation at 400°C (condition 98M7) EM-12 is found to have developed a significant void structure and a dislocation structure typical of irradiation induced climb. Voids were found both in the tempered martensite and in the ferrite, but swelling was significantly greater in the ferrite. Void swelling measurements in the ferrite indicated swelling of 0.63%, consisting of truncated octahedral voids with a mean size of 33 nm at a number density of $2.9 \times 10^{14} \text{ cm}^{-3}$. This is in reasonable agreement with the density change measurement of 0.57% swelling.⁽¹⁾ The dislocation structure in ferrite grains consisted predominantly of straight dislocation segments, similar in structure to that observed following irradiation at 425°C. Also, of note was the observation that precipitation had occurred in ferrite grains during irradiation. The precipitate particles appeared equiaxed, less than 20 nm in diameter and at a fairly high number density. The identity of this precipitate has not yet

been ascertained but it has been demonstrated to be different from those which form in EM-12 at higher temperatures and the chromium rich α' body centered cubic phase may be ruled out based on electron diffraction pattern evidence.

Following irradiation at 425°C (condition 98L7) EM-12 develops a microstructure somewhat different from that produced at 400°C. Void and dislocation structures differed primarily in scale. The maximum void sizes were similar but void swelling was less with measurements in ferrite indicating 0.26% swelling consisting of voids of 24 nm mean size at a number density of $2.4 \times 10^{14} \text{ cm}^{-3}$. This was due to a larger fraction of smaller voids in comparison with the 400°C microstructure. The dislocation structure in ferrite grains consisted predominantly of straight dislocation segments, pure edge in character and with a Burgers vector of $a\langle 100 \rangle$, but $\frac{a}{2}\langle 111 \rangle$ Burgers vectors could also be identified. The major difference observed in comparison with the 400°C condition was as a result of the precipitate structure. Following irradiation at 425°C, precipitation was significantly heavier. In ferrite, it consisted of equiaxed particles as large as 40 nm in diameter. Electron diffraction evidence revealed that this precipitate had a body centered cubic crystal structure with a lattice parameter three times that of the matrix and which formed coincident with the parent lattice. It has been identified therefore as chi phase (Fe-30%Mo-20%Cr) with the orientation relationship

$$\begin{array}{lll} \langle 100 \rangle_{\chi} & \parallel & \langle 100 \rangle_{\alpha} \\ \langle 110 \rangle_{\chi} & \parallel & \langle 110 \rangle_{\alpha} \end{array}$$

In dislocation contrast, the precipitate particles showed strong fringe contrast which indicated that misfit dislocations were present as required to alleviate a lattice misfit between precipitate and matrix.

Following irradiation at 510°C (condition 98K7) the microstructure of EM-12 was again different. In this condition, no void swelling was found, the dislocation structure was not characteristic of that due to irradiation induced climb and precipitation was observed. However, the precipitate was notably different

from that which formed following irradiation at 425°C. Heavy precipitation was observed on all grain and martensite lath boundaries. Adjacent to these boundaries were 250 nm regions denuded of precipitate, and whenever boundaries were sufficiently separated, within grains heavy precipitation of particles on the order of 100 nm were found. The particles were generally globular in shape but many examples of rod shaped precipitates could also be identified. Again, evidence for misfit dislocations was obtained. Electron diffraction results indicated that again the precipitate had a body centered cubic crystal structure with a lattice parameter three times that of the matrix and therefore the precipitate was chi phase. The reason for the morphological difference was due to a different orientation relationship which could be defined as

$$\begin{array}{ccc} \langle 111 \rangle_{\chi} & \parallel & \langle 111 \rangle_{\alpha} \\ \langle 0\bar{1}1 \rangle_{\chi} & \parallel & \langle 1\bar{1}0 \rangle_{\alpha} \end{array}$$

Thus, eight different orientation variants existed for particles of chi phase following irradiation at 510°C.

Following irradiation at 650°C (condition 98H7) EM-12 was found to be much less complicated. The microstructure was similar to that of overtempered martensite and ferrite. Precipitates were large and blocky and generally located at grain and subgrain boundaries. However, again the precipitate phase was found to be different. In this case, several examples of particles containing a large number of internal twins were found. Electron diffraction analysis after allowing for the internal twinning revealed that these precipitates were Laves phase (Fe_2Mo). This result was corroborated by L.E. Thomas of HEDL using energy dispersive analysis of X-rays on a JEOL 100 CX scanning transmission electron microscope. The precipitates were found to be rich in iron and molybdenum with lesser amounts of chromium, vanadium and silicon.

Examples of the EM-12 microstructure are given in Figures 3 and 4. Figure 3 provides comparisons of EM-12 microstructures at low magnification following

irradiation at 400°C in (a), 425°C in (b), 510°C in (c) and 650°C in (d).

Voids can be identified both in ferrite and tempered martensite following irradiation at 400 in Figure 3a and at 425°C in Figure 3b. However, significantly more precipitation is observed in the ferrite grain following irradiation at 425°C. In Figure 3c, even more precipitation is shown, with continuous layers decorating grain boundaries and heavy precipitation decorating subgrain boundaries. Figure 3d provides an example of the blocky precipitate morphology decorating grain and subgrain boundaries following irradiation at 650°C. Figure 4 is constructed to emphasize the differences in precipitate development in ferrite grains following irradiation at 400, 425 and 510°C. The microstructure for each condition is shown and (110) diffraction patterns to demonstrate differences in crystallography are inset in each case. In Figure 4a, small weakly contrasted particles can be seen in the background whereas in Figure 4b the particles are larger and in better contrast. Figure 4c provides examples of the more globular shaped particles formed during irradiation at 510°C but under these imaging conditions, misfit dislocation arrays at particles can be identified; the best examples appear at the left. In order to emphasize that these precipitates are also chi phase, the diffraction pattern inset in Figure 4c has been marked to show the chi phase (110) spot array which is rotated 120° from that observed in the diffraction pattern of Figure 4b.

7.7.4.3.4 416. The heat treatment given to 416 prior to irradiation produced a microstructure which might be described as tempered martensite. The structure appeared to have a subgrain or martensite lath structure with a very high dislocation density within subgrains and a low density of randomly distributed carbide particles. However, the heat treatment defined for 416 in Table 2 should have produced an annealed microstructure resulting from slow cooling through the $\gamma \rightarrow \alpha$ transformation. Therefore, it must be presumed that

the prescribed heat treatment was not followed. A more likely preirradiation treatment can be surmised as follows 1) warm worked 2) cold worked and 3) stress relieved at 360°C.

Following irradiation at 425°C (condition 64L7) 416 was found to be highly resistant to radiation induced void swelling. One small void was identified in the material examined. The dislocation structure had recovered from the pre-irradiation condition to form dislocation arrays characteristic of subgrain boundaries but dislocation tangles typical of radiation induced dislocation climb were not observed. The major effect due to the irradiation was that of precipitation. The precipitation was similar to that observed in EM-12 following irradiation at 400°C but electron diffraction evidence indicated that both the low temperature EM-12 precipitate and chi phase were forming.

Following irradiation at 510°C, the major change in the microstructure of 416 was that due to formation of a subgrain structure. Unlike the other alloys examined, it was not possible to identify precipitates which had formed during irradiation; large blocky $M_{23}C_6$ precipitates were present but they had also been observed in the structure prior to irradiation. Thus, 416 following irradiation at 510°C represents the most stable alloy microstructure encountered.

Examples of the 416 microstructures are given in Figure 5. Figures 5a and b provide comparison of the microstructure of 416 following irradiation at 425°C at low and high magnification respectively. In both cases the dislocation structure can be identified as straight parallel lines. The precipitates appear as poorly contrasted equiaxed particles. Figures 5c and d provide comparison of the microstructure generated in 416 following irradiation at 510°C again at low and high magnification respectively. Subgrain development can be seen to be more clearly defined and precipitation is observed to be much less extensive. The imaging conditions used for Figure 5d showing the dislocation structure would normally reveal effects of fine precipitation. However, none can be seen.

7.7.4.3.5 430F. The preirradiation heat treatment given to 430F produced a duplex microstructure of ferrite and martensite. As a tempering treatment was not used, the ferrite was found to be deformed following martensite transformation and the dislocation structure within the martensite plates was very dense.

Irradiation at 425°C (condition 91L7) produced a very different microstructure in 430F. No void swelling was observed and regions of ferrite were distinguishable from those of martensite. The major impact of irradiation was due to precipitation. Boundaries separating ferrite from martensite were thickly coated and boundaries between ferrite grains showed enhanced precipitation on the boundary with adjacent denuded zones. Everywhere else heavy precipitation was observed consisting of globular and equiaxed particles. The precipitate is identified as α' , a chromium rich body centered cubic phase, based on analysis of ring electron diffraction patterns. The rings are believed to have resulted from an electropolishing artifact; the polish selectively attached the matrix material, lifting the particles free and creating coating of particles on foil surfaces.

In comparison, irradiation at 510°C (condition 91K7) produced a microstructure in 430F with much less precipitation. No void swelling was observed and the dislocation structure in martensite had relaxed to form subgrains with blocky $M_{23}C_6$ particles decorating boundaries. In ferrite grains, α' particles on the order of 40 nm in diameter were observed at low member density. That these particles were α' was deduced by the fact that they could only be imaged in dark field using conditions usually employed for weak beam dislocation imaging.

Examples of 430F microstructures are given in Figure 6. Figure 6a and b provide comparison of 430F following irradiation at 425°C at low and high magnification respectively. In Figure 6a the heavy precipitation coating grain boundaries and developing within ferrite grains can be seen. A martensite region is shown in the upper part of the micrograph. Figure 6b provides an example of α' precipitation at a ferrite/ferrite grain boundary with the adjacent denuded zones. This structure can be compared with that following irradiation

at 510°C as shown in Figures 6c and d. The more extensive subgrain development in martensite is shown at low magnification in the upper part of Figure 6c while the globular α' distribution is visible at higher magnification in Figure 6d.

7.7.4.4 Discussion

This study evolved as a result of an opportunity to take advantage of specimens irradiated to very high fluences in the NCD LMFBF program but no longer needed by that program. The alloys available have provided a complete survey of the ferritic alloy classes which may be employed for intermediate to high temperature use in fusion first wall applications. However, the prime candidate path E alloy, HT-9, was not included in the materials available and therefore the present study was undertaken only as a brief survey. It is now apparent that the microstructures of these ferritic alloys respond in very complex and different ways. It is the intent of this discussion section therefore to consider the consequences of such behavior for fusion applications.

In order to emphasize the wide range of microstructural development observed in the ferritic alloys which were examined, Table 3 has been prepared to summarize the microstructural observations made as a function of alloy and irradiation temperature. (To identify situations where microstructural features were observed at low number density, features have been parenthesized.) Review of Table 3 rapidly reveals that 1) void swelling is not observed at temperatures of 510°C and above and that swelling at temperatures below 510°C is not a straightforward function of composition or precipitation behavior, 2) precipitation is very complex both as a function of alloy composition and irradiation temperature and 3) the variations in precipitation response are not simply due to variations in the form of the carbide phases which appear.

The present microstructural results confirm that although commercial ferritic alloys can develop void swelling, the swelling is probably not going

Table 7.7.3. Summary of microstructural results obtained in the present study.

<u>Alloy</u>	<u>400°</u>	<u>425°C</u>	<u>510°</u>	<u>650°</u>
2½Cr-1Mo		Voids M ₆ C, Mo ₂ C	M ₆ C	(M ₆ C)
H-11		Mo ₂ C, M ₆ C	Mo ₂ C, M ₆ C	
EM-12	Voids ppts	Voids χ	χ*	(Laves)
416		(Voids) (ppts, M ₂₃ C ₆)	(M ₂₃ C ₆)	
430F		α'	(α', M ₂₃ C ₆)	
		$\langle 111 \rangle_\alpha \parallel \langle 111 \rangle_{\chi^*}$		
		$\langle 1\bar{1}0 \rangle_\alpha \parallel \langle 0\bar{1}1 \rangle_{\chi^*}$		

to represent a design limitation. The maximum swelling observed was less than one percent following irradiation to 67 dpa. The maximum swelling observed was found at the lowest test temperature examined and therefore the possibility exists that higher swelling would develop at still lower temperatures. However, the dislocation and void densities observed in that condition are typical of a material at peak swelling condition rather than above the peak swelling condition and therefore it may be predicted that if higher swelling occurs at the still lower temperature, the increase will not be very large.

To speculate on the reason why ferritic alloys swell at a lower rate than similar austenitic alloys is perhaps opportune. Several studies have found that irradiation of ferritic alloys produces dislocations of Burgers vector $a\langle 100 \rangle$.⁽²⁻⁴⁾ Austenitics instead develop Burgers vectors of $\frac{a}{3}\langle 111 \rangle$ or $\frac{a}{2}\langle 110 \rangle$. However, in most cases the austenitic dislocation structure evolves to one dominated by glissile $\frac{a}{2}\langle 110 \rangle$ dislocations. The glissile Burgers vector for ferritics is $\frac{a}{2}\langle 111 \rangle$ and the key difference between these materials, it may be hypothesized, is the ability of the irradiation induced sessile dislocations to evolve into a glissile dislocation network with few obstacles. In the case of ferritics, no simple dislocation reaction between $a\langle 100 \rangle$ dislocations or $a\langle 100 \rangle$ and $\frac{a}{2}\langle 111 \rangle$ will lead to a glissile dislocation structure. Instead, the $a\langle 100 \rangle$ dislocations will remain as obstacles to dislocation motion thereby reducing dislocation climb. Therefore, the overall swelling rate will be lower.

Previous studies of irradiation effects on ferritic alloys have not emphasized observations of precipitation as has the present study. It is therefore worth noting that such response is predicted by thermal equilibrium phase diagrams and does not necessarily represent an effect of irradiation to high fluence. A wide range of carbide phases have been identified in Fe-Cr-Mo alloys with 0.1 to 0.2% carbon.⁽⁵⁾ The published binary iron-chromium phase diagram⁽⁶⁾ includes a miscibility gap below 520°C wherein the binary mixture decomposes into iron rich and chromium rich (α') regions below 520°C. Therefore, observations of α'

in 430F at 425°C and 510°C are predicted by thermal equilibrium. The iron-chromium-molybdenum ternary phase diagram⁽⁷⁾ is generally constructed to show the body centered cubic chi phase in the composition range Fe-20%Cr-30%Mo and therefore observations of chi phase in EM-12 and 416 should not be unexpected. The iron-chromium-molybdenum ternary diagram ~~XXXX~~ shows the Laves phase Fe_2Mo to form as well. Observation of this phase is perhaps unexpected in an alloy with 10% chromium such as EM-12 but alloying additions such as carbon may be encouraging the Laves development. To emphasize these points, a section of the iron-chromium-molybdenum diagram for 650°C compiled by T. Wada⁽⁷⁾ is reproduced in Figure 7 and the compositions of the five alloys in the present study are shown for comparison. From Figure 7, it can be noted that all alloy compositions lie in the single phase field but the $(\alpha + \chi)$ regions is very close to the EM-12 composition and it is not difficult to envision that a lower temperature coupled with further alloying additions would cause the $(\alpha + \chi)$ field to expand.

Observation of extensive precipitation in commercial ferritic alloys can be expected to have bearing on the fusion applicability of these alloys. Precipitation should directly affect postirradiation strength, ductility and toughness. Smidt et al have recently shown that the fracture toughness of HT-9 is degraded significantly as a result of irradiation at 420°C to $1.1 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$).⁽⁸⁾ The ductile brittle transition temperature was found to shift 103°C due to irradiation. Furthermore, a fine scale precipitation was observed by transmission electron microscopy and therefore one of the explanations given for the degradation was the presence of the precipitate. (It can be noted that the phase found in EM-12 following irradiation at 400°C can be expected to be related to that described by Smidt et. al.). Furthermore, microstructures such as that shown in Figure 3c can be expected to have very different mechanical properties than would the preirradiation microstructure. Fortunately, the ADIP path E reactor experiments have emphasized the need for such data and the experiments have been designed accordingly.

Perhaps, the consequence that must be most emphasized regarding postirradiation mechanical properties in path E alloys is the impact of irradiation temperature on microstructure. Changes in irradiation temperature were found to produce major differences in precipitate microstructure. Mechanical properties may therefore change markedly with small changes in irradiation temperatures. Therefore, postirradiation mechanical testing matrices must be prepared to test a wide range of irradiation temperature conditions in order to ensure that a particular precipitation response does not create such adverse properties so as to make a given material unusable.

However, the wide range of precipitation response observed as a function of alloy composition suggests that if a phase is found to develop which is particularly detrimental to mechanical properties, it appears possible to eliminate that phase by minor changes in alloy composition.

7.7.5 Conclusions

The commercial alloys 2½Cr-1Mo, H-11, EM-12, 416 and 430F are found to be highly resistant to void swelling but prone to a wide variety of precipitation behavior following irradiation over the temperature range 400-650°C to a maximum fluence of $17.6 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$). Precipitates found included M_6C , Mo_2C , Chi, Laves $M_{23}C_6$, α' and a low temperature phase as yet unidentified. Gross differences in precipitate development were found not only as a function of alloy composition but also as a function of irradiation temperature for a given alloy. For example, EM-12 was found to develop at least four distinctly different temperature regimes of precipitation behavior.

7.7.6 Future Work

Specimens of these commercial ferritic alloys will be examined following irradiation at 425°C to the lower fluence of $6 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) in order to predict whether changes in mechanical properties due to precipitation will saturate at moderate fluences.

7.7.7 References

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Fig. 1: Examples of microstructures observed in 24Cr-1Mo.

Fig. 7.2: Examples of microstructure observed in H-11.

Fig. 7 - 3: Examples of microstructure observed in EM-12.

Fig. 4: Comparison of precipitation development in EM-12 following irradiation with (110) diffraction patterns inset.

Fig. . / 5: Examples of microstructure observed following irradiation in 416.

Fig. 6: Examples of microstructures observed following irradiation in 430F.

**Fe-Cr-Mo DIAGRAM
AT 650°C**

HEDL 8011-201.10

Fig. 7: A portion of the iron-chromium-molybdenum thermal equilibrium phase diagram showing the compositions of commercial ferritic alloys of interest.

7.8 MICROSTRUCTURAL EXAMINATION OF HT-9 ARCHIVE MATERIAL FROM THE AD-2 TEST - D. T. Peterson

7.8.1 ADIP Task

ADIP Program Task, Ferritic Alloy Development (Path E).

7.8.2 Objective

The objective of this work is to evaluate the microstructures of the HT-9 mechanical property specimens in the AD-2 test.

7.8.3 Summary

The HT-9 fracture toughness and miniature Charpy specimens included in the AD-2 test received a different set of thermomechanical treatments (TMTs) than the other mechanical property specimens.⁽¹⁾ Transmission electron microscopy has been performed on material stock representing the four treatments involved. All four treatments produced essentially identical tempered martensite structures.

7.8.4 Progress and Status

7.8.4.1 Introduction

The objective of the AD-2 test is to determine the effect of irradiation on the mechanical properties of the ferritic alloys HT-9, 9Cr-1Mo and 2 $\frac{1}{2}$ Cr-1Mo. To assess the influence of prior austenite grain size on its mechanical properties two normalization and temper treatments were prescribed for the HT-9 specimens. The first treatment was intended to produce a fine prior austenite grain size while the second treatment was intended to produce a coarse grain size. All of the specimens received such a set of treatments. However, the two TMTs used for the fracture toughness and miniature Charpy specimens were different than those used for the tensile, crack growth and Grodzinski fatigue specimens.

TEM examinations were performed to determine if there were any significant differences in the microstructures produced by these treatments.

7.8.4.2 Experimental Procedure

Material for TEM specimens was obtained from fracture toughness specimens and sheet stock from the AD-2 archives. For the fracture toughness specimens a 0.25 to 0.38 mm thick slice was cut transverse to the face of the specimen. Disks were then punched out of the slice. The sheet stock was ground to a thickness of 0.25 to 0.38 mm after which disks were punched from the sheet.

The disks were prepared for TEM examination with a Metalthin twin-jet electropolishing unit using a solution of 5% perchloric acid in 95% butyl alcohol. The polishing was done using a moderate pump speed and a voltage of about 90V, with a current of 150-200 mA.

7.8.4.3 Results and Discussion

The material that was examined is listed in Table 7.8.1. Optical metallography and microhardness results have been reported on these treatments by Puigh and Panayotou.⁽¹⁾

Table 7.8.1. Thermomechanical Treatments of the Material Examined.

<u>Material</u>	<u>TMT</u>
0.61 mm Sheet	1038°C/4 min/AC + 760°C/0.5 hr/AC
0.76 mm Sheet	1038°C/0.5 hr/AC + 760°C/2.5 hr/AC
Fracture Toughness Specimen T408	Mill Anneal: 1149°C/1 hr minimum followed by hot working and tempering at 750°C/1 hr/AC
Fracture Toughness Specimen T545	Mill Anneal + 1050°C/1 hr/AC + 780°C/2.5 hr/AC

The greatest concern was centered around the mill annealed material. This treatment is not intended to produce a martensitic structure. However, optical metallography revealed that the structure was martensitic.⁽¹⁾ This finding was

supported by the TEM examination. The microstructure of the mill annealed condition is compared with the other three conditions in Figure 7.8.1. All four treatments produced virtually identical tempered martensite structures.

7.8.5 Conclusion

The fracture toughness and miniature Charpy specimens included in the AD-2 test have been found to have the same tempered martensite microstructure as the other mechanical properties specimens. Nonetheless, due to the differences in microhardness and grain size reported previously,⁽¹⁾ comparisons between the two sets of data must be made with care.

7.8.6 References

1. R. J. Puigh and N. F. Panayotou, "Specimen Preparation and Loading for the AD-2 Ferritics Experiment," ADIP Quarterly Progress Report, April-June, 1980.

FIGURE CAPTION

Fig. 7.8.1: The microstructures of the four treatments examined.
(a) $1038^{\circ}\text{C}/4 \text{ min/AC} + 760^{\circ}\text{C}/0.5 \text{ hr/AC}$, (b) $1038^{\circ}\text{C}/0.5 \text{ hr/AC} + 760^{\circ}\text{C}/2.5 \text{ hr/AC}$,
(c) Mill Anneal, (d) Mill Anneal + $1050^{\circ}\text{C}/1 \text{ hr/AC} + 780^{\circ}\text{C}/2.5 \text{ hr/AC}$.

Figure 7.5.1 (a)

Figure 7.51 (6)

Figure 7.21 (c)

7.10 IMMERSION DENSITY ANALYSIS OF COMMERCIAL ALLOYS IRRADIATED TO A VERY HIGH FLUENCE - M. K. Zimmerschied, D. T. Peterson, R. W. Powell, and J. F. Bates (Hanford Engineering Development Laboratory).

7.10.1 ADIP Task

ADIP Task I.C.1, Microstructural Stability.

7.10.2 Objectives

The objective of this work is to provide high-fluence swelling data on commercial alloys of relevance to the ADIP program.

7.10.3 Summary

Immersion density measurements have been performed on a series of high-nickel, refractory and ferritic commercial alloys irradiated in EBR-II to a peak fluence of 16.8×10^{22} n/cm² (E > 0.1 MeV). The specimens were irradiated at temperatures ranging from 400 to 650°C. All of the alloys exhibit swelling resistance far superior to AISI 316. None of the ferritic alloys exhibited more than 0.6% swelling after this high exposure.

The high-nickel superalloys, Inconel X-750 and Inconel 718, both show a low temperature swelling peak. Inconel X-750 swells more than Inconel 718 at all temperatures and has a peak of 2.4% at 427°C.

The refractory alloys, TZM and Nb-1Zr, both show peak swelling at temperatures at or above 593°C. TZM exhibits the greatest swelling with a peak of 3.6% at 593°C.

7.10.4 Progress and Status

7.10.4.1 Introduction

The alloys being studied were irradiated in the EBR-II reactor as part of the AA-I test of the National Cladding/Duct Materials Development Program. This test, which began irradiation six years ago, consists solely of commercial alloys. During its most recent discharge a number of alloys of interest to the ADIP program were examined. The alloys include the high-nickel alloys Inconel X-750 and Inconel 718; the refractory alloys TZM and Nb-1Zr; and the ferritic alloys AISI 430F, AISI 416, EM-12, H-11 and 2½Cr-1Mo.

7.10.4.2 Experimental Procedure

Initial material stock was procured from several vendors. The compositions of the alloys are listed in Table 7.10.1. Specimens were machined in the form of 0.3 cm diameter by 1.3 cm long cylinders. The thermo-mechanical treatments of the specimens are listed in Table 7.10.2.

The specimens were irradiated in capsules B-116 and B-117 of the AA-I test. The capsules were of the B-7a design and contained eight subcapsules. Through gas-gap temperature regulation the subcapsules were designed to run at eight different temperatures. The specimens within the subcapsules were sodium bonded to ensure uniform temperatures. The capsules were irradiated in Row 2 of EBR-II as part of the X-223 subassembly. The peak accumulated fluence of the specimens is 16.8×10^{22} n/cm² (E > 0.1 MeV).

Densities were determined by the immersion density technique. Multiple measurements were made on each specimen with a maximum deviation of $\pm 0.05\%$ being accepted. The swelling was calculated by comparing the densities of unirradiated control specimens with those of the irradiated specimens.

7.10.4.3 Results and Discussion

The swelling results are summarized in Table 7.10.2 and Figures 7.10.1 through 7.10.3. For discussion the results will be grouped by alloy class.

7.10.4.3.1 Path B. No swelling data has yet been generated on the five ADIP path B alloys. However, it is possible to infer their swelling behavior by examining similar commercial alloys. The two superalloys studied in this work were Inconel X-750 and Inconel 718. Inconel X-750 is a γ' -strengthened nickel base alloy very similar to the path B alloy designated B6. Inconel 718 is a γ'/γ'' -strengthened nickel base alloy related to alloy B4.

As can be seen in Figure 7.10.1, both alloys exhibit a low temperature swelling peak. Inconel 718 displays a swelling peak of only 0.35% at 427°C while Inconel X-750 has a peak of 2.38%, also at 427°C. Both alloys exhibit excellent swelling resistance after irradiation to a peak fluence corresponding to about 90 dpa.

Void swelling results have been reported on Inconel 706 irradiated in this same test to a peak fluence of 14.7×10^{22} n/cm² (E > 0.1 MeV).⁽¹⁾

Table 7.10.1 Alloy Compositions, as Supplied by the Vendor (Weight Percent).

Alloy	Code	I.D.	Heat	Mfg.	Fe	Ni	Cr	Mo	Nb	Ti	Al	Si	Mn	C	Other
2 $\frac{1}{4}$ Cr-1Mo	94	941A-3	C4337-14S	Lukens	Bal	0.16	2.17	0.93	-	0.02	-	0.21	0.42	0.12	V 0.004
EM-12	98	981A-3	P-12616	Combustion Engineering	Bal	-	9.42	2.02	0.40	-	-	0.28	1.00	0.085	V 0.27
H-11	93	931A-3	D-12099K-11	Universal Cyclops	Bal	0.12	4.97	1.31	-	-	-	0.88	0.37	0.42	V 0.49
AISI 416	64	641B-2	44221	Jones & Laughlin	Bal	0.27	13.36	0.26	-	-	-	0.48	0.42	0.11	
AISI 430F	91	911A-3	G-8821	Universal Cyclops	Bal	-	17.64	0.04	-	-	-	0.50	0.40	0.043	
InX-750	9A	A691A-3	L-2646K14	Universal Cyclops	7.81	71.52	15.36	-	+Ta 1.06	2.47	0.68	0.07	0.09	0.045	
In718	76	A761A-3	44F9EY	Huntington Alloys of INCO	18.52	52.83	18.57	2.83	+Ta 5.28	0.92	0.50	0.23	0.10	0.04	
Nb-1Zr	82	82 A-2	530126	Teledyne Wah Chang	<0.005	<0.002	-	0.002	Bal	<0.004	-	<0.005	<0.002	0.003	Zr 0.96 Ta 0.095
TZM	83	831A-2	8967	Climax Molybdenum	0.003	0.001	-	Bal	-	0.45	-	0.002	-	0.013	Zr 0.09

Table 7.10.2 Thermomechanical Treatment and Swelling

Alloy	Code	Thermomechanical Treatment	T_{irr} $\phi t, \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$)	400°C	427°C	454°C	482°C	510°C	538°C	593°C	649°C
				11.4	13.9	11.1	13.8	16.0	15.6	16.8	16.6
$2\frac{1}{2}\text{Cr-1Mo}$	94	1010°C/1 hr/WQ + 843°C/2 hr/WQ		+ 0.12	+ 0.08	+ 0.09	+ 0.10	+ 0.09	+ 0.17		+ 0.12
EM-12	98	1052°C/0.5 hr/AC + 754°C/1.5 hr/AC		+ 0.57	+ 0.32	+ 0.01	- 0.13	- 0.21	- 0.13		+ 0.01
H-11	93	1010°C/1 hr/WQ + 565°C/2 hr/WQ		+ 0.14	+ 0.08	+ 0.09	- 0.02	- 0.03			+ 0.16
AISI 416	64	870°C/FC at 13°C per hr to 593°C/AC		+ 0.25	+ 0.11	+ 0.14	+ 0.08	+ 0.06	+ 0.10		+ 0.10
AISI 430F	91	1066°C/1 hr/WQ		+ 0.19	+ 0.14	+ 0.13	+ 0.17	+ 0.11	+ 0.15		+ 0.13
InX-750	9A	1149°C/2 hr/AC + 843°C/24 hr/AC + 704°C/2 hr/AC		+ 1.09	+ 2.38	+ 1.85	+ 1.15	+ 0.88	+ 0.43	+ 0.02	- 0.03
In718	76	954°C/1 hr/WQ + 718°C/8 hr/FC to 621°C/hold for 18 hrs total/AC		+ 0.28	+ 0.35	+ 0.20	+ 0.18	+ 0.05	+ 0.07	+ 0.05	+ 0.02
Nb-1Zr	82	1204°C/1 hr/vacuum cool		+ 0.34	+ 0.38	+ 0.24	+ 0.18	+ 0.21	+ 0.31	+ 0.32	+ 0.71
TZM	83	1316°C/2.5 hr/vacuum cool		+ 0.14	+ 0.05	+ 0.10	+ 0.12	+ 0.35	+ 0.84	+ 3.57	+ 3.34

SWELLING IN COMMERCIAL ALLOYS: AUSTENITIC ALLOYS (PATH B)

Figure 7.10.1. Swelling in the superalloys Inconel X-750 and Inconel 718.

Inconel 706 is a γ'/γ'' -strengthened iron-nickel base alloy very similar to alloy B4. After irradiation to a peak fluence of $14.7 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) the maximum void swelling observed was 0.1%. Based on these results it is anticipated that the path B alloys B4 and B6 will also be highly swelling resistant.

7.10.4.3.2 Path C. The refractory alloys Nb-1Zr and TZM were previously studied in detail following irradiation to a peak fluence of $14.7 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$).⁽²⁾ The present data (see Figure 7.10.2) reveals that for the case of Nb-1Zr extremely low swelling is maintained with a peak of 0.71% at 650°C. Reference to the lower fluence data indicates

SWELLING IN COMMERCIAL ALLOYS: REFRACTORY ALLOYS (PATH C)

Figure 7.10.2. Swelling in the refractory alloys TzM and Nb-1Zr. The peak fluences are indicated in the legend.

that the swelling has increased slightly, with a rate of $0.04\%/10^{22} \text{ n/cm}^2$ at 650°C . This rate is approximately two orders of magnitude lower than the rate of $2.6\%/10^{22} \text{ n/cm}^2$ observed in 20% cold worked AISI 316.⁽³⁾

In TZM a sharp increase in swelling is observed above 538°C with a peak of 3.6% at 593°C (see Figure 7.10.2). Comparison with the lower fluence data reveals that the swelling has increased at all temperatures above 482°C . At the peak temperature of 593°C the swelling has increased at a rate of $0.27\%/10^{22} \text{ n/cm}^2$. This is only slightly lower than the rate of $0.32\%/10^{22} \text{ n/cm}^2$ observed at the lower fluence.

After irradiation to $14.6 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at 650°C , Gelles and Bates⁽²⁾ observed void lattice development in TZM. They postulated that if swelling saturation were to occur with continued irradiation the voids in the array must grow to a critical size at which point a stable microstructure could be attained. It is interesting to estimate at what fluence this saturation would take place. Gelles and Bates observed a void density of $1.3 \times 10^{17} \text{ cm}^{-3}$ and a maximum void size of 10 nm at 650°C .⁽²⁾ As Sprague et al observed apparent void coalescence in TZM irradiated at 650°C ⁽⁴⁾, 8 nm may be a more accurate estimate of the maximum void size. Using a void density of $1.3 \times 10^{17} \text{ cm}^{-3}$, then 10% swelling will be produced if all the voids grow to 8 nm. At the presently observed swelling rate of $0.19\%/10^{22} \text{ n/cm}^2$, a fluence of $52 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$), or about 190 dpa, would be required to achieve saturation. Therefore saturation would not occur until fluences well in excess of the goal exposures for fusion reactor first walls.

7.10.4.3.3 Path E. Only in the past two years has the fusion program begun to examine ferritic alloys. The three ferritic alloys being studied in the ADIP program are HT-9, 9Cr-1Mo and, to a lesser extent, $2\frac{1}{4}\text{Cr-1Mo}$. These alloys are being irradiated in EBR-II in the AD-2 test. They will not be available at their peak fluence of $6.3 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) until September 1982. This study provides the opportunity to assess the swelling resistance of several ferritic alloys irradiated to a peak fluence (80 dpa) on the order of the anticipated first wall goal exposures.