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VOID SWELLING IN BINARY Fe-Cr
ALLOYS AT 200 DPA

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D. S. Gelles

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VOID SWELLING IN BINARY FE-CR ALLOYS AT 200 DPA - D. S. Gelles, (Pacific Northwest Laboratory)*

OBJECTIVE

The objective of this work is to determine lifetime limits for reduced activation ferritic alloys by providing a better understanding of the inherent swelling resistance of ferritic alloys.

SUMMARY

Microstructural examinations have been performed on a series of binary Fe-Cr alloys irradiated in the FFTF/MOTA at 425°C to 200 dpa. The data represent the highest swelling levels reported in neutron irradiated ferritic alloys. The alloy compositions ranged from 3% to 18% Cr in 3% Cr increments, and the irradiation temperature corresponded to the peak swelling condition for this alloy class. Density measurements showed swelling levels as high as 7.4%, with the highest swelling found in the Fe-9Cr and Fe-6Cr alloys. Microstructural examinations revealed that the highest swelling conditions contained well-developed voids, often as large as 100 nm, and a dislocation network comprised of both $\frac{a}{2}\langle 111 \rangle$ and $a\langle 100 \rangle$ Burgers vectors. Swelling was lower in the other alloys, and the swelling reduction could be correlated with increased precipitation. These results are considered in light of the current theories for low swelling in ferritic alloys, but no theory is available to completely explain the results.

PROGRESS AND STATUS

Introduction

The present effort is one of a series that follows a set of simple Fe-Cr alloys ranging from 3% to 18% Cr in 3% Cr increments through a number of irradiation experiments.¹⁻⁴ Irradiations have been performed both in the Experimental Breeder Reactor (EBR) -II^{1,2} and the Fast Flux Test Facility Materials Open Test Assembly (FFTF/MOTA),^{3,4} and the present effort examines the alloys following irradiation in FFTF/MOTA to the highest dose attained, 200 dpa. The intent is to provide further understanding of the processes controlling void swelling in the ferritic alloy class.

Void swelling due to radiation damage is found to be significantly lower in ferritic alloys than in austenitic alloys.^{2, for example} However, the cause of this difference in response is not yet clearly understood. Several theories have been proposed, but none have received completely general acceptance. Three recent papers summarize these theories, and may be referred to for details.⁵⁻⁷ The most pertinent mechanisms are 1) the point defect bias to dislocations is inherently lower in the body centered cubic ferritic crystal structure,⁸ 2) the presence of two competing dislocation Burgers reduces vacancy accumulation at voids,^{1,9} 3) solute trapping of defects leads to increased recombination,¹⁰ and 4) the dislocation and void evolution produce populations that are not ideal for void swelling optimization in ferritic alloys.¹¹⁻¹² However, it is important to differentiate between the two stages controlling swelling response: void nucleation (or swelling incubation) and void growth (or steady state swelling). Many of the mechanisms defined above only provide an

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explanation for the delayed onset of steady state swelling. For example, solute trapping would not be expected to control steady state swelling because the solute would eventually be segregated to point defect sinks. The present microstructural study is more pertinent to steady state swelling response.

Experimental Procedure

The International Nickel Company prepared alloys used for this and previous studies. Compositional impurities have previously been defined,¹ but it is worth emphasizing that the Fe-3Cr alloy has behaved anomalously because it contains significantly more impurity than was originally expected.²

For the present experiment, specimens were put into transmission electron microscopy (TEM) packet H4 and located in in-core weeper positions of the FFTF/MOTA from the beginning of cycle 4 and throughout the remaining operation of FFTF. The irradiation history for packet H4 was as follows:

MOTA 1B basket 2F-5 to 2.98×10^{22} n/cm² at an average temperature of 431°C.
MOTA 1C basket 2F-5 to 6.98×10^{22} n/cm² at 420°C.
MOTA 1D basket 2F-5 to 5.05×10^{22} n/cm² at 404°C.
MOTA 1E basket 2F-2 to 9.67×10^{22} n/cm² at 414°C.
MOTA 1F basket 2F-2 to 9.60×10^{22} n/cm² at 405°C.
MOTA 1G basket 2E-3 to 7.18×10^{22} n/cm² at 417°C.
MOTA 2B basket 3A-5 to 6.35×10^{22} n/cm² at 433°C.

Therefore, during that period, the specimens in packet H4 received a fluence of 4.78×10^{23} n/cm² at an average temperature of $418 \pm 11^\circ\text{C}$, hereafter reported as 420°C. The dose achieved, calculated from reference 13, which is based on dosimetry measurements from MOTA 1F, varies from 198 to 201 dpa, depending on alloy composition.

Microstructural examinations were performed on a JEOL JEM 1200EX scanning TEM operating at 120 kV and outfitted with a Tracor Northern energy dispersive x-ray fluorescence (EDX) detector and TN5500 computer and with a Gatan Electron energy loss spectrometer.

Results

Swelling

Results of density change measurements for simple ferritic alloy specimens irradiated at 420°C to 200 dpa are provided in Table 1. Table 1 contains dose estimates for each alloy based on conversions from reference 13. Table 1 also includes density change results for specimens irradiated to lower dose in FFTF/MOTA experiments that were started later in the reactor history.⁴ As shown from Table 1, swelling values are 7.4% or less. The highest swelling value, 7.4%, is found in Fe-9Cr, and the next highest swelling, 7.0%, is in Fe-6Cr. In comparison, Fe-12Cr swelled 2.8%, Fe-3Cr swelled 1.8%, and Fe-15Cr and Fe-18Cr swelled 1.3%. If these results are compared as a function of dose, assuming linear dependence between data points at two doses, the highest swelling rate is found for Fe-9Cr, 0.097%/dpa. This value is somewhat higher than the value previously estimated from EBR-II data of 0.06%/dpa,² where it was found that swelling levels in Fe-12Cr reached 4.72%, and swelling levels in Fe-9Cr reached 4.14%, at 1.9×10^{23} n/cm² or 91.2 dpa.

Microstructures

Microstructural examinations revealed that all specimen conditions had developed void swelling in the form of uniform arrays of voids. The amounts of void swelling and the relative void sizes were in proportion to the density changes measured. Voids were uniformly distributed, except in a few instances where denuded zones or reduced densities were found adjacent to grain boundaries, apparently associated with grain boundary migration. Also, examples were found in the Fe-18Cr alloy where larger voids were attached to needle shaped precipitates. Examples at low magnification are provided in Figure 1. Of particular note are the wide range of void sizes and the array of precipitates in the Fe-3Cr specimen shown in Figure 1a and the needle shaped precipitate with two large voids attached in the Fe-18Cr specimen in the lower right of Figure 1f. It may be noted that a broad void size distribution that includes small sizes is an indication that steady state swelling has not yet been achieved. The precipitates seen in Figure 1a are due to impurities in the Fe-3Cr alloy. Figures 1b, 1d and 1e display surface etching effects in the form of large pits; these are believed to be artifacts arising from preparation difficulties and were ignored.

Table 1. Swelling as measured by density change for simple ferritic alloy specimens examined in this study with comparison of specimens irradiated to lower dose.

CODE	Alloy	Dose (dpa)	Density Irradiated	Density Unirradiated	Swelling (%)
651A	Fe-3Cr	137.7	7.77042	7.8826	1.444
65H4	Fe-3Cr	197.6	7.74497	7.8826	1.777
661A	Fe-6Cr	138.2	7.65045	7.8185	2.197
66H4	Fe-6Cr	198.3	7.30779	7.8185	6.989
671A	Fe-9Cr	138.6	7.76389	7.7933	1.540
67H4	Fe-9Cr	199.0	7.2573	7.7933	7.386
6A1A	Fe-12Cr	139.1	7.71258	7.7596	0.610
6A1H4	Fe-12Cr	199.6	7.54631	7.7596	2.826
6N1A	Fe-15Cr	139.6	7.73933	7.7461	0.087
6N1H4	Fe-15Cr	200.4	7.64825	7.7461	1.279
6R1A	Fe-18Cr	140.1	7.68686	7.7233	0.474
6R1H4	Fe-18Cr	201.1	7.62793	7.7233	1.250

Contrast experiments have been performed on each irradiated specimen condition to provide a detailed understanding of the dislocation and void structures. Examples are shown for each of the alloys in Figures 2 through 7. In each case, a stereo pair using $\bar{g} = 200$ is shown in parts a) and b), dislocation contrast using $\bar{g} = 0\bar{1}1$ is shown in part c) and void contrast is shown in part d) for a foil near (011) orientation that is tilted near (012) for part b). Void shape analysis in the following description is based on figures included in reference 14.

Figure 2 shows examples of the dislocation and void structures in Fe-3Cr following irradiation at 420°C to 200 dpa. Figure 2a displays horizontal features in strong contrast consisting of $a[100]$ loops and dislocation line segments and more vertical features in lower contrast representing all the $\frac{a}{2}\langle 111 \rangle$ dislocations present superimposed on an array of voids in poor contrast. Examination of

the stereo model from Figures 2a and 2b reveals that the $a[100]$ loops are steeply inclined, that the $\frac{a}{2}\langle 111 \rangle$ dislocation segments form a network that sometimes includes $a[100]$ segments, and that the voids are uniformly distributed. Figure 2c shows a large number of loops that do not appear in Figure 2a that must therefore be of Burgers vector $a[010]$ and $a[001]$, distributed on planes approximately 45° to the plane of the micrograph. Therefore, from this sequence, it can be demonstrated that the dislocation structure in Fe-3Cr after 200 dpa at 420°C is predominantly $a\langle 100 \rangle$ in character, consisting primarily of loops, but $a\langle 100 \rangle$ and $\frac{a}{2}\langle 111 \rangle$ dislocation segments are present in the form of a dislocation network. Figure 2d can be used to demonstrate that voids vary widely in size, that void shape is equiaxed with the predominant shape equally truncated between cubes and dodecahedra (with a truncation factor between 0.4 and 0.5). Also, the precipitation due to an unexpectedly high impurity content is shown to be in the form of dark equiaxed particles (approximately 20 nm in diameter) that are uniformly distributed.

Figure 3 provides examples of the dislocation and void structures in Fe-6Cr following irradiation at 420°C to 200 dpa. Figure 3a reveals that the density of $a[001]$ dislocations is lower than that in Fe-3Cr, that most dislocations are line segments, and that $\frac{a}{2}\langle 111 \rangle$ dislocations are only line segments. The stereo model from Figures 3a and 3b shows a clear example of a dislocation node towards the upper left, but in most cases, the dislocation segments terminate at voids. Figure 3c shows a number of $a[100]$ and $a[010]$ loops and dislocation segments that cannot be seen in Figure 3a, but again segments tend to end at voids. Therefore, the dislocation structure is a mixture of $a\langle 100 \rangle$ and $\frac{a}{2}\langle 111 \rangle$ dislocations primarily in the form of a network with voids at the nodes. Figure 3d shows that voids in Fe-6Cr are larger than in Fe-3Cr and that voids are dodecahedral with (100) truncations (corresponding to a truncation factor between 0.6 and 0.8). However, larger voids are often elongated so that some of the facets are larger than others. Also, some of the smaller voids appear to be without significant truncation, but other small voids are clearly faceted. Therefore, considerable variation in void shape exists.

Figure 4 gives examples of the dislocation and void structures in Fe-9Cr following irradiation at 420°C to 200 dpa. Figure 4a again reveals the presence of both $a[001]$ and $\frac{a}{2}\langle 111 \rangle$ Burgers vectors. The stereo model from Figures 4a and 4b shows that few $a[001]$ loops are present, and most of those are connected to the dislocation network. Again, dislocation segments often terminate at voids. The images are confused by surface pits, and voids that are connected to the surface, often with a mottled appearance. Figure 4c shows a number of incomplete $a[100]$ and $a[010]$ loops and dislocation segments that cannot be seen in Figure 3a. Therefore, the dislocation structure is again a mixture of $a\langle 100 \rangle$ and $\frac{a}{2}\langle 111 \rangle$ dislocations in the form of a network but with several partial loops and with voids at some of the nodes. Figure 4d reveals that the truncation of the dodecahedral voids is even less than in Fe-6Cr (with a truncation factor of 0.8 to 1.0) with considerable variation in void aspect ratio and some variation in facet definition.

Figure 5 provides examples of the dislocation and void structures in Fe-12Cr following irradiation at 420°C to 200 dpa. Figure 5a shows a somewhat different dislocation structure, consisting of both small $a[001]$ loops and large $a[001]$ segments as well as large $\frac{a}{2}\langle 111 \rangle$ segments. The loops are as small as 15 nm in diameter with many features in strong contrast that are even smaller. The stereo model from Figures 5a and 5b verifies that the $a[001]$ loops are steeply inclined, and that the density of small features is much higher than in alloys with lower chromium content. Figure 5c shows the $a[100]$ and $a[010]$ loops and line segments not visible in 5a and 5b, but also reveals that the small features remain in stronger contrast than the dislocation structure. Therefore, the strong contrast is due to precipitation, likely from chromium rich α' formation. Figure 4d reveals that void shapes are dodecahedral with slight cubic truncation (with a truncation factor 0.7 to 0.9), and larger voids are often elongated. A contrast due to precipitation can be identified, indicating that particles are on the order of 5 nm and larger in diameter.

Figure 1. Void swelling at low magnification for a) Fe-3Cr, b) Fe-6Cr, c) Fe-9Cr, d) Fe-12Cr, e) Fe-15Cr, and f) Fe-3Cr following irradiation at 420°C to 200 dpa.

Figure 2. Microstructures in Fe-3Cr irradiated at 420°C to 200 dpa as a stereo pair using $g = 200$ in a) and b), using $g = 011$ in c) and void contrast in d) for a foil near (011) orientation that is tilted near (012) for part b)

Figure 3. Microstructures in Fe-6Cr irradiated at 420°C to 200 dpa as a stereo pair using $g = 200$ in a) and b), using $g = 011$ in c) and void contrast in d) for a foil near (011) orientation that is tilted near (012) for part b)

Figure 4. Microstructures in Fe-9Cr irradiated at 420°C to 200 dpa as a stereo pair using $g = 200$ in a) and b), using $g = 011$ in c) and void contrast in d) for a foil near (011) orientation that is tilted near (012) for part b)

Figure 5. Microstructures in Fe-12Cr irradiated at 420°C to 200 dpa as a stereo pair using $g = 200$ in a) and b), using $g = 011$ in c) and void contrast in d) for a foil near (011) orientation that is tilted near (012) for part b)

Figure 6. Microstructures in Fe-15Cr irradiated at 420°C to 200 dpa as a stereo pair using $\bar{g} = 200$ in a) and b), using $\bar{g} = 011$ in c) and void contrast in d) for a foil near (011) orientation that is tilted near (012) for part b)

Figure 7. Microstructures in Fe-18Cr irradiated at 420°C to 200 dpa as a stereo pair using $g = 200$ in a) and b), using $g = 011$ in c) and void contrast in d) for a foil near (011) orientation that is tilted near (012) for part b)

Figure 6 gives examples of the dislocation and void structures in Fe-15Cr following irradiation at 420°C to 200 dpa. Figure 6a shows again a somewhat different dislocation structure, consisting of small $a[001]$ loops and large $\frac{a}{2}\langle 111 \rangle$ segments; large $a[001]$ segments appear to be absent. The loops range in size from 7 to 20 nm in diameter, but again with many features in strong contrast that are even smaller. The stereo model from Figures 6a and 6b verifies that these features are evenly distributed through the thickness of the foil. Figure 6c reveals the dislocation line segments in better resolution, and several examples can be found of small loops at a different inclination than found previously, probably of type $\frac{a}{2}\langle 111 \rangle$. Therefore, Fe-15Cr contains a higher fraction of $\frac{a}{2}\langle 111 \rangle$ dislocations, both as line segments and small loops, than was found in alloys with lower chromium contents. Figure 6d shows that voids are dodecahedra with moderate (100) truncation (corresponding to truncation factors from 0.4 to 0.7) and that precipitation appears to be more dense, as would be expected for chromium rich precipitates.

Figure 7 provides examples of the dislocation and void structures in Fe-18Cr following irradiation at 420°C to 200 dpa. This sequence is perhaps the most difficult to understand. However, Figure 7a again shows a dislocation structure, consisting of small $a[001]$ loops and larger $a[001]$ and $\frac{a}{2}\langle 111 \rangle$ segments. Many precipitates can also be seen. The stereo model from Figures 7a and 7b verifies that these features are evenly distributed through the thickness of the foil. Figure 7c shows configurations typical of $a[100]$ and $a[010]$ dislocations, but a few examples of small $\frac{a}{2}\langle 111 \rangle$ loops can also be seen. Therefore, the dislocation structure in Fe-18Cr appears to contain predominantly $a\langle 100 \rangle$ loops but with dislocation segments of both $a\langle 100 \rangle$ and $\frac{a}{2}\langle 111 \rangle$. From Figure 7d, the void shape is found to contain greater cubic truncation than seen previously (with truncation factors between 0.3 and 0.6). Precipitation is more extensive, with particle sizes of about 8 nm.

Microstructural Analysis

The void and dislocation microstructures have been analyzed quantitatively to determine void swelling, void density, void size distribution, and dislocation density. Table 2 summarizes the information. Void swelling is measured based on the average truncated void shape with input consisting of measurements in a $\langle 110 \rangle$ direction as recommended in reference 14. Mean void sizes and void distributions are reported based on the equivalent sphere diameter for each truncated void.

Dislocation densities are based on the $\frac{a}{2}\langle 111 \rangle$ and $a\langle 100 \rangle$ dislocations visible using $g = 200$ contrast, but with the $a\langle 100 \rangle$ multiplied by a factor of three to correct for the invisibility of $a[100]$ and $a[010]$ Burgers vectors. Figure 8 presents the void size distributions (reported as equivalent sphere diameters). Based on Figure 8, it is apparent that the enhanced density change as reported in Table 1 is a consequence of enhanced void growth — the larger the largest voids, the higher the density change.

Discussion

The results obtained in this study can be used to provide the basis for discussions on two topics: 1) the reason for the high swelling resistance of ferritic alloys and 2) the cause of differences in swelling response from irradiation in EBR-II and FFTF/MOTA. First, the present microstructural results of importance will be summarized. Then, discussion will be provided relating those and previous results on the Fe-Cr alloy series to the available explanations for swelling resistance. Finally, differences in response in different reactors will be considered.

Table 2. Summary of quantitative microstructural observations.

Code	Alloy	Dose (dpa)	Swelling (%)	Mean Void Size (nm)	Void Density (#/cm ³)	Truncation factor	Dislocation density (cm/cm ³)
65H4	Fe-3Cr	197.6	1.20	19.1	1.69×10^{15}	0.4-0.5	$\frac{a}{2} \langle 111 \rangle 1.25 \times 10^{10}$ $a \langle 100 \rangle 1.8 \times 10^{10}$
66H4	Fe-6Cr	198.3	6.07	47.7	6.8×10^{14}	0.6-0.8	$\frac{a}{2} \langle 111 \rangle 7.2 \times 10^9$ $a \langle 100 \rangle 2.8 \times 10^{10}$
67H4	Fe-9Cr	199.0	9.24	57.3	6.1×10^{14}	0.8-1.0	$\frac{a}{2} \langle 111 \rangle 1.84 \times 10^{10}$ $a \langle 100 \rangle 9.8 \times 10^{10}$
6AH4	Fe-12Cr	199.6	3.76	40.8	7.0×10^{14}	0.7-0.9	$\frac{a}{2} \langle 111 \rangle 8.3 \times 10^9$ $a \langle 100 \rangle 4.1 \times 10^{10}$
6NH4	Fe-15Cr	200.4	2.29	24.9	1.85×10^{15}	0.4-0.7	$\frac{a}{2} \langle 111 \rangle 4.7 \times 10^9$ $a \langle 100 \rangle 2.7 \times 10^{10}$
6RH4	Fe-18Cr	201.1	2.54	19.0	4.0×10^{15}	0.3-0.6	$\frac{a}{2} \langle 111 \rangle 1.7 \times 10^{10}$ $a \langle 100 \rangle 8.1 \times 10^{10}$

Explanation for low swelling in ferritic alloys

The present results provide confirmation from previous studies that void swelling develops in ferritic alloys in much the same ways as in other crystal structures. Voids can nucleate uniformly along with two types of dislocation loops, the voids can grow to sizes exceeding 200 nm, and the dislocation structure can evolve into a network. As had been shown in previous studies, two types of Burgers vectors developed, $a \langle 100 \rangle$ and $\frac{a}{2} \langle 111 \rangle$. The balance between these two Burgers vectors varies with composition, but large changes in this balance do not appear to significantly reduce swelling, an observation also made in a companion study.¹⁵ Also, it is shown that precipitation reduces swelling, for example in Fe-3Cr and in Fe-12Cr to Fe-18Cr. It may well be significant that dislocation populations in α' forming alloys include smaller loops along with a well developed dislocation network. For example, the enhanced dislocation densities that result from the small loop population may be responsible for reduced swelling.

Many of the models that have been developed to explain swelling resistance in ferritic alloys are really arguments concerning the incubation dose for swelling. It must be remembered that the modeling of swelling involves two stages, an incubation period followed by a steady state swelling period. The incubation period includes the void nucleation phase, and the steady state response only applies after the microstructure of voids and dislocations stabilizes. For the purposes of this discussion, those models that argue that ferritic alloys should have longer incubation periods, such as those controlled by solute segregation or those that keep the incubation period indefinitely long, will be ignored. This includes models by Little and Stow regarding segregation control,¹⁰ by Little, Bullough, and Wood based on point defect bias between the $a \langle 100 \rangle$ and $\frac{a}{2} \langle 111 \rangle$ Burgers vectors at the exclusion of a bias to voids,⁹ and by Odette and others based on low helium production, which delays void nucleation.⁵

The models that remain under consideration are therefore 1) an inherent crystal structure effect,⁸ 2) a sink strength effect,^{11,12} 3) a microstructural effect due to precipitation or lath structure,⁶ 4) a bias effect on voids evidenced by changes in void shape due to segregation,³ and 5) reduced dislocation mobility due to the presence of $a \langle 100 \rangle$ sessile dislocations.¹

The present results have provided the highest swelling found in neutron irradiated ferritic alloys, producing 7.4% at 200 dpa with an estimated rate of 0.1%/dpa in a simple Fe-9Cr alloy, which is free of precipitation and subgrain lath structure. This swelling rate is still very low in comparison to 1%/dpa in austenitic alloys¹⁶ and 2%/dpa in some vanadium alloys.¹⁷ Considering the above models in reverse order, it can be noted that the present work has shown that the dislocation network eventually evolves to one where nodes between dislocations with different Burgers vectors become the voids themselves. In other words, such nodes do not actually exist because individual dislocation segments terminate at void surfaces. Therefore, the junction of a $\langle 100 \rangle$ and $\frac{a}{2} \langle 111 \rangle$ Burgers vectors, although sessile, cannot reduce the climb of the dislocation network, and ferritic alloys behave just like other systems where the voids eventually provide the sites for dislocation network junctions. The present work also shows moderate variation in void shape from one alloy to another with dodecahedral shapes dominating, whereas, at low dose, the void shapes were found to be octahedra, with (111) facets dominating,³ so that a large change in void shape has developed with increasing dose. Therefore, large changes in shape have occurred with increasing dose, but at high dose, the void shape is relatively constant. It is true that for the highest swelling rate condition, Fe-9Cr, the highest truncation factor is found, corresponding to voids of almost perfect dodecahedral shape. However, the variations in shape do not seem to correlate directly with a reduction in swelling response, and it appears unreasonable to expect that the flux of vacancies to a void surface will be significantly reduced by a segregated layer. (Instead, the consequence would be to increase recombination, in effect shifting the peak swelling temperature.) A model based on precipitation or lath dislocation substructure cannot apply to the present simple alloys with large grain size and negligible substructure before irradiation.

The sink strength ratio model is worthy of more careful consideration. Table 3 has been prepared to summarize the sink strength ratio information that is available for Fe-Cr binary alloys in the present series of irradiations. Table 3 includes estimates for swelling rate based on density change measurements when they are available, but including TEM measurements, where the rate is defined assuming zero swelling at 0 dpa. Microstructural information for mean void diameter, void density, and total dislocation density (the sum of the $\langle 100 \rangle$ and $\frac{a}{2} \langle 111 \rangle$ dislocation populations) is also given. Finally, the sink strength ratio, Q , is calculated based on the expression from reference 12:

$$Q = L/4\pi \frac{\bar{d}}{2} N$$

where L is the total dislocation density, $\bar{d}$ is the mean void diameter, and N is the void density. The values for sink strength in Table 3 are found to vary from 0.4 to 10, but the majority fall in the range 0.8 to 6. Therefore, most of these microstructures correspond to sink strength ratios close to 1. It may be noted that the first four values for Q in Table 3 do not quite agree with those calculated in reference 12 where the values reported were between 1.5 and 5.4.

The argument has been made that swelling resistance in ferritic alloys is a consequence of the fact that the maximum cavity growth rate occurs when $Q \approx 1$, and that most microstructural measurements on ferritic alloys indicated that the cavity number density is low, leading to a high value for Q .^{11,12} The results shown in Table 3 indicate that at least for the simple Fe-Cr alloys in the present experiment, Q values are indeed ≈ 1 , and yet, swelling rates remain an order of magnitude below those of the higher swelling alloy classes. Therefore, ferritic alloys are indeed able to generate microstructural conditions conducive for high swelling response, but swelling rates remain low.

(a) Fe-3Cr

(b) Fe-6Cr

(c) Fe-9Cr

(d) Fe-12Cr

(e) Fe-15Cr

(f) Fe-18Cr

Figure 8. Void size distributions for Fe-Cr specimens irradiate at 420°C to 200 dpa.

Table 3. Evaluation of Sink Strength Ratio Values for Fe-Cr alloys for which steady state swelling rates can be estimated.

Code	Alloy	Dose (dpa)	Swelling Rate (%/dpa)	Mean Void Size (nm)	Void Density, N (#/m ³)	Total Dislocation Density, L (m/m ³)	Sink Strength Ratio, Q
6507 ¹	Fe-3Cr	21	0.02	26.1	8.0×10^{19}	1.0×10^{13}	0.8
6607 ¹	Fe-6Cr	21	0.04	33.8	2.8×10^{20}	2.1×10^{13}	0.4
6707 ¹	Fe-9Cr	21	0.05	33.9	2.2×10^{20}	7.0×10^{13}	1.5
6A07 ¹	Fe-12Cr	21	0.06	30.3	4.2×10^{20}	6.8×10^{13}	0.9
65FT ¹	Fe-3Cr	15	0.0004	8.6	2.0×10^{20}	1.1×10^{14}	10.3
66FT ¹	Fe-6Cr	15	0.01	12.2	1.7×10^{20}	1.2×10^{14}	0.9
67FT ¹	Fe-9Cr	15	0.02	16.9	1.2×10^{21}	2.0×10^{14}	1.5
6AFT ¹	Fe-12Cr	15	0.01	10.8	1.8×10^{21}	4.7×10^{14}	3.8
6NFT ¹	Fe-15Cr	15	0.004	8.7	2.3×10^{21}	2.2×10^{14}	1.7
6RFT ¹	Fe-18Cr	15	0.006	8.9	3.1×10^{21}	2.8×10^{14}	1.6
65H4	Fe-3Cr	197.6	0.006	20.1	3.1×10^{21}	3.1×10^{14}	0.8
66H4	Fe-6Cr	198.3	0.080	47.7	6.8×10^{20}	3.5×10^{14}	1.7
67H4	Fe-9Cr	199.0	0.097	57.3	6.1×10^{20}	1.08×10^{15}	6.0
6AH4	Fe-12Cr	199.6	0.037	40.8	7.0×10^{20}	4.9×10^{14}	2.7
6NH4	Fe-15Cr	200.4	0.020	24.9	1.9×10^{21}	3.2×10^{14}	1.1
6RH4	Fe-18Cr	201.1	0.013	19.0	4.0×10^{21}	9.8×10^{14}	2.1

Finally, a model based on the inherent atomic packing of the body centered cubic lattice is appealing. The model predicts that the bias for vacancies to accumulate at voids is due to the difference in bias factors for dislocations and voids, and that difference is greater in austenitic than ferritic alloys.¹ The present results provide the argument that swelling appears to develop normally in simple ferritic alloys, but the swelling rates obtained remain low, in agreement with model predictions. However, the model comes into question when trying to explain why alloys of V-5Fe, which are also body centered cubic, can develop swelling rates in excess of 2%/dpa.¹⁷

Therefore, it must be concluded that it is not yet clear why swelling rates in ferritic alloys are low.

Different response in different reactors

When the swelling results for Fe-Cr simple binary are compared following irradiation in different reactors, significant differences in response can be identified. Following irradiation in EBR-II to 19 dpa at 425°C, swelling as high as 0.68% was found in Fe-9Cr and 0.63% in Fe-12Cr based on void measurements.¹ Following irradiation in EBR-II to 90 dpa at 425°C, swelling as high as 4.14% was found in Fe-9Cr and 4.72% in Fe-12Cr based on density change measurements.² In comparison, following irradiation in FFTF/MOTA to 139 dpa at 420°C, swelling of only 1.54% was found in Fe-9Cr and 0.61% in Fe-12Cr based on density change measurements, as noted in Table 1.

Also, further irradiation in FFTF/MOTA to 199 dpa resulted in a swelling rate for Fe-9Cr that was similar to that found in EBR-II, but with continued lower swelling in Fe-12Cr compared to Fe-9Cr, 2.83% versus 7.39%, based on density change measurements. Therefore, irradiation in FFTF/MOTA at 420°C produces swelling response with a higher incubation and produces significantly less swelling in Fe-12Cr than Fe-9Cr.

Comparison of the microstructures for Fe-12Cr in EBR-II and FFTF/MOTA shows further differences. The α' development is much more extensive, and the dislocation structures are more complicated, for the FFTF/MOTA case. The present results indicated that precipitation produces the more complex dislocation evolution and leads to reduced swelling. Therefore, at least part of the difference is likely due to enhanced precipitation of α' in FFTF/MOTA.

As FFTF and EBR-II are similar reactors with very similar neutron energy spectrums, it is unlikely that differences are a consequence of different dose or transmutation responses. A more likely explanation is an uncertainty in irradiation temperature or differences in reactor temperature cycling history. The α' precipitation process is temperature dependent such that more precipitation is produced at lower temperatures. Therefore, the differences in response between Fe-9Cr and Fe-12Cr could be explained by assuming that the EBR-II temperature was underestimated, by 30°C or more. Note that temperatures in FFTF/MOTA were monitored by thermocouples. However, the EBR-II irradiation employed a weeper design at 425°C without thermocouples, and temperatures were usually estimated based on reactor sodium temperature measurements well away from the irradiation test assemblies. Nonetheless, an error as large as 30°C is unexpected.

Although the difference in response as a function of composition can be explained by temperature uncertainty, the difference in swelling incubation cannot. Swelling incubation is a measure of how rapidly a steady state microstructure can develop. That microstructure is generated by nucleation of dislocation loops and voids and then growth and evolution of those features into steady state configurations. The nucleation process will govern that evolution, and nucleation is favored at lower temperatures. Therefore, any operation below the irradiation temperature could promote nucleation and accelerate the approach to steady state swelling. It can be anticipated that the temperatures were maintained more constant during operation in FFTF/MOTA than in EBR-II. However, startup and shutdown operations were more rapid in EBR-II due to required intermediate power levels holds in FFTF. Such intermediate power level holds would provide slightly reduced weeper temperatures, but the differences are not expected to cause significant nucleation of new microstructure. Therefore, it is not yet clear that specific differences in reactor operations can be identified to explain the differences found in swelling incubation response in the different reactors. The irradiation of Fe-Cr simple ferritic alloys is being rerun in the most recent EBR-II irradiation. Those specimens will be examined to check for similar response.

CONCLUSIONS

Density change measurements and microstructural examinations have been performed on a series of Fe-Cr alloys following irradiation in the FFTF/MOTA at 420°C to 200 dpa, and the results have been compared with results on specimens irradiated to lower dose and on similar specimens irradiated in EBR-II. The highest swelling of 7.4%, corresponding to a swelling rate of 0.097%/dpa, is found in Fe-9Cr, but swelling representative of steady state swelling is found in all alloys except Fe-3Cr. The microstructures in all conditions have been quantitatively analyzed, and estimates for the sink density ratios have been shown to be approximately 1. Comparison with previous experiments reveals that response is different in different reactors such that swelling is relatively lower in Fe-12Cr and swelling incubation is larger for irradiation in FFTF/MOTA. Explanations

are proposed that suggest that EBR-II weeper temperatures were higher than expected, but no explanation is available to explain swelling incubation response. Comparison of microstructural features with those found at lower doses provided the unexpected result that void shapes shift from octahedra to dodecahedra as dose increases. Examination of proposed theories for the low swelling rates found in ferritic alloys provided no completely acceptable explanation.

FUTURE WORK

This work will be continued when appropriate specimens become available.

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