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**EVALUATION OF FERRITIC ALLOY Fe-2 1/4Cr-1Mo
AFTER NEUTRON IRRADIATION - MICROSTRUCTURE DEVELOPMENT**

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EVALUATION OF FERRITIC ALLOY Fe-2-1/4Cr-1Mo AFTER NEUTRON IRRADIATION - MICROSTRUCTURAL DEVELOPMENT

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1.0 SUMMARY

Microstructural examinations are reported for nine specimen conditions of 2-1/4Cr-1Mo steel which had been irradiated by fast neutrons over the temperature range 390 to 510°C. Two heats of material were involved, each with a different preirradiation heat treatment, one irradiated to a peak fluence of 5.1×10^{22} n/cm² (E > 0.1 MeV) or 24 dpa and the other to 2.4×10^{23} n/cm² (E > 0.1 MeV) or 116 dpa. Void swelling is found following irradiation at 400°C in both conditions and to 480°C in the higher fluence conditions. Concurrently dislocation structure and precipitation formed. Peak void swelling, void density, dislocation density and precipitate number density developed at the lowest temperature, ~400°C, whereas mean void size, and mean precipitate size increased with increasing irradiation temperature. The examination results are used to provide interpretation of in-reactor creep, density change and post irradiation tensile behavior.

Fe-2-1/4Cr-1Mo steel has not been seriously considered for use in either fusion or fission components because it has limited creep strength at temperatures above about 500°C. However, recent fusion reactor studies such as STARFIRE and MARS indicate that acceptable electrical power generation can be achieved with first walls operating at temperatures $\leq 500^\circ\text{C}$. For temperatures $\leq 500^\circ\text{C}$, the 2-1/4Cr-1Mo steel in the normalized and tempered condition becomes a viable structural material candidate. The 2-1/4Cr-1Mo steel appears to offer advantages over the other ferritic steels based on fabricability and at temperatures $\leq 500^\circ\text{C}$, has equivalent properties. However, little is known about its radiation resistance. The present effort is part of a series of experiments intended to provide an irradiation effects data base for 2-1/4Cr-1Mo steel.

Previous experiments in this series have provided irradiation creep,⁽¹⁾ irradiation induced density change⁽¹⁾ and postirradiation tensile properties⁽²⁾ for 2-1/4Cr-1Mo. Pressurized tube specimens in a normalized and tempered bainitic condition were measured for diameter change following irradiation at 390, 480 and 570°C to a $5.5 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) or 26 dpa.⁽¹⁾ It was found that the creep rate was greatest at 570°C but the creep rate at 390°C was slightly higher than at 480°C. Based on diameter change measurements on unstressed pressurized tubes, void swelling of about 0.3% was predicted and creep at 390°C was in part attributed to swelling enhanced creep.

Density change measurements were made on slug specimens in an unusual heat treatment condition following irradiation at 400, 425, 450, 480, 510 and 540°C to fluences between $1.55 \times 10^{23} \text{ n/cm}^2$ and $2.41 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) or 74 to 116 dpa.⁽¹⁾ The heat treatment condition was complex and was believed to have contained tempered proeutectoid ferrite, overtempered bainite, proeutectoid ferrite and untempered bainite. This condition was found to be highly resistant to irradiation induced swelling. The peak density change, 0.28% swelling, occurred following irradiation at

400°C to 1.6×10^{23} n/cm² or 77 dpa. This peak was attributed to void swelling whereas a lesser peak of 0.22% swelling at 540°C was attributed to precipitation causes.

Miniature sheet tensile specimens in a normalized and tempered bainitic condition were irradiated at 390, 450, 500 and 550°C to about 1.9×10^{22} n/cm² ($E > 0.1$ MeV) or 9 dpa. Following irradiation, tensile tests were conducted at the irradiation temperature and in a few cases at room temperature.⁽²⁾ Thermal control specimens aged for 5000 hours to duplicate the irradiation condition were also tested. The results showed that thermal aging reduced strength moderately for all testing conditions but that reactor irradiation increased yield strength by 25% at 390°C and decreased yield strength by 40% at 550°C for tests run at the irradiation temperature. These results were interpreted as follows. Reductions in strength due to thermal aging at 390 and 450°C were attributed to loss of interaction solid-solution hardening (ISSH), whereas at 500 and 550°C a combination of loss of ISSH and precipitate coarsening were responsible. Similar explanations were used to explain effects of irradiation at 450 and 500°C but the increase in strength following 390°C irradiation was attributed to the development of dislocation-loop structure and the decrease in strength following irradiation at 550°C was concluded to be due to the enhanced kinetics of the precipitation coarsening reaction during irradiation.

The present effort is intended to support the previous studies by providing microstructural information on several specimens of interest. The specimens examined were either sections from the density change specimens or were unstressed disks which matched the irradiation conditions of the pressurized tube specimens. The disk specimens had been irradiated to twice the exposure received by the tensile specimens. Therefore, microstructural information from these specimens should be able to answer several questions raised in previous work. For example, is the mechanism, swelling enhanced creep, applicable in pressurized tubes irradiated to only 25 dpa? Could a density change peak at 540°C be due to void swelling? Is the explanation for postirradiation tensile behavior reasonable?

The present effort is also applicable to a companion study of irradiation effects in 2-1/4Cr-1Mo. A task paralleling the irradiation effects data base development is to provide a basis for extrapolating that data base to irradiation regimes and doses which more closely duplicate fusion first wall conditions.⁽³⁾ The objectives of that task are threefold: 1) to compare microstructural evolution under ion bombardment with that under neutron bombardment in order to accurately model void, dislocation and precipitate evolution during irradiation, 2) to develop theoretical models to predict radiation damage and, 3) to generate lifetime analyses of Tokamak and Tandem Mirror Ferromagnetic Reactor Structures. Therefore, the present microstructural studies are expected to provide both a basis for comparison of microstructural evolution between ion and neutron bombardment and quantitative information which should be directly applicable to theoretical model calculations.

3.0 EXPERIMENTAL PROCEDURE

Specimens of 2-1/4Cr-1Mo selected for microstructural examination were obtained from three irradiation tests. Details regarding alloy chemistry and specimen fabrication have been previously described.⁽¹⁾ Two heats of material are involved and alloy chemistries are reproduced in Table 1. Heat treatment conditions are given in Table 2. All tests were irradiated in the Experimental Breeder Reactor (EBR-II) located in Idaho Falls, ID. The first test designated AAI contained rod specimens 1.25 cm x 0.3 cm in diameter. Details of the irradiation test are given in Reference 1. The second test called AAXIV phase 2 contained, among other conditions, both pressurized tubes and disk specimens of 2-1/4Cr-1Mo. The disk specimens were 0.3 cm x 3.0 cm in diameter and were included to provide specimens for microstructural examination to match irradiation conditions for mechanical properties samples. However, the disk specimens were not in the same pin as the pressurized tubes. The disks were contained in pins B326 and B326a. Pin B326 received an accumulated fast fluence of $2.6 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) or 12.5 dpa and was a weeper design which allowed flowing sodium coolant from the reactor to directly contact the specimens. The

TABLE 1
CHEMICAL ANALYSIS OF 2-1/4Cr-1Mo HEATS AS SUPPLIED BY THE VENDORS
(in weight percent)

<u>Element</u>	<u>Rod Specimens Heat 38649</u>	<u>Disk Specimens Heat C4337-14S</u>
C	0.12	0.093
Mn	0.42	0.52
P		0.011
S		0.011
Si	0.21	0.17
Ni	0.16	0.40
Cr	2.17	2.15
Mo	0.93	0.95
Cu		0.16
Al		0.003
Fe	bal	bal

TABLE 2
HEAT TREATMENTS GIVEN ROD AND DISK
SPECIMENS PRIOR TO IRRADIATION

<u>Specimen Type</u>	<u>Heat #</u>	<u>Heat Treatment*</u>
Rod Specimens	C4337-14S	1010°C/1 hr/WQ + 843°C/2 hr/WQ
Disk Specimens	38649	900°C/30 min./AC + 700°C/1 hr/AC

*Temperature/time at temperature/cooling procedure
where AC = air cooled, WQ = water quenched

design temperature was 400°C and thermal expansion devices (TEDs) indicated that the actual temperature was $395 \pm 5^\circ\text{C}$. Disk specimens were then reconstituted into pin B236a. Pin B236a received an accumulated fast fluence of $2.5 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) or 12 dpa and was of B7 design which employed gas gap regulation of gamma heating losses in order to obtain the desired temperature in an internal subcapsule. The design temperature was 412°C but TEDs indicate that the actual temperature was $425 \pm 10^\circ\text{C}$. Therefore, the temperature is given as $410 \pm 20^\circ\text{C}$ for the entire AAXIV phase 2 irradiation experiment. The specimens irradiated in the third test, designated AD2, involved pins B317, B318, B321, B317a, B318a and B319a. Each was a B7 design and the accumulated fast fluence was estimated at $6.1 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) or 29 dpa. Design temperatures were 400, 450 and 500°C but TED results are not yet available for verification. The disk specimen conditions do not exactly match the tensile specimen conditions irradiated in the same experiment. The disk specimens were more central in the reactor core and therefore received a higher neutron flux by about 25%. Specimens selected for examination are listed by sample identification number in Table 3.

Specimens were prepared for transmission electron microscopy as follows. Rod specimens were sliced into disks 0.25 cm x 3.0 cm in diameter using a slow speed saw with diamond impregnated blades. All specimens were briefly electropolished in a 5% perchloric acid, 95% butanol solution at 30 V to remove surface corrosion. At least two foils of each condition were prepared. Thin foil specimens were then produced using a metalthin twin jet electropolishing machine which was located in a radiation shielded facility equipped with manipulators. The polishing solution was the same with polishing conditions at 65 V and moderate pump speed. As is often found to be the case for lower chromium alloys, surface staining was often found to be a problem in which case, a very brief "flash polish" was helpful.

Microstructural examinations were performed for the most part on a JEOL 1200EX electron microscope operating at 120 KeV. One specimen of 94L6 was also examined on a JEOL 100 CX electron microscope operating at 100 KeV.

TABLE 3
IRRADIATION CONDITIONS OF SPECIMENS SELECTED
FOR MICROSTRUCTURAL EXAMINATION

ID	Experiment	Irradiation	
		Temperature °C	Fluence x 10 ²² n/cm ² (dpa)
94M6	AAI	400	16.0 (76)
94L6	AAI	425	20.7 (99)
94E7	AAI	450	15.5 (74)
94F6	AAI	480	19.8 (95)
94K6	AAI	510	24.1 (116)
NUYL	AAXIV	410 $\pm$ 20	5.1 (24)
31CP	AD2	390	5.0 (24)
31CV	AD2	450	5.0 (24)
31CR	AD2	500	5.0 (24)

The general procedure involved examination of a number of areas of interest, and then following a known tilt of the specimen, a reexamination of those areas. This generally provided void and precipitate information, and in several cases dislocation structure information in areas for which foil thickness could be measured stereoscopically. Thickness determination required measurement of the maximum parallax, ΔH , for the area of interest on a stereo viewer and then computation of the foil thickness from the relationship

$$t = \frac{\Delta H}{2M \sin (\theta/2)} \quad (1)$$

where t is the foil thickness, M is the magnification of the images and θ is angle of tilt between the images.

Quantitative measurements of void, precipitate and dislocation structures employed a measurement device coupled directly to a minicomputer. With the push of a foot pedal, a void image width in the $\langle 110 \rangle$ direction, a precipitate diameter (or length in the case of rod specimens) or a dislocation intersection with a line grid was input to the computer. The computer was programmed to accept a large number of measurements and given the foil thickness, compute the size distributions, total volume fraction and number densities for the input data on request. For void swelling, the program accounted for the faceted nature of the void shape and was able to allow large variation in truncation in a given area of interest with a maximum error due to truncation of 20%. As each feature was measured on a 3X print, the apparatus pin pricked the feature to prevent counting the same feature twice.

4.0 RESULTS

Reporting of the results of microstructural examinations will be divided into three sections. First the microstructures of the AAI specimens will be described. Then the microstructure of the normalized and tempered bainite (AAXIV and AD2) will be described. Finally the results of quantitative measurements for both specimen conditions will be given.

4.1 AAI Specimens

The heat treatment given the AAI specimens (Table 2) produced a complex microstructure. The material was in the form of 1.2 cm thick plate when heat treated and the water quenches called for at the end of each treatment step would have resulted in cooldown within about 100 seconds. Primarily upper bainite with no proeutectoid ferrite is expected when cooling from the austenite region under such conditions as can be shown from the continuous cooling transformation (CCT) diagram⁽⁴⁾ given in Figure 1. The first step of the treatment, normalization at 1010°C for one hour followed by a water quench should produce ~75% upper bainite with the remainder lower bainite and martensite. Reheating to 843°C put the specimens in the two phase region (between the Ac_1 at 740°C and the Ac_3 at 880°C). Therefore, austenite formed and some ferrite was retained. However, all carbides should be dissolved. Water quenching should again cause the austenite to transform primarily to upper bainite with no proeutectoid ferrite formed during cooling. The microstructure produced by this treatment is shown in Figures 2 and 3. Figure 2 gives optical metallography at a series of magnifications for the preirradiation structure. The structure is fine (prior austenite grain size ASTM #8) with at least two phases easily identified: a bainite structure similar to lath martensite which appears as gray regions with internal linear structure and nonequiaxed ferrite which appears as white, substructure-free, regions. Figure 3 provides examples of transmission electron microscopy for this structure. Three distinctly different regions can be identified. Large precipitate-free regions with low dislocation densities are regions which were retained ferrite at 843°C. The remaining structure was austenite at 843°C which transformed to bainite on cooling. Both upper and lower bainite are present. In upper bainite, the carbide particles frequently form in elongated configurations between the bainite (ferrite) grains whereas in lower bainite the carbides tend to precipitate at an inclined angle to the major growth direction, or longitudinal axis, of the bainitic ferrite grains.⁽⁵⁾ In Figure 3, examples of retained ferrite, upper bainite and lower bainite have been labeled. Therefore, postirradiation microstructural examination of AAI conditions should include each of these regions.

FIGURE 1. Continuous Cooling Transformation Diagram for 0.1 Percent Carbon 2-1/4Cr-1Mo Steel, ASTM Grain Size 7. F = polygonal ferrite, P = pearlite, B = bainite, M = martensite.

FIGURE 2. Optical Metallography of Specimen 94 Prior to Irradiation.

FIGURE 3. Microstructure Prior to Irradiation for Specimen 94 at Low Magnification. The various regions are labeled.

Specimen 94M6, irradiated at 400°C to 1.6×10^{23} n/cm² ($E > 0.1$ MeV) or 76 dpa was found to retain the preirradiation microstructure but void swelling, dislocation network evolution and precipitation occurred as a result of irradiation. An example of the microstructure at low magnification is shown in Figure 4. At the upper left, a retained ferrite region is shown in dislocation contrast. Small voids can be seen but the precipitate structure is not very apparent. In comparison, at the upper right a region corresponding to upper bainite can be seen in heavy strain contrast. The void density is lower, and elongated carbide particles separating bainite plates can be seen. At the lower right a region of lower bainite is present. The precipitate particles are larger and more numerous than in the retained ferrite region, and the void density appears to be lower. Examples of the three structures are shown at higher magnifications in Figure 5. The most notable differences between them are in precipitate size and distribution and void distribution. Precipitate number density is greatest in the retained ferrite region and lowest in the upper bainite region. However, both the upper bainite and lower bainite regions contain the elongated precipitate particles characteristic of the retained ferrite region. It is anticipated that such particles have formed during irradiation whereas the larger particles originated in the preirradiation heat treatment. Voids are uniformly distributed in retained ferrite regions and lower bainite regions whereas they are non-uniformly distributed in the upper bainite region. Some areas in the upper bainite region contain very few voids.

One area of retained ferrite in specimen 94M6 was used to examine the irradiation induced dislocation network in detail. An area oriented near [011] was imaged using $\bar{g} = 01\bar{1}$, $21\bar{1}$ and 200 for dislocation contrast as shown in Figure 6. Comparison of the images from $\bar{g} = 01\bar{1}$ and 200 reveals that few dislocations appear in both micrographs. This indicates that these dislocations are predominantly of $a\langle 100 \rangle$ character because if they were of type $a/2 \langle 111 \rangle$, then all would show up for $\bar{g} = 200$ and half would show up for $\bar{g} = 01\bar{1}$. A table which can be used to demonstrate this point is provided in Table 4. For those $\bar{g} \cdot \bar{b}$ products equal to zero, dislocations of that Burgers vector should be invisible whereas products which

FIGURE 4. Microstructure in Specimen 94M6 Irradiated at 400°C to $1.6 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at Low Magnification.

FIGURE 5. Examples of Microstructure in Specimen 94M6 Irradiated at 400°C to $1.6 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) Showing a) Ferrite, b) Upper Bainite and c) Lower Bainite.

FIGURE 6. Examples of Dislocation Structure in Specimen 94M6 Irradiated at 400°C to 1.6×10^{23} n/cm² ($E > 0.1$ MeV) Showing the Same Area Under a) $\bar{g} = 01\bar{1}$, b) $\bar{g} = 21\bar{1}$ and, c) $\bar{g} = 200$ Contrast Conditions.

TABLE 4

THE $\bar{g} \cdot \bar{b}$ IMAGING CRITERION FOR VARIOUS IMAGING
CONDITIONS AND BURGERS VECTORS POSSIBLE IN 2-1/4Cr-1Mo STEEL

$\bar{g}$	$\bar{b}$						
	$\frac{a}{2} [111]$	$\frac{a}{2} [\bar{1}11]$	$\frac{a}{2} [1\bar{1}1]$	$\frac{a}{2} [11\bar{1}]$	$a[100]$	$a[010]$	$a[001]$
200	1	1	1	1	2	0	0
020	1	1	1	1	0	2	0
002	1	1	1	1	0	0	2
01 $\bar{1}$	0	0	1	1	0	1	1
$\bar{1}10$	0	1	1	0	1	1	0
10 $\bar{1}$	0	1	0	1	1	0	1
11 $\bar{2}$	0	1	1	2	1	1	2
1 $\bar{2}1$	0	1	2	1	1	2	1
$\bar{2}11$	0	2	1	1	2	1	1
2 $\bar{1}1$	1	1	2	0	2	1	1
21 $\bar{1}$	1	1	0	2	2	1	1
1 $\bar{1}2$	1	0	2	1	1	1	2
12 $\bar{1}$	1	0	1	2	1	2	1

are nonzero should allow visibility. Note that for $\bar{g} = 200$ only dislocations with Burgers vector $a[100]$ should be visible; $a[010]$ and $a[001]$ should be invisible. (However, all $a/2 [111]$ should be visible.) From Figure 6 it can be shown that perfect dislocation loops are present, that they tend to be on $\{001\}$ planes and that they are pure edge in character. Many examples of $a\langle 001 \rangle$ dislocation segments can also be found. Therefore, the dislocation structure is in the process of transferring from a loop dominated structure to a network dominated structure.

Specimen 94L6, irradiated at 425°C to $2.1 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) or 99 dpa in many ways resembled specimen 94M6 irradiated at 400°C . Regions of retained ferrite, upper bainite and lower bainite could be identified, and void swelling was present. However, the amount of swelling was reduced due to reduced void number densities and voids were absent in upper bainite regions. Also the precipitates in retained ferrite regions were larger so that differentiation between retained ferrite and lower bainite regions was much more difficult. An example of the structure at low magnification is shown in Figure 7. The central region contains upper bainite and by comparison with Figures 3 and 4 it is apparent that spheroidization of the carbide particles at bainite plate boundaries has occurred. In the lower part of Figure 7 is a region of retained ferrite and at the upper left, a region can be seen of lower bainite which appears to be in dislocation contrast. The precipitate structure in these two regions appear similar in size. Examples of structures are shown at higher magnification in Figure 8. In Figure 8a, an example is given of non-uniform void development within a retained ferrite region and similar conclusions can be made from Figure 8c, a lower bainite region. This comparison also shows that swelling in retained ferrite is higher and that the precipitate structures appear very similar.

Specimens 94E6 and 94F6 irradiated at 450°C to $1.6 \times 10^{23} \text{ n/cm}^2$ and 480°C to $2.0 \times 10^{23} \text{ n/cm}^2$ respectively, were quite similar. In fact, due to foil preparation difficulty which resulted in surface pitting, insufficient thin area was available in these specimens for low magnification micrographs which provide a better understanding of this complicated microstructure.

FIGURE 7. Microstructure of Specimen 94L6 Irradiated at 425°C to $2.1 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at Low Magnification.

FIGURE 8. Example of Microstructure in Specimen 94L6 Irradiated at 425°C to $2.1 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) Showing a) Ferrite, b) Upper Bainite and, c) Lower Bainite.

However, it was possible to show that retained ferrite regions did contain voids in both cases. The void density was low and not all regions contained voids. In comparison, no voids were identified in upper bainite or lower bainite regions. Also, lower bainite regions appeared to contain more rod shaped precipitates than had been found for lower irradiation temperatures. (However, this generalization may be incorrect due to poor sampling procedures.) Examples of these structures in 94E6 and 94F6 are given in Figures 9 and 10, respectively.

Specimen 9LK6, irradiated at 510°C to 2.4×10^{23} n/cm² was the only specimen in this series of AAI specimens which did not contain voids. The structure was overaged compared to the lower irradiation temperature conditions and grain growth in the upper bainite regions was apparent. A low magnification example of this structure is shown in Figure 11. A small grain of retained ferrite can be seen in the lower portion of this figure, two regions of lower bainite appear at the left edge of the micrograph and the central portion is an upper bainite region. Comparisons with the pre-irradiation structure in Figure 3 and the 400°C irradiation condition in Figure 4 show why the strength of this material is not maintained at 510°C in-reactor. Examples of the structure of the 510°C sample at higher magnification are shown in Figure 12. Both the retained ferrite and lower bainite are shown in dislocation contrast. The dislocation density is low in both cases.

4.2 AAXIV and AD2 Specimens

Descriptions of microstructural observations on specimens from the AAXIV and AD2 tests are combined because the irradiation experiments were designed to be almost identical. The same heat of 2-1/4Cr-1Mo was used with the same heat treatment. The irradiation temperatures were similar (the AAXIV test did not include a 500°C condition) and the goal fluences were both 6×10^{22} n/cm². As an objective of this part of the experiment is to determine the effect of irradiation on microstructure for a standard heat treatment condition in order to provide interpretation of in-reactor creep and tensile test response, this series of specimens provides the most complete set available for microstructural examination.

FIGURE 9. Examples of Microstructure in Specimen 94E6 Irradiated at 450°C to 1.6×10^{23} n/cm² ($E > 0.1$ MeV) Showing a) Ferrite, b) Upper Bainite and c) Lower Bainite.

FIGURE 10. Examples of Microstructure in Specimen 94F6 Irradiated at 480°C to 2.0×10^{23} n/cm² ($E > 0.1$ MeV) Showing a) Ferrite, b) Upper Bainite and c) Lower Bainite.

FIGURE 11. Microstructure of Specimen 94K6 Irradiated at 510°C to $2.4 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at Low Magnification.

FIGURE 12. Examples of Microstructure in Specimen 94K6 Irradiated at 520°C to 2.4×10^{23} n/cm² ($E > 0.1$ MeV) Showing a) Ferrite, b) Upper Bainite and c) Lower Bainite.

The heat treatment given this material was intended to provide a fully tempered bainite structure. Air cooling (from 900°C) was expected to result in cooling rates slower than for the AAI material but fast enough to avoid proeutectoid ferrite (i.e., in less than 1000 seconds) as shown in Figures 13 and 14. The microstructure was found to be lower bainite and consisted of large bainite plates with large needle shaped M_7C_3 precipitate particles within the bainite plates and blocky M_6C or $M_{23}C_6$ particles on or near grain boundaries. Examples of the structure in specimen NU which was heat treated with the specimens irradiated in the AAXIV test as revealed by optical metallography are given in Figure 13 and as found by transmission electron microscopy are shown in Figure 14. In Figure 14, a grain boundary node can be seen towards the top of the micrograph. Needle shaped carbide particles are found to be non-uniformly distributed in the structure. The microstructure in specimen 31, heat treated with the specimens irradiated in the AD2 test was found to be very similar to specimen NU. A comparison is provided in Figure 14c showing the dislocation and precipitate structure for specimens NU and 31 at higher magnifications. A good example of subgrain dislocation cell walls can be seen at the upper right of Figure 14c.

Specimens NUYL irradiated at 410°C to $5.1 \times 10^{22} \text{ n/cm}^2$ and 31CP irradiated at 390°C to $5.0 \times 10^{22} \text{ n/cm}^2$ were found to contain very similar microstructures. In both cases, a high density of uniformly distributed small voids was found and small spherical particles had developed between the rod shaped carbide particles which were present prior to irradiation. The major difference between these structures was in the size of the particles which formed during irradiation. Examples of the microstructure of specimen NUYL are shown in Figures 15 and 16 and those of specimen 31CP are shown in Figures 17 and 18. Figure 15 shows a prior-austenite grain boundary node approximately in the center of the micrograph with several subgrain (bainite) boundaries in the field of view. The grain at the right is in heavy strain contrast and therefore somewhat obscured. The larger precipitate particles which can be seen at grain boundaries and within grains were created prior to irradiation. The structure which formed during irradiation between these particles is shown at higher magnification in Figure 16. Figure 16a provides

FIGURE 13. Optical Metallography of Specimen NU prior to Irradiation.
Specimen 31 was similar.

FIGURE 14. Microstructures Prior to Irradiation for Specimen NU at a) Low Magnification and b) Intermediate Magnification and for Specimen 31 at c) Intermediate Magnification.

FIGURE 14 (Cont'd).

FIGURE 15. Microstructure of Specimen NUYL Irradiated at 410°C to $5 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at Low Magnification.

FIGURE 16. Microstructures of Specimen NUYL Irradiated at 410°C to $5 \times 10^{23} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) Showing Examples of a) Void and b) Dislocation Structure. Note the fine precipitate in the background.

FIGURE 17. Microstructure of Specimen 31CP Irradiated at 390°C to $5 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at Low Magnification.

FIGURE 18. Microstructure of 31CP Irradiated at 390°C to $5 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) Showing Examples of a) Void and b) Dislocation Structure.

an example of void and precipitate development near grain boundaries and Figure 16b shows a region in strain contrast in order to image the dislocation structure. The voids are found to be faceted and to form quite close to grain boundaries. The dislocation structure consists of a dislocation net- work as opposed to individual dislocation loops. However, on a finer scale a high density of equiaxed particles can be seen. Figure 17 shows similar features at low magnification in specimen 31CP. A contorted grain boundary runs across the micrograph and a region at the upper left is obscured by strong strain contrast. Some large pits can be seen (one right in the center of the micrograph) and should be ignored because they are specimen preparation artifacts. Figure 18 shows this structure at higher magnification. The void density in specimen 31CP as shown in Figure 18a appears to be higher than specimen NUYL and the precipitates formed during irradiation appear to be smaller. Figure 18b provides an example on the left of a small region in dislocation contrast. Again a dislocation network rather than a dislocation loop structure is found.

The microstructure of specimen 31CV irradiated at 450°C to $5.0 \times 10^{22} \text{ n/cm}^2$ was quite different. No voids were found. Precipitates which formed during irradiation could be identified but the density was lower than the 400°C sample and the dislocation structure consisted of a loose tangle with no loops. This microstructure is shown at low magnification in Figure 19. Effects of irradiation are difficult to identify. However, at higher magnification, as shown in Figure 20, a low density of precipitate particles can be seen and examples of a dislocation network can be found.

Specimen 31CR irradiated at 500°C to $5.0 \times 10^{22} \text{ n/cm}^2$ was not very different. No voids were found. The precipitate structure may have contained somewhat more acicular precipitate (a tendency which can also be observed in AAI specimens irradiated at ~500°C) but this may be a consequence of statistical uncertainty and not a true effect. An example of this structure is given in Figure 21 showing a prior austenite grain boundary node at low magnification and examples at higher magnification are given in Figure 22. Figure 22b shows a region in strong strain contrast. Examples of subgrain boundary development can be found in several places.

FIGURE 19. Microstructure of Specimen 31CV Irradiated at 450°C to 5×10^{22} n/cm² ($E > 0.1$ MeV) at Low Magnification.

FIGURE 20. Microstructures of Specimen 31CV Irradiated at 450°C to $5 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) Showing Two Regions in Precipitate and Dislocation Contrast.

FIGURE 21. Microstructure of Specimen 31CR Irradiated at 500°C to $5 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) at Low Magnification.

FIGURE 22. Microstructure of Specimen 31CR Irradiated at 500°C to $5 \times 10^{22} \text{ n/cm}^2$ ($E > 0.1 \text{ MeV}$) Showing a) Precipitate Structure and b) Dislocation Structure.

4.3 Quantitative Microstructural Measurements

The preceeding results have shown that the major consequences of fast neutron irradiation on 2-1/4Cr-1Mo microstructures are the formation and development of voids, precipitates and dislocations. Voids were only found for irradiation temperatures below 500°C with swelling apparently at a maximum for the lowest temperature examined, 400°C. In both heat treatment conditions new precipitates formed during irradiation. This was most noticeable at the lowest irradiation temperature, 400 to 450°C whereas at a higher temperature it was often not possible to easily differentiate between precipitates which formed during irradiation and those which were present prior to irradiation and only grew during irradiation. Both equiaxed and rod-shaped precipitates were present, but no attempt was made in this study to identify either the crystal structures or compositions of these phases. A possible identification is Mo_2C for the rod shaped precipitates and M_6C or M_{23}C_6 for the equiaxed precipitates. However, it is conceivable that equiaxed precipitates of two types coexist. Dislocation structures which developed due to the agglomeration of irradiation induced point defects could be identified only at 400°C. At higher temperatures, a dislocation network was formed which was difficult to distinguish from the dislocation structure present prior to irradiation or the scale of the dislocation structure was obscured by the precipitate structure. The intent of this section is to present results of quantitative measurements of voids, precipitates and dislocations made from micrographs of these structures.

The results of quantitative measurements on micrographs of AAI specimens are given in Table 4 and similar measurements on AAXIV and AD2 specimens which were examined are provided in Table 5. Both tables identify the micrograph negative number from which measurements were made, provide swelling, number density and mean diameter for void populations, volume fraction, number density and mean diameter for equiaxed particles, number density and mean particle length for rod shaped particles and dislocation density. In many cases, micrographs did not provide clear images of all the features of interest in a single micrograph. Therefore, features which were out of con-

SUMMARY OF QUANTITATIVE MICROSTRUCTURAL MEASUREMENTS FOR AAI SPECIMENS

Specimen	Structure	Negative Number	Voids			Precipitates/Spheres			Rods		Dislocations
			Swelling $\Delta V/V_0, \%$	Density $\#/cm^3$	Mean Diameter nm	Volume %	Density $\#/cm^3$	Mean Diameter nm	Density $\#/cm^3$	Mean Length nm	
94M6	Ferrite	303	0.47	1.05×10^{15}	16.2	1.61	1.5×10^{15}	17.8	1.13×10^{15}	28.2	
		303	0.35	1.5×10^{15}	15.4	1.95	9.7×10^{14}	25.6	9.9×10^{14}	32.7	
		608	0.26	1.14×10^{15}	16.7						1.7×10^{10}
		601	0.38	1.24×10^{15}	17.0	0.13	9.1×10^{14}	11.5	2.8×10^{15}	27.0	3.2×10^{10}
		585	0.19	1.18×10^{15}	13.1	1.72	1.9×10^{15}	23.3	3.3×10^{15}	28.4	
		592	0.24	1.08×10^{15}	15.6	1.58	8.1×10^{14}	23.6	2.2×10^{15}	27.9	4.1×10^{10}
		576	0.21	0.93×10^{15}	16.5	0.74	9.6×10^{14}	23.2	1.3×10^{15}	31.5	
		301	0.13	0.93×10^{15}	14.2					33.3	
		363	0.06	0.52×10^{14}	12.7	0.68	8.67×10^{14}	15.6	9.6×10^{14}	30.7	
		116	0.12	1.5×10^{15}	10.3				8.1×10^{14}	25.9	
		median	0.22	1.1×10^{15}	15.5	1.6	9.5×10^{14}	23.0	1.2×10^{15}	28.0	3×10^{10}
	Lower Bainite	363	0.063	2.9×10^{14}	16.0	1.83	9.7×10^{14}	27.4	4.1×10^{16}	44.2	
		363	0.031	2.4×10^{14}	13.0	1.99	5.3×10^{14}	36.3	3.0×10^{16}	46.7	
		357	0.060	4.7×10^{14}	12.0						4.9×10^{10}
		357	0.041	6.3×10^{14}	8.1						
		353	0.067	2.8×10^{14}	14.8	1.42	6.6×10^{14}	22.5	1.9×10^{15}	23.3	
		576				3.83	1.5×10^{15}	32.2	9.4×10^{15}	30.5	
		median	0.06	3×10^{14}	13.0	1.9	8×10^{14}	30.0	7×10^{14}	37.0	5×10^{10}
	Upper Bainite	591	0.026	9.3×10^{13}	16.3	4.4	8.4×10^{16}	38.5	4.6×10^{14}	33.7	
		591	0.039	1.3×10^{14}	18.4	8.5	6.7×10^{14}	42.2			
		301	0.065	2.2×10^{14}	11.6	7.4	9.6×10^{14}	38.6			
		301	0.060	1.8×10^{14}	15.2		4.7×10^{14}	43.4	2.6×10^{14}	35.0	
		360	0.009	1.2×10^{14}	10.8	2.3	5.7×10^{14}	32.5	4.0×10^{14}	30.4	
		116				12.0	5.8×10^{14}	45.9			
		589	0.048	9.3×10^{13}	16.4	11.3	4.2×10^{14}	46.9			
		median	.045	1.3×10^{14}	16.0	8.0	6.0×10^{14}	42.0	4.0×10^{14}	33.0	N.M.
94L6	Ferrite	287	0.098	3.8×10^{13}	38.5	1.59	5.6×10^{14}	36.9*		36.9	1.3×10^{10}
		249	0.088	7.6×10^{13}	17.7	1.63	1.34×10^{15}	22.0	1.35×10^{15}	40.2	
		264	0.107	3.1×10^{13}	34.6	1.31	8.6×10^{14}	24.8	1.29×10^{15}	40.0	1.2×10^{10}
		259	0.074	4.5×10^{13}	27.7	1.32	7.7×10^{14}	26.2	1.16×10^{15}	40.5	2.9×10^9
		276	0.055	4.8×10^{13}	23.4	2.16	9.6×10^{14}	24.0	1.17×10^{15}	40.0	1.5×10^{10}
		273	0.000	0	---						1.5×10^{10}
		15929	0.000	0	---	2.10	5.2×10^{14}	36.6	9.1×10^{14}	58.3	1.5×10^{10}
		median	.075	4.0×10^{13}	28.0	1.6	8.0×10^{14}	25.0	1.2×10^{15}	40.0	1.5×10^{10}
	Lower Bainite	251	0.027	4.5×10^{13}	22.7	2.6	1.5×10^{15}	24.2	1.3×10^{15}	47.9	8.1×10^9
	Upper Bainite	287	0.007	1.9×10^{13}	13.8	9.0	5.2×10^{14}	37.3			
		273	0.00	0	---	8.8	5.7×10^{14}	35.5	9.5×10^{13}	41.0	
		median	0.004	1×10^{13}	13.8	8.9	5.5×10^{14}	36.0	1×10^{14}	41.0	N.M.
	Ferrite	178	0.021	2.3×10^{13}	47.7	3.7	3.8×10^{14}	41	6.0×10^{14}	41.0	5.4×10^9
			0.000	0	---						1.4×10^{10}
		192	0.031	1.6×10^{13}	30.0	2.7	6.6×10^{14}	35	3.5×10^{14}	60.2	
			0.000	0	---				2.8×10^{15}	41.4	
		median	0.01	8×10^{12}	38	3.2	7.5×10^{14}	38	6×10^{14}	41.0	9×10^9
	Upper Bainite	173	0	0	---	1.5	1.1×10^{15} *	22.8*	6.1×10^{14}	26.2	
94F6	Ferrite	213	0.214	6.1×10^{13}	45.1	4.5	3.6×10^{14}	52.9			6.1×10^9
		217	0.020	2.3×10^{13}	42.0	4.6	1.9×10^{14}	59.3			
		212	0.161	4.6×10^{13}	41.0	6.2	4.0×10^{14}	56.1			
		214									3.2×10^9
	Upper Bainite		0.16	5×10^{13}	42	4.5	4×10^{14}	56			5×10^9
		237	0	0	---				1.1×10^{15}	42.3	
94K6	Ferrite	371	0	0	---	7.6	9.4×10^{14}	50.4			
		367	0	0	---	9.8	7.5×10^{14}	47.6			4.6×10^9
		367	---	---	---	---	1.8×10^{14}	65.3			5.0×10^9
		median	0	0	---	8.5	7.5×10^{14}	50	4×10^{14}	70	5×10^9
	Upper Bainite										
		375	0	0		4.9	2.2×10^{14}	37.1	4.9×10^{13}	48.9	

*Bimodal distribution

N.M. Not measured

TABLE 5

SUMMARY OF QUANTITATIVE MICROSTRUCTURAL MEASUREMENT FOR AAXIV AND AD2 SPECIMENS

Specimen	Negative Number	Voids			Precipitates/Spheres			Rods		Dislocations
		Swelling $\Delta V/V_0$, %	Density $\#/cm^3$	Mean Diameter nm	Volume %	Density $\#/cm^3$	Mean Diameter nm	Density $\#/cm^3$	Mean Length nm	Density $\#/cm^2$
NUYL	314	.065	7.4×10^{14}	11.7	0.53	4.9×10^{15}	11.5	3.1×10^{14}	31.1	N.M.
31CP	338									4.4×10^{10}
	336	.11	2.1×10^{15}	9.6	0.1	3.0×10^{15}	8.1	1.6×10^{15}	34.5	
	336	.12	1.7×10^{15}	10.3	8.0	6.3×10^{15}	10.1	1.02×10^{15}	45.5	
	347	.23	2.7×10^{15}	10.1	3.3	4.5×10^{15}	13.7			
	median	.1	2×10^{15}	10	2.	5×10^{15}	11	1×10^{15}	31	4×10^{10}
31CV	469	0	0		2.0	1.2×10^{15}	22.5	8.8×10^{14}	31.3	3.4×10^{10}
	465	0	0		2.2	1.15×10^{15}	21.3	2.5×10^{14}	69.2	
		0	0		2.	1.2×10^{15}	22	5×10^{14}	50	3×10^{10}
31CR	478	0	0		3.2	4.5×10^{14}	35.0	5.2×10^{14}	56.5	
	497	0	0		1.7	3.9×10^{14}	32.8	5.0×10^{14}	84.7	
	483	0	0			2.6×10^{14}	46.1	2.6×10^{14}	102.	
	median	0	0		2.5	4×10^{14}	35.	5×10^{14}		

trast were not measured and there are gaps in the tables. In Table 4, measurements are reported for the three microstructural areas of interest: ferrite, upper bainite and lower bainite. However, because of difficulty differentiating between ferrite and lower bainite following irradiation at higher temperature, lower bainite results are reported as measurements in ferrite. This is the case for 94E6, 94F6 and 94K6.

The results of Tables 4 and 5 may be interpreted as follows. Void swelling as high as 0.5% was measured in specimen 94M6 irradiated in AAI at 400°C but large variations in swelling were found from area to area with median values of 0.22% in ferrite, 0.06% in lower bainite and 0.045% in upper bainite. Void densities in this specimen condition were on the order of $1 \times 10^{15} \text{ cm}^{-3}$ in ferrite, $3 \times 10^{14} \text{ cm}^{-3}$ in lower bainite and $1.3 \times 10^{14} \text{ cm}^{-3}$ in upper bainite. Void densities were similar in specimens NUYL and 31CP in the bainitic condition, $2 \times 10^{15} \text{ cm}^{-3}$, but swelling was lower, with peak swelling measured of 0.23% and median swelling measured of 0.10% following irradiation at 400°C. Figure 23 has been constructed to show the temperature dependence of void development. Figure 23a gives the swelling as a function of irradiation temperature for ferrite in AAI specimens. Results for AD2/ AAXIV specimens at 400 and 450°C are also given. Swelling is highest at 400°C but void measurements indicate a secondary peak occurs at 480°C in the AAI specimen condition. Density change measurements did not show this secondary peak. It is therefore possible that the peak appears due to area to area variation and a resultant statistical bias in measurement which arose due to specimen preparation difficulties. Further examinations would be required to eliminate this possibility. The void density results shown in Figure 23b also show a secondary maximum at 480°C but the void density is much higher at 400°C.

Precipitate development follows similar trends. With increasing temperature, precipitate number density decreases and mean precipitate size, or length for rod shaped precipitates increases. However estimates of precipitate volume fraction for spherical precipitates does not vary systematically with temperature. This is believed to be a consequence of precipitates present prior to irradiation in the area of interest which are included in some measurements. The results of precipitate measurements in AD2/AAXIV

FIGURE 23. Void Measurements in Ferrite AAI and AD2/AAXIV Specimen Conditions as a Function of Temperature. Only results for ferrite in AAI specimens are shown.

specimens and in ferrite in AAI specimens are shown in Figure 23. Comparison of Figures 22 and 23 indicates that precipitate size and number densities of spherical precipitates are at least as large as those for voids.

Dislocation densities are found to be between 10^9 and 10^{11} cm³. The AD2/AAIV condition was somewhat higher than the AAI condition but both followed the same trend with temperature: as irradiation temperature was increased the dislocation density decreased. Figure 25 has been prepared to demonstrate this behavior.

5.0 DISCUSSION

The present results provide information which answers several questions concerning the behavior of irradiated 2-1/4Cr-1Mo steel but which also raises several further questions. It is now demonstrated that void swelling, dislocation loop development and precipitation occur as a result of fast neutron irradiation in the temperature range 400 - 480°C in 2-1/4Cr-1Mo steel. The purpose of this discussion will be to summarize these results and to assess the implications of these results on the interpretation of in-reactor creep, density change and post irradiation tensile behavior

5.1 Effects of Irradiation on Microstructure

Fast neutron irradiation of 2-1/4Cr-1Mo produces microstructural features which are similar to those found in other steels⁽⁶⁾. For example, the AAI material has now been examined following irradiation at three fluence levels, 5×10^{22} n/cm², 1.8×10^{23} n/cm² and 2.5×10^{23} n/cm².⁽⁶⁾ It is found that for irradiation at 425°C, precipitation and dislocation evolution occur rapidly, by 5×10^{22} n/cm² but void formation is only found after 1.6×10^{23} n/cm² and further void

FIGURE 24. Measurements of Precipitates in Ferrite in AAI and AD2/AAXIV Specimen Conditions as a Function of Temperature.

FIGURE 25. Measurements of Dislocation Density Plotted as a Function of Irradiation Temperature.

development after $2.1 \times 10^{23} \text{ n/cm}^2$ is found to have produced little further swelling⁽⁶⁾. The peak swelling temperature is at 400°C or below and density measurements show an increase in swelling of only 0.16% from 1.4×10^{22} to $1.6 \times 10^{23} \text{ n/cm}^2$ at 400°C. (1) Therefore, 2-1/4Cr-1Mo is highly swelling resistant but swelling does occur in the alloy.

The onset of swelling occurred at much lower fluence for specimens in both the AD2 and AAXIV test. The reason for this difference cannot be determined at this time; several possibilities exist. However, it is likely to be a consequence of either differences in heat treatment procedure or heat-to-heat variation. Heat treatment differences provide the possible correlation that more carbon in solution delays the onset of swelling whereas compositional differences such as nickel or minor impurity effects may have controlled the swelling response. Examination of alternate heat treatment conditions included in the AD2 test may provide the answer.

5.2 Implications for Irradiation Creep Modeling

The present results validate two assumptions drawn in the companion study of irradiated pressurized tubes(1). That study concluded that at 480°C and below, effects of irradiation controlled behavior where at 570°C the thermal creep contributions were significant. The study also concluded that in-reactor creep at 390°C was increased compared with that at 480°C, probably due to swelling enhanced creep. The microstructural results of this study have shown void swelling in 2-1/4Cr-1Mo at temperatures as high as 480°C whereas no irradiation induced microstructure could be identified in a specimen irradiated at 510°C. Therefore, the transition from irradiation induced microstructure controlled to thermally controlled behavior occurs at approximately 500°C for 2-1/4Cr-1Mo. Also, void swelling is found in disk specimens which correspond to the same condition of composition and irradiation history as the pressurized tubes irradiated at 390°C. Therefore, swelling enhanced in-reactor creep compared to the 480°C condition and void swelling causes for diameter change in unstressed tubes can be validated.

5.3 Interpretation of Density Change Measurements

Microstructural examinations reveal two mechanisms which can cause the density changes observed in irradiated AAI specimens. Void swelling can contribute to density change for irradiation temperatures of 480°C or below. Void volume fraction is greatest at the lowest irradiation temperature examined, 400°C, but the accumulated swelling is low, on the order of 0.2% or less even at 400°C. Concurrently, precipitation occurs during irradiation. Precipitation can be expected to lead to changes in density and effects as large as 0.2% are not unreasonable. Therefore, density change measurements for 2-1/4Cr-1Mo specimens irradiated in the AAI test are expected to include contributions due to void swelling and precipitation. However, further work will be required in order to determine which precipitate phases have formed, whether those phases also form during thermal aging and whether those phases can explain the differences between density change measurements and void swelling measurements.

Interpretation of Tensile Test Results

5.4 The present microstructural results demonstrate that interpretation of postirradiation tensile tests for 2-1/4Cr-1Mo specimens must include effects due to irradiation induced (or enhanced) precipitate development. The precipitate structures represent the most densely packed obstacles for dislocation motion in specimens irradiated at 400°C. Therefore, they likely control plastic flow behavior. Void may contribute but are expected to have a smaller effect; the number density is lower, and it is likely that the lower fluence condition of the tensile specimens (compared to those used for microstructural examination) will contain no void swelling whereas irradiation induced precipitates will be present. Dislocation loops will also contribute to plastic flow behavior. The present quantitative microstructural measurements can provide a basis for comparing the effect of these different microstructural components on strength. However, such a comparison is considered to be beyond the scope of the present effort.

Microstructural studies confirm that irradiation at 550°C leads to decreases in postirradiation strength as a consequence of overaging of the precipitate structure and the concurrent loss of ISSH. However, because microstructural comparisons with thermally aged specimens have not yet been made, the reason for strength differences between specimens thermally aged and irradiated at 550°C cannot be identified. Possibilities are that different precipitation reaction sequences apply for the two environments or that the kinetics are different.

6.0 CONCLUSION

A series of nine specimens conditions of 2-1/4Cr-1Mo have been examined by transmission electron microscopy following irradiation by fast neutrons over the temperature range 390 to 510°C. Irradiation was found to produce void swelling, precipitation and dislocation network development at temperatures of 480°C or below whereas at 500°C and above, behavior can best be described as precipitate overaging. Void swelling varied as a function of irradiation temperature, preirradiation microstructure and possibly heat treatment or composition differences with peak swelling on the order of 0.2% found following irradiation at 400°C. The dislocation structure in a retained ferrite grain following irradiation at 400°C was found to contain predominantly $a\langle 100 \rangle$ dislocations with some $a/2\langle 111 \rangle$ dislocations also present.

Quantitative measurements have been made of void, precipitate and dislocation structures in the various distinctly different microstructural regions. Precipitates which formed during irradiation are shown to be a dominant feature which should affect properties.

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