Neutron Irradiation Effects in Fe and Fe-Cr at 300°C

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Abstract

Fe and Fe-Cr (Cr = 10-16 at.%) specimens were neutron-irradiated at 300°C to 0.01, 0.1 and 1 dpa. The TEM observations indicated that the Cr significantly reduced the mobility of dislocation loops and suppressed vacancy clustering, leading to distinct damage microstructures between Fe and Fe-Cr. Irradiation-induced dislocation loops in Fe were heterogeneously observed in the vicinity of grown-in dislocations, whereas the loop distribution observed in Fe-Cr is much more uniform. Voids were observed in the irradiated Fe samples, but not in irradiated Fe-Cr samples. Increasing Cr content in Fe-Cr results in a higher density, and a smaller size of irradiation-induced dislocation loops. Orowan mechanism was used to correlate the observed microstructure and hardening, which showed that the hardening in Fe-Cr can be attributed to the formation of dislocation loops and α' precipitates.

Keywords: Fe-Cr; neutron; irradiation; dislocation loop; void; hardness; Orowan

1. Introduction

Ferritic-martensitic (F/M) steels are leading candidate materials for fusion reactors and Generation-IV fission reactors [1, 2] due to their superior properties such as higher thermal conductivity, lower thermal expansion [2], and their resistance to swelling [3, 4], helium embrittlement [5, 6] and irradiation creep at high temperatures ($T > 0.4 T_m$) [7]. However, F-M steels exhibit low-temperature ($T < 500^\circ\text{C}$) irradiation-induced embrittlement that leads to a substantial increase in the ductile-to-brittle transition temperature (ΔDBTT) even at very low irradiation doses [8]. It is important to understand the embrittlement mechanism and how it depends on the parameters such as irradiation conditions, composition, etc.

The Cr content of F/M steels is one of the key parameters that determines the irradiation-induced ΔDBTT [9], swelling [10] and other mechanical properties [11, 12]. Remarkably, these properties depend on Cr concentration in a non-monotonic manner. For ΔDBTT , a minimum was observed at $\sim 10\%$ Cr (in atomic percent, hereafter) [9], leading to the selection of this Cr concentration in many steels proposed for nuclear applications.

Binary Fe-Cr model alloys have been the focus of basic research for studying the effect of Cr on the irradiation embrittlement of F/M steels [11-16]. Those studies showed that the formation of dislocation loops and Cr-rich α' precipitates are the primary hardening mechanisms. It has been shown that the increase of hardening above $\sim 10\%$ Cr can be qualitatively explained with α' phase precipitation [17-19],

however it does not explain the increase below $\sim 10\%$ Cr where α' phase is in absence and dislocation loops are the primary source of the embrittlement.

The Cr content affects the dislocation loops as a strengthening mechanism in many aspects. First above all, Cr decreases the mobility of small dislocation loops [20, 21], which results in smaller loop sizes and larger loop densities as compared to high purity Fe [11, 14, 22]. However, the loop size and density are relatively constant beyond $\sim 2\%$ Cr as compared to the significant variation observed for $\Delta DBTT$. In addition, Cr affects the proportion of dislocation loops with $\frac{1}{2}\langle 111 \rangle$ and $\langle 100 \rangle$ Burgers vectors. The $\frac{1}{2}\langle 111 \rangle$ and $\langle 100 \rangle$ loops were suggested to have different contributions to strength as obstacles to dislocation movements [23]. Also, the two loop types vary significantly in mobility, which lead to different loop morphologies. The Cr dependence of the ratio of two Burgers vectors has been reported previously, but the results were not consistent [11, 24].

This paper presents the distinctive microstructural evolution of Fe and Fe-Cr under neutron irradiation at 300°C . The aim of this present paper is to show the effect of Cr on the process of defect clustering, and to correlate the microstructure with the hardness measurements using the Orowan model.

2. Materials and Experimental Procedure

Materials

This study examined neutron irradiation damage in Fe, Fe-10Cr polycrystals and Fe-Cr single crystals. Table 1 shows the chemical composition and grain size of the un-

irradiated specimens. The grain size was measured with electron backscatter diffraction (EBSD) [25].

The Fe-10Cr polycrystals were made by Carpenter in Wyomissing, PA. Before irradiation, samples were heated at 980°C for 30 minutes, air cooled, then heated at 780°C for 24 hours, and followed by air cooling. The Fe-Cr single crystals were made by Welsch Metallurgy in Cleveland, OH using Czochralski growth aiming at a nominal composition of 14% Cr. The Cr concentration is homogeneous within each specimen, however as shown in Table 2 the Cr concentration varied between specimens due to the evaporation of Cr during the growth process. For clarity, single crystal specimens and polycrystalline specimens are labeled with a SC and a PC, respectively.

Irradiation

Irradiation was conducted in the Advanced Test Reactor (ATR), Idaho National Laboratory (INL). Specimens with identical irradiation conditions were contained in the same capsule during irradiation. The target irradiation temperature was 300°C and the target doses were 0.01, 0.1 and 1 dpa. The 0.01 dpa and 0.1 dpa specimens were irradiated in the ‘rabbit system’ where the capsules were inserted and extracted in the middle of the cycle. The 1 dpa specimens were irradiated in standard positions where specimens stayed in the reactor for a complete cycle. As a result, 1 dpa specimens may receive some irradiations at a temperature below target value during reactor power ramping up and down [26].

Transmission Electron Microscopy (TEM)

TEM specimens were prepared in the Electron Microscopy Laboratory (EML) at INL and the Low Activation Material Design and Analysis (LAMDA) facility at Oak Ridge National Laboratory (ORNL). Microscopy was performed in the Center for Advanced Energy Studies (CAES), EML and LAMDA facility.

Instead of using the standard 3 mm TEM specimens, this study adopted 2.3 mm specimens in order to reduce the magnetic effect and to save materials. Specimens were mechanically thinned down to around 80 μm , and then jet-polished at -30 to -40°C to perforation with an electrolyte of 5 vol.% perchloric acid and 95 vol.% methanol. After perforation, specimens were mounted on 3 mm slot grids and then stored in dehydrated ethanol or methanol to reduce oxidation.

TEM was used to observe dislocations, dislocation loops and voids. Dislocations and dislocation loops were imaged under kinematical bright field and centered dark field imaging conditions ($g=110$ and 200 , $s_g \sim 0.1 \text{ nm}^{-1}$). The dislocation density was estimated using the linear intersect method on the TEM images [27]. The $\mathbf{g \cdot b}$ analysis was conducted to determine the Burgers vectors. The habit planes of dislocation loops were determined by observing their inclination planes at the zone axis. The voids were imaged with under- and over-focused imaging conditions.

To obtain the volume density of defects, the foil thickness of the TEM image was estimated primarily with the thickness fringes, and sometimes with convergent beam electron diffraction (CBED) and tilting method if the thickness fringe method was not feasible.

Hardness Measurements

Microhardness was measured in the LAMDA facility with a Vickers diamond hardness indenter. Vickers hardness HV is defined as

$$HV = \frac{F}{A}$$
$$A = \frac{d^2}{2 \sin(68^\circ)}$$

where F is the load in kgf and d is the average of indent diagonals in millimeters. The load and dwell time were 100g and 15 seconds, respectively. At least 7 indents were performed for each specimen.

Nanoindentation was performed in CAES in a Hysitron TI-950 TriboIndenter with a high load Berkovich indentation head. A Fixed displacement of 5 μm was used for all measurements. Before indentation, specimens were mechanically polished, and then electro-polished with 5 vol.% perchloric acid in methanol electrolyte for about 10 seconds to achieve a deformation-free surface. The hardness H obtained from nanoindentation is defined as

$$H = \frac{P}{A_r}$$

where A_r is the residual area of indentation and P is the maximum load.

3. Results

Microstructural evolution of Fe irradiated at 300°C

Figure 1 shows the microstructural evolution of Fe with increasing dose as observed via TEM. Not irradiated Fe had a small grain size of a few microns where sub-grain boundaries and free dislocations were generally observed inside grains. The free

dislocations clustered within sub-grains, resulting in regions of high and low dislocation densities. The dislocation density, dislocation morphology and grain size (measured with EBSD [25]) did not change noticeably with increasing dose.

While the grown-in dislocations were stable, irradiation-induced dislocation loops appeared after irradiation. The dislocation loops showed a highly heterogeneous distribution in the vicinity of pre-existing dislocations. As dose increased, loop density increased. The average loop size, however, was largest in the 0.1 dpa specimen. Burgers vector analysis was performed on the 0.1 dpa specimen because of its larger loop size and reasonable loop density. Most loops had a Burgers vector of $\langle 100 \rangle$.

Voids were observed in the 1 dpa Fe specimen as shown in Figure 2, and not in the specimens irradiated to lower doses. The average void size was 1.8 nm with a maximum of 5.0 nm. Their spatial distribution is reasonably uniform but with some regional variations. An example of a cluster of large voids is indicated by an arrow in the figure. The quantitative measurements of the microstructure are summarized in Table 3.

Microstructural evolution of Fe-Cr alloys irradiated at 300°C

In general, the Fe-10Cr polycrystals and Fe-Cr single crystals shared a similar microstructural evolution with increasing dose as shown in Figure 3 and Figure 4, respectively. Both non-irradiated Fe10Cr-PC and Fe14Cr-SC samples had a relatively low dislocation density. At 0.01 dpa, small dislocation loops were observed in both Fe-10Cr-PC and Fe14Cr-SC. However, the loop density was much lower in Fe10Cr-

PC ($2 \times 10^{19} \text{ 1/m}^3$) than in Fe14Cr-SC ($2 \times 10^{21} \text{ 1/m}^3$). The spatial distribution of dislocation loops was uniform and was not associated with line dislocations.

In contrast, dislocation decoration with small clusters was observed in specimens irradiated to 0.1 dpa. For the Fe10Cr-SC 68% of the dislocation loops were located next to the pre-existing dislocations, and 32% of dislocation loops were dispersed sparsely in the matrix. Compared to other single crystalline Fe-Cr specimens, Fe-10Cr-SC had a particularly low Cr content and a high density of pre-existing dislocations. For Fe10Cr-PC, the overall dislocation density was lower, and the degree of dislocation decoration was much less as compared to Fe10Cr-SC. Figure 5 shows one of the few observations of the slight dislocation decoration in Fe10Cr-PC.

For the 1 dpa Fe10Cr-PC and Fe16Cr-SC, a high density ($\sim 10^{22} \text{ 1/m}^3$) of uniformly distributed dislocation loops (size $\sim 6 \text{ nm}$) was observed. The loop density was higher and the loop size was smaller in Fe-16Cr-SC than in Fe-10Cr-PC. Burgers vector analysis shows that the Burgers vectors of 65% and 84% of the dislocation loops were $\langle 100 \rangle$ in Fe-10Cr-PC and in Fe-16Cr-SC, respectively. The rest of the loops had Burgers vector of $\frac{1}{2}\langle 111 \rangle$. In contrast to 1 dpa Fe, no voids were observed in 1 dpa Fe-Cr alloys.

Hardness Measurements

The microhardness and nanohardness measurements are summarized in Table 3. The two measurements were consistent, but microhardness had smaller data uncertainty compared to nanoindentation. Figure 6 shows the nanohardness H and the relative change ΔH defined as

$$\Delta H = \frac{H - H_0}{H_0} \times 100\%$$

Where H_0 is the hardness of un-irradiated specimens. As shown in Figure 6 and in Table 3, the hardness of not irradiated Fe was higher than that of not irradiated Fe-10Cr-PC and Fe-14Cr-SC. This should be due to the smaller grain size and higher dislocation density of Fe. With increasing irradiation, little changes were observed in the hardness of Fe. The hardness increases slightly by 7% at 1 dpa. In contrast, the hardness of Fe-Cr alloys increased significantly after irradiation: 96% and 158% for 1 dpa Fe-10Cr-PC and Fe-16Cr-SC, respectively.

Oddly, the hardness of **both single and poly-crystalline** Fe-Cr specimens was lower at 0.1 dpa than at 0.01 dpa. In order to exclude possibility of switching specimens, the radiological dose rate of the specimens at a distance of 30 cm was measured. The radiological dose rate of 0.1 dpa specimens was roughly an order of magnitude larger than the 0.01 dpa specimens, indicating that the specimens of the two target doses were not switched by mistake.

4. Discussion

4.1 Irradiation condition

Testing materials in a nuclear reactor is desired since it simulates the irradiation environment in a way closest to the real conditions. However, it is difficult to precisely control the irradiation condition (i.e. dose, dose rate and temperature) compared to other irradiation techniques such as ion irradiation. The irradiation

conditions of this study (0.01, 0.1 and 1 dpa at 300°C) were values predicted by calculations prior to the experiments, and may deviate from actual values.

Because the measures that can be employed to evaluate the deviation were not available, including as-run calculations, SiC temperature rod, melt wires or flux wires, the irradiation conditions of this study can only be assumed to be the initial predicted values of fluence and temperature. This study qualitatively verified the irradiation condition by comparing the obtained Fe microstructure with literature since irradiation damage in Fe has been extensively studied.

In Fe, the Burgers vector is primarily $\frac{1}{2}\langle 111 \rangle$ from room temperature to 70°C [28, 29]. It gradually changes to $\langle 100 \rangle$ as irradiation temperature increases. Above 280°C, $\langle 100 \rangle$ loops become dominant [30-32]. In this study, the dislocation loops in 0.1 dpa Fe specimen were primarily $\langle 100 \rangle$ loops, suggesting that its irradiation temperature was much higher than 70°C; otherwise, $\frac{1}{2}\langle 111 \rangle$ loops would **have been** observed.

The distribution of dislocation loops is another indicator. Heterogeneous loop distribution (e.g. dislocation decoration [30, 33], loop rafting [28] and loop clustering [30]) occur in Fe at irradiation **temperatures** lower than 300°C. Above 400°C, a uniform distribution of immobile $\langle 100 \rangle$ loops were unambiguously observed [11, 14, 30, 34]. Since 0.1 and 1 dpa Fe specimens exhibited evident dislocation decorations, their irradiation temperature should not exceed 300°C much; otherwise a uniform loop distribution **would have been** observed.

In addition, the density and size of dislocation loops can be used as a benchmark. Figure 7 shows the loop density and size in irradiated Fe along with literature data [28-30, 32, 33, 35, 36]. The measured values of this study fit fairly well in the $\sim 300^{\circ}\text{C}$ region of the map, which is another evidence of reasonably controlled irradiation condition of this study.

On the other hand, Figure 7 also shows that variation in irradiation temperature does exist. For instance, the loops size in 1 dpa Fe is 2 nm smaller than the 0.1 dpa Fe while their largest loops being observed was similar, which is unexpected since loop size should grow with dose. In addition, dislocation decoration was observed in 0.1 dpa but not in 1 dpa Fe-Cr specimens, indicating that the mobility of $\frac{1}{2}\langle 111 \rangle$ loop was higher in 0.1 dpa specimens than in 1 dpa specimens. Both observations above suggest that 0.1 dpa specimens had a higher actual irradiation temperature than 1 dpa specimens despite the same predicted irradiation temperature of 300°C .

4.2 Mechanisms for dislocation decoration in Fe under irradiation

One of the primary microstructural modifications in Fe after neutron irradiation at 300°C was the decoration of dislocations with point defect clusters/dislocation loops. Two mechanisms have been proposed previously:

1. The strain-field and Cottrell atmosphere around the pre-existing dislocations traps interstitials and promotes the formation of dislocation loops [30, 33, 37]
2. Irradiation randomly creates dislocation loops that travel freely until being trapped by the strain field of dislocations [38-41].

Since *in situ* information is not available in this study, it is unclear which mechanism was primary one. With the first mechanism, it is difficult to explain the very few loops in the dislocation-sparse regions in 1 dpa Fe specimen. Since 1 dpa is not a very low dose, considerable **number of** loops should be observed in these regions. Although the strain field and Cottrell atmosphere may increase the chance of loop formation at grown-in dislocations, it should not prevent loop formation in other regions.

In contrast, the second mechanism is more plausible since dislocation loops can move to grown-in dislocations and leave behind a loop-sparse region. However, for the second mechanism to be valid, the irradiation-induced dislocation loops must be the mobile $\frac{1}{2}\langle 111 \rangle$ loops, and not the immobile $\langle 100 \rangle$ loops. To be consistent with the observation of prevailing $\langle 100 \rangle$ loops in 0.1 dpa Fe specimen and in literature [31, 32, 35], a transformation of Burgers vector from $\frac{1}{2}\langle 111 \rangle$ to $\langle 100 \rangle$ is required after loops were captured by the grown-in dislocations. Loop transformations have been observed experimentally and **predicted** by modeling [42, 43].

4.3 The Cr effects on the microstructure

The addition of 10-16% Cr to Fe has several effects on the evolution of microstructure under irradiation, including the Burgers vector, spatial distribution, size and density of dislocation loops. In addition, it caused α' precipitation [19] and a suppression in void formation. The Burgers vector, spatial distribution, size and density are discussed as follows.

The loops in Fe irradiated at 300°C have the $\langle 100 \rangle$ Burgers vector (e.g. 0.1 dpa Fe and ref. [30-32, 34]), whereas irradiated Fe-Cr alloys have a mixed population of $\frac{1}{2}\langle 111 \rangle$ and $\langle 100 \rangle$ Burgers vector (e.g. 1 dpa Fe-10Cr-PC, Fe-16Cr-SC and ref.[12]). Considering similar results from previous studies at 400-450°C [11, 15], it leads to **the conclusion** that the addition of Cr to Fe increased the fraction of $\frac{1}{2}\langle 111 \rangle$ loops under neutron irradiation at 300-450°C.

It has been shown with atomistic modeling that the $\langle 100 \rangle$ loops **form** in Fe through the interaction of two $\frac{1}{2}\langle 111 \rangle$ loops, and **it** has been suggested that the reduced mobility of $\frac{1}{2}\langle 111 \rangle$ loops in Fe-Cr leads to **a** lower chance of $\langle 100 \rangle$ loop formation [43, 44]. These modeling results help explain the higher fraction of $\frac{1}{2}\langle 111 \rangle$ loops observed in irradiated Fe-Cr alloys. It also explains the lower fraction of $\frac{1}{2}\langle 111 \rangle$ loops in 1 dpa Fe-16Cr-SC (16%) compared to 1 dpa Fe-10Cr-PC (35%) because the mobility of small $\frac{1}{2}\langle 111 \rangle$ loops reaches a minimum at about 11% Cr [45, 46].

However, the mobility of $\frac{1}{2}\langle 111 \rangle$ loops fails to explain the room temperature experiments where the proportion of $\langle 100 \rangle$ loops increased with Cr addition [24]. It indicates that Cr tends to equalize the fraction of two Burgers vectors instead of increasing only $\frac{1}{2}\langle 111 \rangle$ type. A more sophisticated theory is needed to interpret this conflict of experimental data.

Cr also affects the spatial distribution of dislocation loops. The 1 dpa Fe specimen had a highly heterogeneous microstructure of dislocation decoration, whereas the 1 dpa Fe-Cr alloys had a homogeneous loop distribution. The addition of Cr decreased the mobility of $\frac{1}{2}\langle 111 \rangle$ loops [20], and deterred them from migrating to grown-in

dislocations, resulting in a uniform distribution. For 0.1 dpa Fe-10Cr-SC, some dislocation decoration was observed probably due to the higher actual irradiation temperature that increased the mobility of $\frac{1}{2}\langle 111 \rangle$ loops. However, its percentage of decorating loops is still less than that in Fe specimens.

The Cr addition in Fe-Cr affects the size and density of irradiation-induced dislocation loops. It is well established that an addition of about 2% Cr significantly decreases the loop size as compared to Fe [11, 12, 14, 47]. Beyond 2%, however, the loop size dependence on Cr concentration is much weaker and is still unclear. While in general loop size continues to decrease gradually with increasing Cr contents, some studies showed that loop size may have a local maximum at $\sim 10\%$ Cr [11, 12, 14]. In this study, the wide variation in grain size and grown-in dislocation density between Fe and Fe-Cr specimens posed difficulties in showing the single parameter effect of Cr concentration. For 1 dpa Fe-10Cr-PC and Fe-16Cr-SC, while the effects of grain boundary and grown-in dislocations are insignificant and the measurement statistics are sufficient due to high loop density, it shows that increasing Cr concentration led to smaller loop size and larger loop density.

4.4. Relationship between hardness and microstructure

The Orowan model was used to compare the microstructure to the mechanical properties. The Orowan formula is given as follows:

$$\Delta\sigma_y = \alpha_{dis}\mu b\sqrt{\rho_{dis}} + M\alpha_{loop}\mu b\sqrt{N_{loop}d_{loop}} + M\alpha_{\alpha'}\mu b\sqrt{N_{\alpha'}d_{\alpha'}}$$

where $\Delta\sigma_y$ is the increase in yield strength. M is the Taylor factor, here $M = 3.06$ [12, 48], μ is the shear modulus, here $\mu = 83$ GPa [11, 12], and b is the length of Burgers vector, here $b = 0.248$ nm. The α_{dis} , α_{loop} and $\alpha_{\alpha'}$ are barrier strength constants for line dislocations, dislocation loops and α' precipitate, respectively. Here, $\alpha_{dis} = 0.64$ [11], $\alpha_{loop} = 0.4$ [48], $\alpha_{\alpha'} = 0.048$ [48]. There are other plausible hardening mechanisms including, but not limited to, invisible (beyond TEM resolution) defect clusters, ratio of $\langle 100 \rangle$ -to- $\frac{1}{2}\langle 111 \rangle$ loops, voids and extent of Cr segregation to the dislocation loops [11, 12, 49]. They were neglected in the calculation because sophisticated models describing their effect are not currently available. For the voids, accurate density was not obtained.

The calculation and the corresponding hardness measurements are shown in Figure 8. For Fe-Cr alloys, the calculation is qualitatively consistent with the measured hardening, indicating that the hardening can be attributed to the observed defect microstructure. The irradiation induced hardening was little at 0.01 dpa and 0.1 dpa due to the low density of dislocation loops and α' precipitates. The appearance of dense loops and α' precipitates is clearly responsible for the sharp hardening at 1 dpa. The increased hardening with more Cr in Fe-Cr is mostly due to the effect of Cr on α' precipitates, and less significantly on dislocation loops.

In contrast, the calculations for Fe were less consistent with the measured hardening. While the calculated hardening increases with dose due to loop formation, the measured hardness is relatively constant and is only slightly increased at 1 dpa. The discrepancy may be partially attributed to the use of the same loop barrier strength α_{loop} in Fe and in Fe-Cr. In Fe-Cr, the Cr segregation on

dislocation loops has been shown to strengthen the dislocation loops as the obstacles for dislocation gliding [50]. The barrier strength $\alpha_{\text{loop}} = 0.4$ is adopted from Fe-Cr alloys in Ref [48], and therefore may be an overestimation for Fe. In addition, Orowan model assumes a uniform distribution of obstacles, whereas the loop distribution in irradiated Fe was highly heterogeneous, resulting in possible break down of the model.

Conclusion

In this work, Fe and Fe-Cr irradiated at 300°C to 0.01, 0.1 and 1 dpa were examined with TEM and hardness measurements. Dislocation loops were observed in both Fe and Fe-Cr systems, whereas voids were observed only in Fe. The presence of Cr clearly influenced the morphology of dislocation loops. For Fe, a highly heterogeneous structure of dislocation decoration by dislocation loops was observed. In contrast, the loop distribution in Fe-Cr was more uniform due to the decreased mobility of $\frac{1}{2}\langle 111 \rangle$ loops. At 1 dpa, Fe-16Cr had a slightly smaller loop size and higher loop density as compared to Fe-10Cr. Dislocation loops in 1 dpa Fe-Cr were shown to have a mixture of $\frac{1}{2}\langle 111 \rangle$ and $\langle 100 \rangle$ Burgers vectors. For the 0.1 dpa Fe, $\langle 100 \rangle$ loops that decorated grown-in dislocations were observed. We suggested that loops were initially $\frac{1}{2}\langle 111 \rangle$ loops and were later transformed into $\langle 100 \rangle$ loops

The hardening is higher in Fe-Cr than in Fe due to the presence of α' precipitates, higher loop density and possibly higher loop strength as obstacles. For Fe-Cr, it appears that α' precipitation was the primary mechanism that lead to the Cr

dependence of hardening. The contribution of dislocation loops to the hardening seems to be less sensitive to the Cr concentration.

Acknowledgement

This study was supported by ATR NSUF under identification number 08-092 and by NEUP under grant number 485363-973000-191100. Both funding are titled 'Irradiation Performance of Fe-Cr Base Alloys'. Authors would like to thank Dr. Marquis Kirk, Dr. Zhongwen Yao and Dr. Bai Cui for meaningful discussions and suggestions. Dr. Kun Mo and Carolyn Tomchik were thanked for initiating the irradiation experiments.

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Figure 1. TEM micrographs showing the microstructural evolution of Fe irradiated at 300°C.

Figure 2. TEM micrographs of Fe polycrystalline specimen irradiated at 300°C to 1 dpa. Under and over focused conditions showed the voids. The dark contrast at the center is a contamination.

Figure 3. TEM micrographs showing the microstructural evolution of Fe-10Cr polycrystals irradiated at 300°C.

Figure 4. TEM micrographs showing the microstructural evolution of Fe-Cr single crystals irradiated at 300°C.

Figure 5. TEM micrograph of 0.1 dpa Fe10Cr-PC showing dislocation decorated with dislocation loops.

Figure 6. The hardness H and the relative hardness ΔH measured with nanoindentation. The error bars correspond to the standard deviation of the data.

Figure 7. A summary of loop density and size measurements in neutron-irradiated Fe from this study and from literature. The background color of orange, white and gray is to guide the eyes. Data reported in Horton et al. was presented as squares with colors from blue to red indicating irradiation temperatures from 275°C to 500°C. The representative uncertainty of the loop size in this study is about ± 0.5 nm.

Figure 8. (Top row) The calculated increase in yield strength based on the observed grown-in dislocations, dislocation loops and α' precipitates. (Middle row) The relative increase in calculated yield strength compared to the non-irradiated samples. (Bottom row) The relative nanohardness compared to non-irradiated samples.

Table 1. Chemical composition (at%), grain size and dislocation density of the un-irradiated specimens

	Fe polycrystals ¹	Fe-10Cr polycrystals ²	Fe-Cr single crystals ³
Cr	< 0.000002	9.7	13.7
C		0.023	<0.09
Mn	0.00001	< 0.01	<0.02
Si	< 0.00001	< 0.02	
P		< 0.009	
S			
Ni	< 0.000005		
Mo		< 0.06	
Cu	< 0.000005	< 0.009	
Co			
Al	< 0.000005	< 0.021	
N		0.006	<0.04
V			<0.02
Ag	< 0.000005		
Ca	< 0.00008		
Mg	0.000002		
Fe	bal.	bal.	bal.
Grain Size (μm)	2.7	181	-

¹ Analysis performed by Goodfellow

² Analysis performed by Carpenter

³ Measured with APT [1]

Table 2. Cr concentrations of Fe-Cr single crystals **measured by APT**

	Specimen Label	Irradiation Conditions	x _{Cr} (at.%)
Single-Crystals	Fe16Cr-SC	300°C-1 dpa	16.0
	Fe10Cr-SC	300°C -0.1 dpa	10.0
	Fe14Cr-SC	300°C -0.01 dpa	14.0
	Fe14Cr-SC	No irradiation	13.7

Table 3. Quantitative Measurements of Irradiation-Induced Defect Clusters

Fe	No dose	0.01 dpa	0.1 dpa	1 dpa
ρ_d ($10^{14} \frac{1}{m^2}$)	0.9	1.1	1.2	1.2
$\bar{d}_{loop}$ (nm)	-	6.1	8.5	6.0
$d_{loop,Max}$ (nm)	-	10.9	19.2	20.54
N_{loop} ($10^{21} \frac{1}{m^3}$)	-	0.19	1.2	5.0
$\bar{d}_{void}$ (nm)	-	n.o.	n.o.	1.8
$d_{void,Max}$ (nm)	-	n.o.	n.o.	5.0
H_V (HV)	145.4 ± 3.5	147.4 ± 7.4	167.2 ± 6.4	n.m.
H_{nano} (GPa)	2.7 ± 0.3	2.5 ± 0.2	2.6 ± 0.2	2.9 ± 0.1
Fe-10Cr polycrystals	No dose	0.01 dpa	0.1 dpa	1 dpa
ρ_d ($10^{13} \frac{1}{m^2}$)	1.3	0.9	0.5	-
$\bar{d}_{loop}$ (nm)	-	3.5	3.5	6.5
$d_{loop,Max}$ (nm)	-	5.0	6.6	19.0
N_{loop} ($10^{21} \frac{1}{m^3}$)	-	0.017	0.073	10
H_V (HV)	n.m.	n.m.	n.m.	n.m.
H_{nano} (GPa)	1.8 ± 0.1	1.9 ± 0.1	1.80 ± 0.04	3.5 ± 0.1
Fe-Cr single crystals	No dose Fe-14Cr-SC	0.01 dpa Fe-14Cr-SC	0.1 dpa Fe-10Cr-SC	1 dpa Fe-16Cr-SC
ρ_d ($10^{12} \frac{1}{m^2}$)	3.5	-	14	-
$\bar{d}_{loop}$ (nm)	-	2.6	5.2	4.9
$d_{loop,Max}$ (nm)	-	6.1	10.7	15.6
N_{loop} ($10^{21} \frac{1}{m^3}$)	-	1.1	0.32	15
H_V (HV)	127.0 ± 5.4	159.2 ± 1.9	136.4 ± 2.1	n.m.
H_{nano} (GPa)	1.90 ± 0.02	2.5 ± 0.1	2.2 ± 0.1	4.8 ± 0.2
n.o.: not observed.	ρ_d : Dislocation density		$d_{loop,Max}$: Maximum loop size	
n.m.: not measured.	$\bar{d}_{loop}$: Average loop size		$d_{void,Max}$: Maximum void size	
$\bar{d}_{void}$: Average void size	N_{loop} : Loop density		H_V : Vickers Microhardness	
H_{nano} : Nanohardness				

1dpa Fe 300°C

1dpa Fe10Cr 300°C

Addition of Cr in Fe suppressed the mobility of mobile $1/2\langle 111 \rangle$ dislocation loops and increased the proportion of immobile $\langle 100 \rangle$ dislocation loops, leading to a transition of loop distribution from highly heterogeneous to uniform.