Microstructural changes in a neutron-irradiated Fe-6at.%Cr alloy

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Abstract

The microstructural and chemical changes in a Fe-6 at.% Cr binary model alloy neutron irradiated to 1.82 dpa at 290 °C were investigated using atom probe tomography. After irradiation, Si and Cr are found segregated to dislocation loops, which were analyzed in terms of number density, size, and habit plane. Grain boundary chemistry was quantitatively compared between the as-received and the neutron irradiated alloys. The results are discussed in the context of equilibrium segregation, radiation-enhanced diffusion, and/or radiation induced segregation.

Keywords: Fe-Cr, neutron irradiation, atom probe tomography

1. Introduction

High chromium ferritic/martensitic (F/M) steels are one of the promising structural materials for nuclear reactors [1, 2], due to their low swelling behavior, low initial ductile-brittle transition temperature and good corrosion resistance. However, the next generation of nuclear reactors requires structural materials that are able to sustain very high irradiation doses and extended lifetimes. Therefore, accurate microstructural evolution and mechanical property predictions over a component lifetime are essential. In particular, radiation induced elemental segregation or depletion, defect clustering and second phase precipitation are of primary concern.

Past studies have shown that a number of such microstructural changes take place during irradiation. Those include Si segregation to dislocations [4-6], Ni/Si clustering [4, 7-9], Cr depletion to grain boundary [10-13], Cr segregation to grain boundary [7, 14-19], Si and P clustering [3] segregation to grain boundaries [4]. There is also some evidence that resistance of F/M steels to irradiation embrittlement and swelling depends on Cr content [20-24]. The multiplicity of interactions between alloying elements and impurities present in FM steels makes it difficult to isolate the behavior of any single element. For instance, carbon [25] or hafnium [14] have been shown to affect Cr behavior at grain boundaries under irradiation. Thus studies of nominally simple binary Fe-Cr model alloys are needed to help isolate the effects of this prime alloying element in multicomponent F/M alloys.

Cr-mediated irradiated phenomena are complex. The high number of point defects can lead to accelerated phase decomposition or solute segregation, that would otherwise be too slow to be observed under thermal conditions, Cr being a very slow diffusing species in α -Fe [26]. On the other hand, the biased interactions between Cr and vacancies and interstitials could favor non-equilibrium configurations, such as radiation-induced segregation (RIS) or depletion at interfaces, which would not be observed under thermal conditions. Separating these two irradiation effects can be difficult, and the literature data concerning Cr behavior under irradiation is still indecisive, despite a number of previous studies. Lu et al. [27] provided a comprehensive review of published results revealing either Cr depletion or segregation with no obvious trends with irradiation particle type, alloy composition, or temperature. More recently, Cr depletion was observed in ion implanted Fe-Cr alloys at temperature above 300°C [19, 28]. Assuming a RIS mechanism, Wharry et al. proposed that Cr should segregates at low

temperature where diffusion is dominated by vacancy exchange while depletion due to interstitial binding should dominate at high temperature [29]. Recent molecular dynamics simulations by Pechenkin et al. [30] showed that radiation induced Cr segregation is possible and would result from a competition between interstitial and vacancy migration. The ability of grain boundaries to act as a sink for point defects depends on their structures and therefore on misorientation. As was previously observed in austenitic steels [31], Hu et al. found that in an ion-irradiated ferritic Fe-14Cr alloy low angle and $\Sigma 3$ boundaries are much less affected by irradiation-induced depletion [28] compared to random large angle boundaries. Fields et al. reported the same trends for irradiation-induced Cr segregation in neutron and proton ion irradiated F/M steels [32]. Yet, whether segregation is a result of thermodynamic equilibrium or an irradiation-induced non-equilibrium phenomenon remains an open question.

In addition to the possible RIS behavior, the presence of carbides and nitrides at grain boundaries complicates the observations. The growth of carbides in thermally treated steels results in Cr depletion at grain boundaries, which is also known as “sensitization”. Carbide formation under irradiation may have a similar effect that could be confused for radiation-induced depletion.

In light of these considerations, the present work focuses on the detailed microstructural changes occurring in matrix and at grain boundaries in a Fe-6 at.%Cr model alloy. Atom probe tomography (APT) is used for both as-received and neutron-irradiated materials for proper assessment of the radiation effects. Based on previous work on a series of neutron irradiated Fe-Cr alloys, α' precipitation is expected for Cr contents above 9 at.% [33]. Therefore the present work focused on a single phase under-saturated Fe-Cr composition while a companion article reports on the effect of neutron irradiation in an over-saturated Fe-15Cr model alloy exhibiting α' precipitation.

2. Materials and experimental methods

An Fe-6 at.%Cr alloy was annealed under an argon atmosphere at 950°C for 15 minutes followed by air cooling, then stress relieved at 750°C again for 1 hour, followed by air cooling.

This produced a simple, fully-recovered/recrystallized ferrite microstructure with a range of relatively coarse grain sizes. Irradiation was carried out in the Advance Test Reactor (ATR) at Idaho National Laboratory at a neutron fluence ($E > 1$ MeV) of 1.1×10^{21} n/cm² at 563 ± 15 K and to a damage level of 1.8 displacements per atom (dpa). Nominal neutron flux and dpa rate are 2.3×10^{14} n/cm²/s and 3.4×10^{-7} dpa/s respectively. The calculated fluence produced a dose of 1.82 displacements per atom (dpa) based on the MPMC neutronic model of the ATR and the ASTM Displacement Cross Sections Standard (the as run irradiation conditions were provided by J. Nielsen at INL). The nominal neutron flux and dpa rate are 2.3×10^{14} n/cm²/s and 3.4×10^{-7} dpa/s, respectively.

The surface of the as-received and neutron-irradiated samples were prepared by polishing for scanning electron microscopy (SEM) and electron back-scattered diffraction (EBSD) observations. The approximate grain size is between 5 and 15 μ m. Atom probe tomography specimens with and without grain boundary (matrix) were prepared by focused ion beam milling and a standard liftout process [34] using a dual beam SEM Quanta 3D 200i.

APT specimens cooled to a base temperature of 55 K were analyzed in voltage mode (with a pulse fraction of 20%) or laser mode (pulse energy in the range of 30-60 pJ/pulse), a pulse repetition rate of 200 KHz, and a detection rate of 0.003-0.005 atoms/pulse. Data were reconstructed and analyzed using the Cameca Visualization and Analysis Software (IVAS 3.6.6). The average evaporation field (33 V/nm), and the detector efficiency (0.36) were used for reconstruction of the dataset, while the image compression factor was adjusted to fit either the {100} or {110} plane spacing calculated from spatial distribution maps (SDM) [35]. The full crystallographic orientations of the datasets were determined from the pole patterns. In addition, a number of APT dataset contained solute segregated dislocation loops. To determine the orientation of the dislocation loops, small plates (5 x 5 x 1 nm) containing a dislocation loop were analyzed via 3D Hough transformations to determine the crystallographic direction normal to the plate. More details on the use of 3D Hough transformations for extracting structural information from 3D point data can be found in [36].

3. Results

3.1 Microstructural changes in the matrix

Specimens in the as-received and neutron-irradiated conditions were analyzed in APT under identical condition in voltage mode to determine the overall alloy composition away from grain boundaries. The measured compositions are provided in **Table 1**. Values obtained are in good agreement with the nominal composition and showed traces of impurities (P, Si, Mn, V and C).

Visual inspection of the 3-D reconstructed data from the as-received condition shows a uniform distribution of Cr in the matrix, as well as all the impurity elements (**Fig. 1(a)**). Note that the usual evaporation artifacts associated with surface migration of Si, Ni and P were observed with higher concentration of these solute or impurity elements measured at zone lines and poles [38].

After neutron irradiation, Cr and Si decorated dislocation loops and clusters are observed in the matrix as illustrated in **Fig. 1(b)**. The combined number density of segregated loops and solute clusters is $2.9 \pm 0.7 \times 10^{22} \text{ m}^{-3}$. The observed spatial distribution of Cr and Si-enriched dislocation loops within the APT datasets suggest a defect-free region about 20 to 25 nm wide on either side of grain boundaries (as illustrated in **Fig. 1(c)**). Two types of dislocation loops are observed based on the Si and Cr spatial distributions. The smaller loops (distinct from clusters by their flattened appearance) exhibit Cr enrichment at their periphery with Si atoms located inside the loop, as shown in **Fig. 2(a,b)**. For the larger loops, the spatial positions of Si and Cr atoms are the same, i.e. segregated on the outside of the dislocation line (**Fig. 2(c,d)**). The sizes of these visible dislocation loops are determined from the spatial distributions of Si and Cr. The size distribution shown in **Fig. 2(e)** highlights the two populations of loops: smaller loops with diameters 8 to 10 nm and larger loops with diameters about 18 to 20 nm. Using the crystallographic orientation of the APT data reconstructions, the habit planes of the dislocation loops were indexed using the direction of the normal to the loop habit planes. These are distributed along a zone line between the $\langle 110 \rangle$ and $\langle 112 \rangle$ directions, as shown in **Fig. 2(f)**.

3.2 Grain boundary chemistry

In order to quantify the role of grain boundary misorientation, analyses were carried out on specific grain boundaries: a low angle boundary with a misorientation angle smaller than 14° , a $\Sigma 3$, a $\Sigma 5$, and a random grain boundary with a misorientation angle greater than 15° . The latter was also analyzed in the as-received condition. Concentration profiles across grain boundaries are obtained by placing a cylinder of radius 15 to 25 nm perpendicular to the grain boundary, which is divided into small slices of 0.5 nm thickness to calculate local concentrations along the cylinder length. Poles and zone lines are avoided for grain boundary profile measurement to avoid evaporation artifacts.

The comparison between the Cr behavior for high angle random boundaries in the as-received and neutron-irradiated conditions is shown in **Fig. 3(a)**. Cr exhibits a flat concentration profile in the as-received condition while it is segregated to the grain boundary in the neutron-irradiated condition. In the neutron-irradiated state, Cr is also segregated to other grain boundaries of different orientations with comparable segregation levels, as shown in **Fig. 4(a)**. Note that in most cases widths and heights of segregation profiles cannot be used as quantitative measurements of segregation behaviors due to inherent technique artefacts such as beam spreading in TEM [38] or trajectory aberrations in APT [40]. We are rather providing Gibbsian excess values [40], which are 10.5, 7.6, 10.5, and 10.8 atoms/nm² for the low angle, the $\Sigma 3$, the $\Sigma 5$, and the large angle random boundary respectively.

Carbon is the main impurity element observed at a high angle boundary of the as-received alloy, while no significant phosphorus and little silicon are observed (**Fig. 3**). Comparatively, segregation at a large angle grain boundary of the irradiated alloy is enhanced for Si and P but slightly less for C. The segregation levels observed in the irradiated alloy appear to depend on grain boundary orientation: no or little segregation is observed for C and P at the $\Sigma 3$ and low angle boundaries while clear segregation peaks are observed for the $\Sigma 5$ and large angle boundary (**Fig. 4**).

One of the analyses from the low angle grain boundary showed the presence of Cr and Si segregated along line features that are interpreted as dislocation lines, as shown in **Fig. 5(a)**. A

concentration profile for Cr and Si within the plane of this grain boundary is shown in **Fig. 5(b)**. From the measured distance between the dislocation lines ($d \sim 9\text{nm}$), the misorientation angle for the grain boundary is about 3° (using a value of the Burgers vector of 0.248nm).

4. Discussion

The evolution of microstructures under neutron irradiation is a result of competing mechanisms driven by thermodynamics and kinetics as modified by the high concentration of excess point defects generated by displacement damage (dpa). Atomic displacements form self-interstitial and vacancy defects, which can recombine, cluster in the form of extended defects like dislocation loops and nanovoids, or annihilate at defect sinks such as pre-existing dislocations, grain boundaries and other interfaces. The higher concentrations of mobile point defects, compared to normal thermal conditions, lead to radiation-enhanced diffusion (RED) and phase equilibrium at much shorter times than those normally expected at low temperatures. In addition to radiation enhanced precipitation (REP), preferential interactions of the migrating defects with alloying elements and impurities can lead to non-equilibrium microstructures, such as radiation induced segregation (RIS) to point defect sinks and radiation induced precipitation (RIP). Ideally the comparison between thermally treated and irradiated samples would clarify the controlling mechanism. In the case of the Fe-Cr system, however, the very slow kinetics of Cr diffusion under thermal conditions complicates the direct comparison.

The observation of Cr segregation in the neutron-irradiated alloy is consistent with either, or both, RED and RIS mechanisms. A RIS mechanism would imply a preferential Cr diffusion by interstitials and/or binding to vacancies in order to lead to the observed Cr segregation to grain boundaries during irradiation. However, accelerated diffusion kinetics under irradiation can also lead to Cr segregation at grain boundaries as would be expected under thermal conditions at longer times [41] and, therefore, a RED mechanism cannot be excluded. Indeed, the previously reported segregation enthalpy (-2895 J/mol [43]) would predict segregation levels that are comparable to those measured here. Assuming a RIS mechanism, the slightly lower Gibbsian excess of the $\Sigma 3$ grain boundary can be explained by its smaller free volume and therefore lower sink strength, as previously reported for austenitic steels [42]. On the other hand, in the case of

an accelerated kinetics RED mechanism, the lesser segregation at the $\Sigma 3$ boundary may be consistent with its inherent lower interfacial energy and, especially, the Cr segregation enthalpy.

The same question pertains to the responsible mechanism controlling the segregation behavior of the various impurity elements. Notably, P, Si, and C that are all expected to strongly thermally segregate to grain boundaries, given their large segregation enthalpy of order 30, 15 and 50 kJ/mol respectively [43]. Indeed there is a large literature on thermal segregation of these elements [43], including the corresponding consequences to phenomena such as temper embrittlement. Therefore thermal segregation calculations using a simple McLean type model are approximately consistent with observed grain boundary solute enrichments in the irradiated condition; indeed, in the case of P the simple models predict even higher concentrations than observed in this study [44]. However, thermal segregation depends on kinetics, as well as thermodynamics, hence on the entire alloy time-temperature history and solute diffusion properties [44-46]; and only interstitial C is expected to be fully equilibrated. Further, thermal non-equilibrium segregation (TNES), due to flow of excess quenched-in vacancies to boundaries, may also occur [47] along with other complications not treated in simple pure binary alloy Langmuir-McLean type models [43]. Thus further discussion of RIS versus RED segregation mechanisms for Cr, P and Si is beyond the scope of this paper. However, our hypothesis is that both RIS and RED are likely to be operative, and both must also be considered in a balanced and experimentally informed way. Insight on equilibrium (RED) versus non-equilibrium (RIS) mechanisms are being pursued by long-term thermal annealing experiments on the irradiated alloys over times that are sufficient to approach thermal equilibrium conditions.

As previously discussed in Refs. [48,49], dislocations can only be observed by APT if decorated by solute atoms. Therefore the analysis of dislocations using APT relies on the assumption all are segregation sites for solute elements making them visible. The segregation is also assumed to lock dislocations in place preventing them from moving under the stresses created during field evaporation. These assumptions are supported by the fact that the measured number density of dislocations is comparable to or larger than the numbers reported in the literature. Using TEM, Matijasevic et al. [23] reported a dislocation density of $2.1 \times 10^{21}/\text{m}^3$ with an average loop diameter of 10nm for a Fe-5Cr irradiated to similar conditions (300°C, 1.7dpa).

Two types of dislocation loops have generally been reported in neutron irradiated Fe and Fe-Cr alloys: $\langle 100 \rangle$ loops lying on $\{100\}$ planes and $\frac{1}{2}\langle 111 \rangle$ loops, for which different habit planes have been observed ($\{111\}$ [49], $\{110\}$ [50], and $\{112\}$ [51]). The measured dislocation loops habit planes would suggest that $\frac{1}{2}\langle 111 \rangle$ loops are formed. This observation is in agreement with the temperature dependence reported by Jenkins et al. [52] for ion implanted Fe-Cr. Note that Matijasevic et al. [23] found both $\langle 100 \rangle$ and $\frac{1}{2}\langle 111 \rangle$ dislocation loops in a Fe-6Cr alloy irradiated at 300°C to 1.7dpa but did not indicate the relative proportions.

For loops with larger diameter (18-20 nm) Si and Cr are both segregated around the dislocation line whereas for smaller loop (in the range of 8-10 nm) Cr is found on the periphery of the loop with Si residing inside the loop. While it may be conceivable that Si being clustered in the center of small loops is a result of trajectory aberrations or migration during field evaporation, the two distinct populations of sizes would suggest a different nature (interstitial or vacancy) for the smaller and larger loops. However a change in the elastic strains from small to larger loops irrespective of the loop nature may also cause Si to distribute differently. On the other hand, Cr is only found in the periphery of the loops where the tensile strains are highest in agreement with recent Monte Carlo and molecular dynamics calculations [53]. This is also consistent with the segregation of Cr and Si to vacancy dislocation lines forming a low angle grain boundary (Fig. 5).

5. Conclusions

Atom probe tomography has been employed to quantify the microstructural changes occurring in a model Fe-6 at.%Cr alloy after neutron irradiation to 1.82 dpa at 290 °C. Cr and Si are segregated to dislocation loops with diameters smaller than 20 nm. The different inside-outside locations of Cr (outside) and Si (both) would suggest two types of loops. However, the observed loop habit planes are most consistent with $\frac{1}{2}\langle 111 \rangle$ type dislocation loops. Thus loop size may simply play a role in local chemistry. The APT chemical analyses of different grain boundaries in the as-received and neutron-irradiated conditions are consistent with Si and P thermal

equilibrium segregation assisted by RED. This is also the case for Cr, but there is much less data on the much lower segregation enthalpy in this case. Indeed, the observed segregation of all the solutes and particularly that of Cr may result from both RED and RIS. Ongoing thermal annealing experiments will clarify this point.

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Table 1: Compositions determined from analyses taken within grains away from grain boundaries (in at.%)

	Cr	Si	C	P	Mn	V	Fe
As received	5.5 ±0.2	0.04 ±0.02	0.03 ±0.02	0.005 ±0.003	0.003 ±0.001	0.01 ±0.01	(balance)
Neutron irradiated	5.7 ±0.2	0.03 ±0.01	0.02 ±0.01	0.005 ±0.002	0.001 ±0.001	0.01 ±0.01	(balance)

Figure 1: Representative 10nm thick slices from reconstructed datasets from the as-received alloy (a) and from the irradiated material showing distribution of Si and Cr away from (b) and near (c) grain boundaries.

Figure 2: (a) Cr atoms with an iso-concentration surface at 11 at%Cr for visualization and (b) Si atoms with the same thin slice containing a small dislocation loop; (c) Cr and (d) Si spatial distributions with the same thin slice containing a larger dislocation loop; (e) Size distribution of the visible dislocation loops; (f) Distribution of the normal to the habit planes for visible dislocation loops

Figure 3: (a) Cr, (b) C, (c) Si, and (d) P concentration profiles before and after irradiation for random high angle grain boundaries

Figure 4: (a) Cr, (b) C, (c) Si, and (d) P concentration profiles across different types of grain boundaries after neutron irradiation.

Figure 5: Cr and Si distribution with the low angle grain boundary plane