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Effect of dose on irradiation-induced loop density and Burgers vector in ion-irradiated ferritic/martensitic steel HT9

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ABSTRACT
Samples of F/M steel HT9 were irradiated to 20 dpa at 420°C, 440°C and 470°C in a transmission electron microscope with 1 MeV Kr ions so that the microstructure evolution could be followed \textit{in situ} and characterised as a function of dose. Dynamic observations of irradiation-induced defect formation and evolution were made at the different temperatures. Irradiation-induced loops were characterised in terms of their Burgers vector, size and density as a function of dose and similar observations and trends were found at the three temperatures: (i) both $a/2 <111>$ and $a <100>$ loops are observed; (ii) in the early stage of irradiation, the density of irradiation-induced loops increases with dose (0–4 dpa) and then decreases at higher doses (above 4 dpa), (iii) the dislocation line density shows an inverse trend to the loop density with increasing dose: in the early stages of irradiation, the pre-existing dislocation lines are lost by climb to the surfaces while at higher doses (above 4 dpa), the build-up of new dislocation networks is observed along with the loss of the radiation-induced dislocation loops to dislocation networks; (iv) at higher doses, the decrease of number of loops affects more the $a/2 <111>$ loop population; the possible loss mechanisms of the $a/2 <111>$ loops are discussed. Also, the ratio of $a <100>$ to $a/2 <111>$ loops is found to be similar to cases of bulk irradiation of the same alloy using 5 MeV Fe\textsuperscript{2+} ions to similar doses of 20 dpa at similar temperatures.

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\textit{In situ} TEM; ion irradiation; radiation damage; ferritic/martensitic steel; HT9

1. Introduction

High-Cr ferritic/martensitic steels are potential candidates for fuel cladding and structural materials in Gen IV nuclear reactors because of their superior resistance to radiation swelling (relatively to austenitic steels) and their enhanced corrosion resistance at higher operating temperatures \cite{1,2}. A fundamental
understanding of radiation damage formation and evolution in these alloys is necessary to develop predictive models of irradiated microstructures and corresponding mechanical behaviour. Neutron irradiation is time-consuming, expensive and access to adequate facilities is restricted. In contrast, ion irradiation provides better control of irradiation parameters such as dose rate (through the choice of the ion type and energy) and temperature. Whether irradiated with neutrons or ions, usually the irradiated samples are examined ex situ when the irradiation is over, and the samples are cooled down so that only snapshots of the microstructure evolution are available at limited doses. Hence, coupling ion irradiation with in situ transmission electron microscopy (TEM) can be powerful as it allows to follow the evolution of radiation-induced microstructures as a function of dose and to correlate radiation-induced microstructures with pre-existing microstructures (e.g. grain boundaries, dislocation lines) in the material.

In the literature, several in situ TEM studies of irradiation-induced microstructures can be found on pure Fe and Fe–Cr model alloys [3–8] whereas only a few studies are available on commercial and advanced ferritic/martensitic steels [9,10]. Thus, in this work, the advanced F/M steel HT9 was irradiated in situ to 20 dpa at 420°C, 440°C and 470°C in a TEM using 1 MeV Kr\(^{2+}\) ions. The size and density of dislocation loops and dislocation lines were characterised as a function of dose and temperature. Also, special emphasis was put on identifying the Burgers vector of radiation-induced dislocation loops. In addition, the results of these in situ experiments were compared with the ex situ bulk irradiation of the same alloy using 5 MeV Fe\(^{2+}\) ions to the same dose at same temperatures.

2. Experimental

2.1. Materials and TEM specimen preparation

The F/M steel HT9 was provided by Los Alamos National Laboratory with a tempered martensitic structure due to a heat treatment consisting of 1038°C/5 min and air cooling followed by 760°C/30 min and air cooling. Typical microstructures prior to irradiation were examined and reported in [11], including prior austenite grain boundaries (PAGBs), martensitic lath grains and M\(_{23}C_6\) carbides usually forming along with grain boundaries, as well as V-rich nitride precipitates. For in situ and ex situ ion-irradiated HT9, TEM specimens were prepared before and after irradiation, respectively. TEM specimens were prepared using a focused ion beam (FIB) lift-out technique with an FEI Quanta 3D FIB system. A thick lamella was firstly prepared by trenching the selected region with a 30 keV Ga\(^+\) beam. The lamella with thickness ∼ 2 μm was then cut from the bulk of the sample, attached to a TEM grid via a nano-manipulator and Pt deposition. The lamella was thinned to a thickness of ∼100 nm using a series of lower beam currents. Final cleaning of the lamella was performed with a 2–5 keV Ga\(^+\) beam to minimise FIB damage introduced by the Ga\(^+\) beam.


2.2. In situ ion irradiation and TEM characterisation

The *in situ* experiments were carried out at the IVEM-Tandem Facility, at Argonne National Laboratory. The facility consists of a Hitachi 9000 NAR TEM coupled with a 500 keV NEC implanter. The HT9 specimens were mounted in a double-tilt heating holder and irradiated in the TEM at 420°C, 440°C and 470°C using 1 MeV Kr\(^{2+}\) ions with the ion beam incident at 30° from the electron beam and typically ∼13–16° from the specimen normal. The temperature was measured by a thermocouple attached to the heating cup of the holder and was kept within ± 5°C of the nominal temperature. During irradiation, the flux was kept at 6.25 × 10\(^{11}\) ions cm\(^{-2}\) s\(^{-1}\) and measured to an accuracy of ± 10% with an annular Faraday cup located within the microscope at 4 cm from the TEM specimen.

The Kr ion beam induced damage profile in HT9 was predicted using SRIM-2008 [12] using the ‘quick damage calculation’ mode using a density of 7.86 g/cm\(^3\) and displacement energies of 40 eV/at for metallic elements and 28 eV/at for other nonmetallic elements [13]. The displacement per atom (dpa) value was calculated using the following equation:

\[
dpa = \frac{\Phi \times 10^8 \times v}{N},
\]

where \(\Phi\) is the fluence in ions.cm\(^{-2}\), \(v\) is the number of vacancies per ion per unit depth obtained from SRIM, and \(N\) is the atomic number density in atoms.cm\(^{-3}\).

As shown in Figure 1, \(v\) was determined to be ∼1.44. As a result, the dose rate used in the experiments was ∼1.07 × 10\(^{-3}\) dpa/s. The retention of Kr ions was evaluated to be ∼4.25%. Regarding the *ex situ* ion-irradiation experiments

![Figure 1. SRIM calculations showing the damage profile and retention of Kr ions vs. depth in HT9 irradiated with 1 MeV Kr\(^{2+}\) ions.](image-url)
The visible defect clusters and loops were assumed to have a generic elliptic shape with a small diameter $a$ and a large diameter $b$; their size was then taken as the diameter ($d_{eq}$) of the disc of the equivalent area such that:

$$d_{eq} = \sqrt{a \times b}.$$ 

The relative thickness ratio ($t/\lambda$) was measured using the electron energy loss spectroscopy (EELS) zero loss method, with an associated error of $\sim 10\%$, as suggested by Malis et al. [14].

In addition, video recording was performed during the irradiations for subsequent frame-by-frame analysis. The frame rate was $\sim 15$ frames/second. Video snapshots and clips were created and edited using Adobe© premiere pro CS4.

### 2.3. Dislocation loop Burgers vector identification using the on-zone STEM-BF imaging method

For ferritic Fe–Cr alloys, it is generally accepted that two types of dislocation loops with Burgers vectors $a <100>$ and $a/2 <111>$ can form [15–17]. The identification of the loop Burgers vector is typically done by taking a series of images of the same area of interest using different diffracting $g$ vectors, and using the $g \cdot b = 0$ invisibility criterion. Recent work by Yao et al. [18] has sought to facilitate the identification by illustrating the expected dislocation loop morphologies in ferritic Fe–Cr alloys. However, this method still requires to carefully tilt the sample to different $g$ vectors and can be prone to errors due to misinterpretation of the contrast observed in the TEM images. Parish et al. [19] applied this method using scanning transmission electron microscopy (STEM) imaging which helps image the dislocation loops and identify their Burgers vector more easily since STEM significantly reduces the elastic contrast in the background by smearing out the thickness contrast. In this study, this method was performed on both in situ and ex situ HT9 irradiated to 20 dpa at 420°C, 440°C and 470°C, using an FEI Titan 80–300 probe aberration-corrected microscope operating at 200 kV.

### 3 Results

#### 3.1. TEM characterisation of in situ ion-irradiated HT9

During the experiments, both the evolution of the initial microstructure (i.e. initial dislocation network) and the evolution of the population of irradiation-
induced defects (black dots and dislocation loops) were followed as a function of dose at each temperature. All the TEM images were taken under identical kinematical diffraction conditions, in which \( g = \overline{1}10 \) was strongly excited near the [111] zone axis. Regardless of the temperature, similar dynamic observations of irradiation-induced defect formation and evolution were made during the irradiations with ‘black-dot’ defects forming very early on (at doses less than 0.3 dpa) and resolvable loops appearing between 0.6 and 1 dpa. Throughout the irradiations, black dots are constantly created (appearance) and eliminated (disappearance). As an example, Figure 2 shows the case of the 470°C irradiation, where small defect clusters appear firstly as black dots at doses as low as 0.04 dpa (2.3 \( \times 10^{13} \) ions \( \text{cm}^{-2} \)) and resolvable dislocation loops originating from the defect clusters become visible between 0.8 and 1 dpa ((4.69–5.86) \( \times 10^{14} \) ions \( \text{cm}^{-2} \)). With increasing irradiation dose, the number density of black dots and resolvable loops both initially increase. Meanwhile, as shown in Figure 3, radiation-induced defects also interact with the pre-existing dislocation lines resulting in the alteration of the dislocations shape and curvature. Dislocation climb to the surfaces also seems to happen as the initial dislocation lines progressively disappear, resulting in the decrease of dislocation line density in this early stage of irradiation. In Figure 3, the initial dislocation lines are highlighted in red and the progressive disappearance is well apparent in the early stage of the irradiation (up to 4 dpa). At higher doses (above 4 dpa), the build-up of dislocation network and loss of irradiation-induced loops result in an increase of dislocation line density (but a decrease of dislocation loop density).

Such trends were quantitatively substantiated by measuring the dislocation line density and loop density and size on each sequential TEM image taken from 0 to 20 dpa, at each temperature. An example of how the measurements

![Image](image-url)

**Figure 2.** Sequential TEM bright-field images showing the accumulation of defect clusters and the formation of resolvable dislocation loops through the defect clusters in HT9 irradiated *in situ* at 470°C.
Figure 3. (Top) Sequential TEM bright-field images showing the evolution of initial dislocation lines and radiation-induced dislocation loops in HT9 irradiated in situ at 420°C. (Bottom). The initial dislocation lines are highlighted with red lines in the sequential TEM images.

were done is shown in Figure 4. The measured lines and loops are highlighted in red and yellow, respectively.

Figure 5 shows the plots of dislocation line density versus dose and dislocation loop density versus dose as well as the average dislocation loop size

Figure 4. Measurements of dislocation lines and dislocation loops in the TEM image of HT9 irradiated in situ at 440°C. The dislocation lines and dislocation loops are highlighted with red lines and yellow cross, respectively.
versus dose for each temperature investigated. For the dislocation line density measurement, the lengths of each segments were added and the total measured length was divided by the volume (using the thickness measured with EELS).

Overall, the trends are similar for all three temperatures: (i) black-dot defects start to appear after a small threshold dose; (ii) Resolvable loops begin to show up around 1 dpa and the loop density increases by 3–4 orders of magnitude from 1 to 4 dpa; (iii) in the meantime, the climb and loss of dislocation lines to surfaces are observed, resulting in the decrease of dislocation line density in this early stage of irradiation (1–4 dpa); (iv) at higher doses (above 4 dpa), the build-up of a new dislocation network is observed along with the loss of dislocation loops to the network formation, resulting in an increase of dislocation line density (and decrease of dislocation loop density); (v) as far as the average size of the irradiation-induced loops, it increases with dose but the size seems to

**Figure 5.** Quantitative analyses of dislocation lines and loops in HT9 irradiated in situ to 20 dpa at 420°C, 440°C and 470°C: (left) Average loop size vs. dose; (right) Dislocation line and loop volume density vs. dose.
saturate at the higher doses when the larger loops may contribute to the build-up of the network. The dislocation line density shows an inverse trend to the loop density at all three temperatures.

Note that systematic errors of the experiment can come from diffraction imaging condition. In fact, the extinction criterion $g \cdot b = 0$ determines that only a fraction of the total loops is accounted for a given reflection vector. As mentioned in the experimental part, for ferritic Fe–Cr-based alloys, it is accepted that Burgers vectors of dislocation loops are typically $a/2 <111>$ and $a <100>$ types [15–17]. When using $g = 1\overline{1}0$ reflection vector near [111] zone axis, only one half of $a/2 <111>$ and two third of $a <100>$ loops are visible. Although the number density of dislocation loops was underestimated, no correction was made in Figure 5.

At the highest doses, the microstructure is dominated by a complex network of dislocation segments being created during irradiation. The complex network has no apparent correlation with the initial pre-existing dislocation lines which indeed climb and are lost to the surfaces of the foil in the early stages of the irradiation. The formation of dislocation segments and/or networks in irradiated HT9 in the later stage of irradiation is evidenced by in situ TEM observations. One example of such dynamic observations is given in Figure 6, showing the coalescence of two adjacent dislocation loops which results in the build-up of dislocation network in HT9 in situ ion-irradiated at 440°C. Similarly, dynamic observations done during the irradiations show the interaction of dislocation loops with dislocation lines, contributing to the build-up and dynamic ‘re-organization’ of the dislocation network as the irradiation proceeds, as shown in Figure 7 at 440°C.

### 3.2. Dislocation loop Burgers vector analysis

The dislocation loop Burgers vector analysis was performed on both in situ (4 and 20 dpa) and ex situ (20 dpa)-irradiated HT9. Examples of dislocation

![Figure 6](image)

**Figure 6.** (a–d) Dynamic observations show the coalescence of two adjacent dislocation loops, forming the dislocation network in HT9 irradiated in situ to 5.0–5.3 dpa at 440°C. (e–h) Schematic illustrations correspond to (a–d).
loop imaging using the on-zone STEM imaging are shown in Figures 8–10. Insets in Figure 8 and Figure 9 illustrate the expected dislocation loop morphologies near the [110] zone axis: a <100> loops appear as either edge-on perpendicular to the <002> directions or elongated ellipses aligned with the <002> directions, and a/2 <111> loops appear as either edge-on ellipses aligned with the

Figure 7. (a–f) Dynamic observation show a dislocation loop interacts with a dislocation line, resulting in the build-up and dynamic ‘re-organisation’ of the dislocation network in HT9 irradiated in situ to 4.6–4.8 dpa at 440°C. (g–l) Schematic illustrations correspond to (a–f).
<112> directions or elongated ellipses aligned with the <110> directions. Similarly, the insets in Figure 10 illustrate the expected dislocation loop morphologies near the [100] zone axis: a <100> loops appear as either edge-on ellipses or in-plane circles aligned with the <002> directions, and a/2 <111> loops appear as elongated ellipses aligned with the <110> directions. a/2 <111> type and a <100> type loops are highlighted with red arrows and blue arrows, respectively. For each dose, the on-zone STEM imaging method was performed on multiple areas (2–3 grains) in order to minimise statistical errors. The results are listed in Table 1. Overall, the ratio of a <100> loops/total is very similar at all temperatures investigated in this study. At 4 dpa (i.e at the peak of the dislocation loop density versus dose), both a <100> type loops and a/2 <111> type loops are present in similar proportions, whereas at the end of irradiation (20 dpa), the a <100> type loops are found to be the predominant type at all temperatures. Moreover, this result is comparable with the measurements done on the ex situ experiments at the same irradiation temperatures at 20 dpa (where the a <100> type loops are also found to be the predominant type).

Figure 8. The on-zone STEM imaging shows dislocation loops in HT9 irradiated in situ to 4 dpa at 420°C. Insets are the selection area electron diffraction (SAED) pattern and the expected dislocation loop morphologies near the [110] zone axis. a/2 <111> and a <100> loops are highlighted with red and blue arrows, respectively.
4. Discussion

4.1. On the thin-foil orientation and free-surface effects:

The foil orientation can influence the measured number density of irradiation-induced loops because of possible glide and loss to the free surfaces due to the geometry. Indeed glide and loss to free surfaces has been observed during in situ experiments of ultra-high purity (UHP) Fe irradiated at both RT and 420°C.

![Figure 9](image1.png)

**Figure 9.** The on-zone STEM imaging shows dislocation loops in HT9 irradiated in situ to 20 dpa at 420°C. Insets are the selection area electron diffraction (SAED) pattern and the expected dislocation loop morphologies near the [110] zone axis. a/2 <111> and a <100> loops are highlighted with red and blue arrows, respectively.

![Figure 10](image2.png)

**Figure 10.** The on-zone STEM imaging shows dislocation loops in HT9 irradiated ex situ to 20 dpa at 420°C. Insets are the selection area electron diffraction (SAED) pattern and the expected dislocation loop morphologies near the [100] zone axis. a/2 <111> and a <100> loops are highlighted with red and blue arrows, respectively.
300°C [20], and showed directly that the defect yield i.e. the number of loops remaining at the end of the irradiation was often less than the total number produced during irradiation. Moreover, in this UHP Fe [20,21], the defect yield was dependent on the foil orientation (i.e. normal to the foil surface). In orientations such as <111> [20], the defect yield was lower than in <110> orientations [21] for a same dose. A possible explanation was found in the fact that mobile a/2 <111> loops of all four Burgers vector variants can be lost to the surface by glide in a foil of UHP Fe with <111> orientations; however, in the <110> orientations, two of the four variants have Burgers vectors in the foil plane, thus cannot be lost by glide to the surface. It was also noted that the foil orientation effect on the defect yield was less significant in Fe–Cr system where the mobility of the loops was reduced. In our case, differences that may arise from foil orientation effects were alleviated by preparing samples which had a similar orientation (<111>). Also, the loss of a/2 <111> loops to the free surfaces is expected to be less prevalent than reported in the literature for much more simple and pure systems (such as UHP Fe) because of the presence of fine precipitates, the build-up of irradiation-induced dislocation network at the higher irradiation doses, and the overall high alloying content, reducing the mobility of the loops.

### 4.2. On the Burgers vector analysis of the irradiation-induced loops

The literature shows that a consensus exists on the Burgers vector type of dislocation loops which develop in ferritic Fe and Fe–Cr systems [3–10]. Both experimental observations and simulations [22–24] have confirmed the formation of a/2<111> and a <100> dislocation loops. In our study, both a/2 <111> and a <100> dislocation loops are observed in the in situ and the ex situ ion-irradiated HT9. Moreover, the fraction of a <100> loops in the in situ-irradiated HT9 is similar to that found in the ex situ-irradiated HT9 at 20 dpa for the same temperatures. However, the in situ ion irradiations reveal that (i) the total number of dislocation loops increases at early doses and then decreases at higher doses (above 4 dpa) and (ii) the relative fraction of a <100> loops with increasing

<table>
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<tr>
<th>Irradiation type</th>
<th>Dose (dpa)</th>
<th>Temperature (°C)</th>
<th>No. of a &lt;100&gt; loops</th>
<th>No. of a/2 &lt;111&gt; loops</th>
<th>a &lt;100&gt; + a/2 &lt;111&gt;</th>
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<tr>
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<td>420</td>
<td>22</td>
<td>27</td>
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<td></td>
<td>4</td>
<td>470</td>
<td>23</td>
<td>24</td>
<td>48.9%</td>
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<tr>
<td></td>
<td>20</td>
<td>420</td>
<td>35</td>
<td>13</td>
<td>72.9%</td>
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<tr>
<td></td>
<td>20</td>
<td>440</td>
<td>33</td>
<td>13</td>
<td>71.7%</td>
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<td></td>
<td>20</td>
<td>470</td>
<td>39</td>
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<td>73.6%</td>
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<tr>
<td>Ex situ</td>
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<td>420</td>
<td>26</td>
<td>11</td>
<td>70.3%</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>440</td>
<td>13</td>
<td>4</td>
<td>76.5%</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>470</td>
<td>13</td>
<td>5</td>
<td>72.2%</td>
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dose above 4 dpa at all the temperatures, which suggests that the loss of dislocation loops at higher doses affects more the a/2 <111> loop population. To explain such trend, several mechanisms can be invoked based on what is known or assumed of the transition of a/2 <111> loops to a <100> loops in bcc Fe and Fe–Cr systems. (i) The so-called 111 mechanism whereby a<100> loops form from the interaction of a/2 <111> loops, according to the reaction
\[
a/2[111] + a/2[\overline{1}1\overline{1}] \rightarrow a[100]
\]
could be at play. This ‘111 mechanism’ was first deduced from experimental observations in 1965 [25] and later explored and supported by MD simulations [24,26]. (ii) Arakawa et al. [27] reported in situ TEM observations in bcc Fe of a/2 <111> interstitial loops with diameters less than ∼50 nm transforming from a/2 <111> to the energetically unfavourable a <100> type without contact with external dislocations during high-energy electron irradiation or simple heating. They thus proposed a different mechanism for the spontaneous transformation from a/2 <111> loops to a <100> loops. (iii) Also, more recently, another mechanism has been suggested which involves spontaneous transformation to a <100> loops from primary damage sessile clusters (including the C15 clusters) [28], which, according to MD simulations of displacement cascades in Fe, represent a significant fraction of the primary damage SIA clusters (30–50%) [17]. Such a mechanism can explain an increase in the number of a <100> loops but not the decrease of the number of a/2 <111> loops seen in this study (cf. Table 1) since the loops counted were too large to be C15 clusters. Mechanisms (i) and (ii) can account for both the increase of the a <100> relative fraction and the concomitant decrease in number of a/2 <111> loops.

The effect of temperature needs to be discussed as well as it can have an impact on the mechanisms mentioned above, thus on the relative fraction of a/2 <111> loops and a <100> loops observed in TEM. Schaublin et al. [6] irradiated UHP Fe in situ at liquid–nitrogen temperature and found the a/2 <111> loops to be predominant over the a <100> loops. Upon heating to room temperature, the ratio of a/2 <111> loops to a <100> loops decreased to ∼1 and the density of a <100> loops slightly increased, which was related to the increase of mobility of the a/2 <111> loops upon heating, so that they could escape more easily to the free surfaces and/or interact to form a <100> loops. Yao et al. [29] carried out in situ irradiations of pure Fe at 300–500°C. Below 400°C, a/2 <111> loops were reported to be the predominant type, although a <100> loops were also present. In contrast, only a <100> loops were observed at 500°C and a gradual transition between the two types of loops was reported at intermediate temperatures between 400°C and 500°C. Such results seem to be supported by the work of Dudarev et al. [30] who, based on calculations of the anisotropic elastic self-energies of loops of different Burgers vectors in bcc Fe as a function of temperature, pointed out that a <100> loops become relatively more stable with respect to a/2 <111> loops at high temperatures. In this study on HT9, in the (narrow) range of temperatures investigated [420–470°C], an
effect of temperature was not really registered since, for a given dose, the relative proportion of the two types of loops was found to be similar at all three temperatures.

Finally, the role of Cr content should also be discussed, since it may impact the mobility of $\frac{a}{2} <111>$ and $a <100>$ loop, and the relative fraction of each type. For instance, in their irradiations of Fe and Fe–Cr systems at 25 and 300°C to $2 \times 10^{14}$ ions cm$^{-2}$ (~1 dpa), Yao et al. [29] observed that $a <100>$ loops predominated in pure Fe, whilst the proportion of $\frac{a}{2} <111>$ loops increased in Fe–Cr alloys, which was attributed to the reduced mobility of $\frac{a}{2} <111>$ loops in Fe–Cr in contrast to pure Fe in which more loops were lost from the foil. The mechanism by which the loop mobility is reduced in the Fe–Cr alloys is still not entirely clear, although Terentyev et al. [31] have shown that a reduction in the mobility of self-interstitial clusters in Fe–Cr alloys relative to pure Fe may be caused by a long-range, attractive interaction between Cr atoms and crowdions.

In their study of UHP Fe irradiated at room temperature in situ to low doses (0.05 to 1 dpa), Schaublin et al. [5,6] reported that only $\frac{a}{2} <111>$ loops were visible at 0.05 dpa whereas $a <100>$ loops were present at 0.45 and 1 dpa, indicating that when more time is given, the $\frac{a}{2} <111>$ loops can move and interact to form the $a <100>$ loops via the ‘111 mechanism’. Interestingly, in their Cr containing systems (Fe–xCr, $x = 5\%$, 10\%, 14\%), $\frac{a}{2} <100>$ loops appeared at the earliest dose of 0.05 dpa, suggesting that Cr actually favours the formation of $a <100>$ loops, which somewhat contradicts the ‘111 mechanism’ because in this view Cr, reducing the mobility of $\frac{a}{2} <111>$ loops, should not favour $a <100>$ loops formation. They suggested possible mechanisms by which Cr could favour the formation of $a <100>$ loops such as a catalytic effect whereby the Cr atoms could be the locations where the $\frac{a}{2} <111>$ loops spontaneously transform to $a <100>$ loops as proposed by Arakawa [27]; however, none of the proposed mechanisms seem to be supported by recent MD simulations [32–35]. The spontaneous transformation to $a <100>$ loops of sessile SIA clusters present at the end of cascade events (including the C15 clusters) was also invoked.

To summarise, in our study of HT9, a complex alloy with 12%Cr and other alloying elements, all the mechanisms of transformation of $\frac{a}{2} <111>$ loops to $a <100>$ loops mentioned in the Section ‘Discussion’ above could be at play, in addition to the possible loss of the $\frac{a}{2} <111>$ loops to the build-up of the dislocation network at higher doses. In fact, during in situ irradiations of foils, we believe that in advanced alloys such as HT9 where the complex dislocation network develops under irradiation, $\frac{a}{2} <111>$ loops are less likely to be lost to free surfaces and more likely to be lost to transformations to $a <100>$ loops via the mechanisms discussed above, and interactions with the dislocation network built-up during irradiation.
5. Conclusions

F/M steel HT9 was irradiated to 20 dpa at 420°C, 440°C and 470°C, in situ, in a TEM with 1 MeV Kr\textsuperscript{2+} ions. The observed phenomena are similar for all three temperatures: (i) black-dot defects start to appear after a small threshold dose; (ii) resolvable dislocation loops appear around 1 dpa and their density increases by 2–3 orders of magnitude from 1 to 4 dpa. Meanwhile, the interaction of the radiation-induced defects with the pre-existing dislocations helps their climb and loss to the free surfaces of the thin foil, resulting in a decrease of dislocation line density in this early stage of irradiation (0–4 dpa); (iii) at higher doses (above 4 dpa), the build-up of a new dislocation network is observed along with the loss of dislocation loops to this dislocation network build-up, resulting in an increase of total dislocation line density and a decrease of dislocation loop density; (iv) at the end of the irradiation, the microstructure is dominated by a complex dislocation network which has no apparent correlation with the pre-existing dislocation lines initially present in the samples and lost to the free surfaces. Overall, the dislocation line density shows an inverse trend to the dislocation loop density with increasing dose for all temperatures investigated.

In terms of loop Burgers vector analysis, both a/2 <111> dislocation loops and a <100> dislocation loops exist in similar proportion at 4 dpa whereas the a <100> type is the predominant type at 20 dpa. With increasing dose, the decrease of dislocation loop density seems to affect more the population of a/2 <111> loops, which are possibly lost (i) to interactions with each other resulting in the formation of more a <100> loops via the ‘111’ mechanism, (ii) to possible direct transformation to a <100> loops and (iii) to the formation and evolution of the irradiation-induced dislocation network. Finally, it is noted that the relative fractions of both types of loops observed in situ at 20 dpa are in agreement with those observed in the same alloy irradiated ex situ (using bulk samples) to the same dose at the same temperatures, i.e. a similar higher proportion of the a <100> loops is reported in both cases.

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Disclosure statement

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