The influence of material purity and irradiation temperature on self-ion damage in molybdenum

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Abstract
An electron microscope study has been made of vacancy loops formed as a result of displacement cascade collapse in molybdenum irradiated with 60 keV self-ions. The experiments were directed towards exposing two aspects of this process. Firstly, the influence of material purity was investigated by comparing the structures generated in three crystals, one being of high purity, the second containing a significantly higher level of substitutional impurities and the third doped specifically with nitrogen. The results demonstrated that nitrogen in solution was the most effective in reducing both the number of visible vacancy loops formed by cascade collapse and the fraction of vacancies surviving in the cascades to form loops. The second major aspect studied was the influence of irradiation temperature on vacancy loop formation in the high purity crystal. In this case the number of visible loops decreased sharply with temperature over the range studied, i.e. up to 535°C. The decrease is shown to follow a different form to that observed in copper and possible mechanisms to account for this are considered. Comparisons of the experimentally observed images in the high purity crystal with those predicted theoretically for pure edge and non-edge loops showed that all of the loops are perfect with $b = \frac{1}{2} \langle 111 \rangle$. Moreover, the loops are mostly non-edge, lying on $\{110\}$ planes in the room temperature irradiated foils, but they rotate towards the pure edge configuration as irradiation temperature increases.

§ 1. Introduction
An important feature of the damage process occurring in metals during neutron irradiation is the high energy primary recoils which generate displacement cascades. It is comparatively simple to simulate the cascade damage produced by the recoils by carrying out irradiation with heavy ions (preferably self-ions) having similar energies, i.e. typically 1–100 keV. Results from such experiments, mostly carried out on pure f.c.c. and b.c.c. metals, have demonstrated that the collapse of the vacancy rich centres of displacement cascades to form planar vacancy defects can play an important role in the development of the damage structure (for a review see Eyre 1973). More recently, a theoretical analysis has shown that this collapse process can have a major effect on the release of vacancies to form voids during irradiation at elevated temperatures (Bullough, Eyre and Krishan 1975). There is, therefore, a need to develop an understanding of the cascade collapse process and, in particular, how it is influenced by irradiation temperature and material composition.
In an earlier paper (English, Eyre and Summers 1975), we reported on the influence of temperature on the numbers and sizes of vacancy loops formed by cascade collapse in self ion irradiated copper. In this paper we report the results of an electron microscope study of self ion damage in molybdenum in which emphasis has been placed on elucidating the effects of temperature and material purity. Previously published work has shown that heavy ion irradiation of both molybdenum and tungsten at room temperature results in the formation of small vacancy loops (Maher 1970, Haussermann 1972 a, b). Haussermann obtained some indication that the fraction of cascades collapsing to form visible vacancy loops, defined as defect yield, and the fraction of vacancies surviving in each cascade to form a loop, defined as cascade efficiency, is influenced by material purity. In the present work we have explored more systematically the influence of both purity and irradiation temperature on the quantitative features of vacancy clusters in molybdenum irradiated with 60 keV molybdenum ions. We have also studied vacancy loop geometry and how it is influenced by irradiation temperature in high purity molybdenum.

§ 2. Experimental procedure

2.1. Specimen preparation

The starting material was Climelt arc cast molybdenum rod supplied by the Climax Molybdenum Co. Specimens of different purities were prepared and in all cases the first step consisted of electron beam zone refining in a vacuum of $5 \times 10^{-6}$ torr to produce seeded single crystals having an [011] axis. The zone refining procedures and subsequent heat treatments, first in a low partial pressure of oxygen to reduce the carbon level to $\leq 1$ atom p.p.m. and then in high vacuum to reduce the gaseous impurities oxygen and nitrogen to a few p.p.m. level, have been described elsewhere (Capp, Evans and Eyre 1975). It was shown that the level of substitutional impurities remaining in the single crystals following the post zone refining heat treatments is a function of the number of zone passes up to four to six passes. This, therefore, provides one method of obtaining specimens of different purity. It has also been shown (Evans and Eyre 1968, J. H. Evans and D. S. Capp, unpublished work) that low levels of interstitial impurities, i.e. carbon, nitrogen and oxygen, can be retained in supersaturated solid solution in molybdenum single crystals by quenching after annealing in the appropriate gaseous atmosphere. This then, provides a second method of varying crystal purity.

<table>
<thead>
<tr>
<th>Material identity</th>
<th>Treatment</th>
<th>$R_{293 , K}/R_{4.2 , K}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>4 PZR</td>
<td>10 300 ± 1000</td>
</tr>
<tr>
<td>B</td>
<td>1 PZR</td>
<td>678 ± 100</td>
</tr>
<tr>
<td>C</td>
<td>6 PZR + doping with nitrogen</td>
<td>1 200 ± 50</td>
</tr>
</tbody>
</table>

Table 1. Resistivity ratios of molybdenum crystals.
The procedure adopted in the present work was to zone refine three [011] crystals giving one, four and six passes, respectively. The crystals were then centreless ground to produce rods 0·3 cm in diameter and these were subjected to the following post zone refining heat treatments: 11 hours at 1880°C and $6 \times 10^{-5}$ torr of oxygen followed by 4 hours at 1800°C in a vacuum of $< 10^{-7}$ torr. The six pass zone refined (6 PZR) crystal was subsequently doped with nitrogen by heating for 2 hours at 1400°C in 1 torr of nitrogen followed by quenching into a liquid metal bath. Activation analysis showed that this crystal contained 17 atom p.p.m. nitrogen. Thus, specimens of three different purities, designated A, B and C, were produced and these are listed in table 1 together with their resistivity ratios $(R_{100K}/R_{4.2K})$ which provide a sensitive comparative measure of their soluble impurity levels. The final stage in the specimen preparation was to slice the single crystal rods into discs 0·3 cm dia. $\times$ 0·05 cm thick which were then electro-polished to perforation to produce specimens suitable for examination by transmission electron microscopy.

### 2.2. Irradiation procedure

The thinned disc specimens of the three materials were irradiated at room temperature to a dose of $7.4 \times 10^{11}$ ions cm$^{-2}$ with 60 keV Mo$^+$ in the 100 keV linear accelerator at Harwell. The high purity material A was also irradiated to the same dose at temperatures of 200°C, 400°C and 535°C. Details of the irradiation procedure have been described elsewhere (English et al. 1975).

### 2.3. Electron microscopy

Following irradiation the specimens were loaded into a double tilt holder so that the damage layer was located adjacent to the electron exit surface of the foil and examined in a Philips EM 300 microscope. In order to facilitate comparison of structures in different specimens sets of micrographs were obtained from the same area in each specimen using standard two-beam imaging conditions as defined by foil thickness, $t$, foil normal, $\mathbf{z}$, operating reflections, $\mathbf{g}$, and Bragg deviation parameter $w_g$. The imaging conditions used can be summarized as follows:

<table>
<thead>
<tr>
<th>$\mathbf{z}$</th>
<th>$\mathbf{g}$</th>
<th>$w_g$</th>
</tr>
</thead>
<tbody>
<tr>
<td>[155]</td>
<td>$\pm [011]$</td>
<td>0 and $\sim 1$</td>
</tr>
<tr>
<td>[023]</td>
<td>$\pm [200]$</td>
<td>0</td>
</tr>
<tr>
<td>[135]</td>
<td>$\pm [211]$</td>
<td>0</td>
</tr>
<tr>
<td>[135]</td>
<td>$\pm [211]$</td>
<td>0</td>
</tr>
</tbody>
</table>

The foil thickness was $\sim 5\xi_{110}$ where $\xi_{110}$ is the two-beam extinction distance for a $\langle 110 \rangle$ type reflection and is equal to 232 Å (Howie and Basinski 1968).

It is well known that small defects located within 1·25$\xi_g$ of a foil surface and imaged under dynamical conditions, i.e. $w_g \sim 0$, exhibit easily observable characteristic black–white images. In the present work we used micrographs obtained under dynamical imaging conditions to determine defect
number in the different specimens. However, large uncertainties exist in the relationship between dynamical images size and true defect size. Defect sizes were, therefore, measured using micrographs taken with \( g = \pm [011] \) and \( w_g = 1.0 \) on which the images appear as black dots irrespective of location. Calculations indicate that in this case the ratio of image size to true defect size is \( 1.2 \) (Rühle 1967 a, b).

With regard to loop geometry, measurement of \( l \), defining the direction of streaking of dynamical black-white images from small dislocation loops, has been widely used to determine Burgers vector directions. This method appears to work well in the case of imperfect edge loops in anisotropic cubic crystals, e.g. Frank loops in copper (Rühle 1967 a, b). However, theoretical image simulation results have shown that the method breaks down in approximately isotropic cubic crystals, with \( l \) deviating away from \( b \) or the projection of \( b \) in the image plane, \( b_p \), as the angle between \( g \) and \( b \) increases (Eyre 1972, Eyre, Maher and Perrin 1974). It has also been emphasized that the \( l \) analysis cannot be applied to determining \( b \) for non-edge loops (Wilkens and Rühle 1972). An alternative, and to some extent complementary approach, is to compare the symmetry of observed black-white images from small loops with theoretically simulated images from known loops calculated using the same diffracting conditions (Eyre 1972, Haussermann et al. 1972, Eyre, Maher and Perrin 1976 a, b). For this method to be successful it is in general necessary to compare experimental and theoretical images for two or more \( g \) s. The image matching approach has been used in the present work to determine the geometry of loops in the high purity material A irradiated at different temperatures. In some cases measurements of \( l \) have also proved useful in obtaining an insight into the relationship between \( b \) and \( n \).

§ 3. Experimental results

3.1. Effect of purity

Micrographs showing the appearance of the damage structures in materials A, B and C are presented in fig. 1. It can be seen qualitatively that both the number density and mean size of visible images decreases as material purity decreases. The number density of visible images were measured on micrographs obtained with \( g = \pm [011] \) and \( w_g = 0 \). To avoid uncertainties in accounting for defects not producing visible images when \( |g \cdot b| = 0 \) we have normalized the image counts with respect to the number obtained for material A. Image sizes were measured on micrographs obtained with \( g = \pm [011] \) and \( w_g \geq 1.0 \). The size distributions for all three materials are plotted in fig. 2 and the mean image sizes are presented in table 2. It can be seen that the fraction of smaller defects increases as material purity decreases and this is reflected by the decrease in mean size by \( \sim 30\% \) on going from material A to material C. The number of vacancies in a defect of mean size was calculated assuming the defects to be perfect loops with \( b = \frac{1}{2}[111] \). If we further assume that each defect is formed by the collapse of a single displacement cascade, we can obtain a mean value for the cascade efficiency by expressing the measured number of vacancies as a fraction of the total number generated in a 60 keV cascade. This was done using the Norgett, Robinson and Torrens (1972) damage model and the results are summarized in table 2.
Fig. 1

Micrographs of the damage created by a dose of $7.4 \times 10^{11}$ 60 keV Mo$^+$ ions/cm$^2$ in (a) material A, (b) material B and (c) material C. Imaged with $g = [011]$. 
Size distributions of the defect images for materials A, B and C. Measured from micrographs with \( w \geq 1.0 \) and \( \mathbf{g} = [011] \).

The important feature of the results is that the fraction of vacancies lost in the cascade during irradiation increases with decreasing material purity.

Table 2. Summary of quantitative data from irradiated crystals.

<table>
<thead>
<tr>
<th>Material identity</th>
<th>Defect numbers in A</th>
<th>Mean defect image size (Å)</th>
<th>Mean cascade efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1-0</td>
<td>33-3</td>
<td>70%</td>
</tr>
<tr>
<td>B</td>
<td>0-32</td>
<td>32-3</td>
<td>64%</td>
</tr>
<tr>
<td>C</td>
<td>0-2</td>
<td>23-0</td>
<td>35%</td>
</tr>
</tbody>
</table>

3.2. Effect of irradiation temperature

Micrographs illustrating the damage structures in material A irradiated at temperatures in the range 20°–550°C are presented in figs. 3 (a)–(c). The measured number of visible defect images normalized with respect to the number in the room temperature irradiated specimen is plotted as a function of irradiation temperature in fig. 4. Figures 3 and 4 both show the rapid
Micrographs of the damage created by a dose of $7.4 \times 10^{11}$ 60 keV Mo$^+$ ions/cm$^2$ in material A irradiated at (a) room temperature, (b) 200°C and (c) 400°C. The diffraction vector $g=[0\overline{1}\overline{1}]$. 
Temperature dependence of the ratio of the number of vacancy loops present after irradiation at temperature $T^\circ C$ to the number present after room temperature irradiation, $N_T/N_0$. Measured from micrographs with $\psi_0=0$ and $\mathbf{g}=[0\overline{1}1]$.

Size distribution of the defect images for material A irradiated at room temperature and 535°C. Measured from micrographs with $\psi_0 \approx 1\cdot0$ and $\mathbf{g}=[0\overline{1}1]$. 
decrease in defect numbers with increasing temperature particularly between 20°C and 400°C. The measured size distributions of the defects for the lowest and highest irradiation temperatures are presented in fig. 5 while the variation in mean defect diameter, together with the total number of vacancies stored in the visible loops, are plotted as a function of irradiation temperature in fig. 6. It can be seen that as irradiation temperature increases, the peak in the size distribution and the mean size shifts to larger sizes.

![Fig. 6](image)

Plot of the dependence on irradiation temperature of the mean defect size and $N_{xT}/N_{x0}$, where $N_{xT}/N_{x0}$ is the ratio of the number of vacancies stored in dislocation loops after irradiation at temperature $T^\circ$C to the number stored after irradiation at room temperature.

With regard to loop geometry a detailed analysis has been made of dynamical black–white images to determine Burgers vector directions for visible defects. The approach adopted has been to compare experimentally observed images with theoretically simulated dynamical images from known edge and non-edge loops located within $0.25\xi_g$ from the electron exit surface of an isotropic b.c.c. crystal (Eyre et al. 1976a, b, Holmes, Eyre and Perrin 1976). In the particular case of perfect edge loops having $b = \frac{1}{2}\langle 111 \rangle$ lying in the plane of foil (so-called edge-on loops) the predicted symmetry varies in a distinctive manner with $|g \cdot b|$. More specifically, three basic image types are obtained when $|g \cdot b| = 0, 1$ and 2, respectively, and these are illustrated for the case of a $b = \frac{1}{2}\langle 111 \rangle$ loop in a foil with $z = [011]$ and imaged with $g = [211], [011]$ and $[211]$ in figs. 7(a)–(c). It can be seen that for $|g \cdot b| = 0$ the image has a characteristic 'butterfly' symmetry (subsequently referred to as type 1), for $|g \cdot b| = 1$ a simple black–white image is obtained (referred to as type 2), while for $|g \cdot b| = 2$ a more complex black–white image having an above background region in the black lobe (referred to as type 3) is obtained. The corresponding images from a non-edge loop again having $b = \pm \frac{1}{2}\langle 111 \rangle$ but with $n = [011]$ are presented in figs. 7(d)–(f). It
can be seen that the $|\mathbf{g} \cdot \mathbf{b}| = 0$ image is now a distorted 'butterfly' (referred to as type 1 (a)) while the basic symmetry of the $|\mathbf{g} \cdot \mathbf{b}| = 1$ and $\mathbf{g} \cdot \mathbf{b} = 2$ images (referred to as types 2 (a) and 3 (a)), respectively, are not changed significantly, although there is some rotation in the direction of the vector $\mathbf{1}$, joining the centres of the black–white lobes (Rühle 1967 a) away from that predicted for the pure edge loops.

Fig. 7

**Edge Loops** $n = \mathbf{b}$

**Non-Edge Loops**

Simulated images of a perfect loop with $\mathbf{b} = \frac{1}{2}[11\bar{1}]$. In (a)–(c) the loops are pure edge with $\mathbf{n}$ and $\mathbf{b}$ coincident in a [111] direction. In (d)–(f) the loop is non-edge with $\mathbf{n} = [01\bar{1}]$. The diffraction conditions are $\mathbf{g} = [01\bar{1}]$ in (a) and (d), $\mathbf{g} = [01\bar{1}]$ in (b) and (e), and $\mathbf{g} = [21\bar{1}]$ in (c) and (f). The scale marker = 90 Å.

In practice the method of analysis consisted of obtaining micrographs from the same area using the [011], [200] and two <211> reflections lying in the (011) plane. Images matching those predicted for $|\mathbf{g} \cdot \mathbf{b}| = 0$ (types 1 and 1 (a)) and $|\mathbf{g} \cdot \mathbf{b}| = 2$ (types 3 and 3 (a)) were in general clearly recognizable on micrographs obtained with $\mathbf{g} = \pm [21\bar{1}]$ thus enabling loops with $\mathbf{b} = \pm \frac{1}{2}[11\bar{1}]$ and $\pm \frac{1}{2}[11\bar{1}]$ to be unambiguously identified. In the case of the specimens irradiated to an ion dose of $7.4 \times 10^{11}$ ions cm$^{-2}$ at room temperature the
Micrographs of material A irradiated with a low dose $\sim 2 \times 10^{11}$ ions/cm$^2$ of 60 keV Mo$^+$ ions at room temperature. Imaged with $u_g = 0$ and in (a) $g = (211)$ and in (b) $g = (211)$. 
visible defect density was high making it difficult to follow individual images from micrograph to micrograph and also the image symmetry was not always very distinct. Nevertheless, examples of types 1 (a) and 3 (a) images were identified for the two \(\langle 211 \rangle\) reflections. However, in specimens irradiated to a much lower dose (2 \(\times\) 10^{11} ions/cm²) it was possible to compared images of individual defects in each reflection, and this is illustrated in fig. 8. It can be seen that each defect appears in only one of the \(\langle 211 \rangle\) reflections and that some images are good examples of image types 1 (a) or 3 (a). As irradiation temperature increased the decrease in image numbers eased the problem of identification and a more detailed analysis was possible for specimens irradiated at 200\(^\circ\)C, 400\(^\circ\)C and 535\(^\circ\)C. Examples of micrographs obtained using [211] and [211] reflections from a specimen irradiated at 400\(^\circ\)C are presented in figs. 9 (a) and (b). Virtually all images corresponded to those theoretically predicted for \(|\mathbf{g} \cdot \mathbf{b}| = 0\) (types 1 and 1 (a)) and \(|\mathbf{g} \cdot \mathbf{b}| = 2\) (types 3 and 3 (a)) illustrated in fig. 7. Examples of these four image types are indicated in fig. 9. Thus, in the 400\(^\circ\)C irradiated specimen we conclude that there is a mixed population of non-edge and pure edge perfect loops having \(\mathbf{b} = \pm \frac{1}{2}[111]\) or \(\pm \frac{1}{2}[111]\). A more detailed comparison of the images with the theoretically simulated images (unpublished work) shows that the majority of the non-edge loops lie on a plane between (011) and (111) or (111) in agreement with the work of Jäger and Winkel (1975) on vacancy loops in an [011] tungsten crystal. In the case of the specimens irradiated at 200\(^\circ\)C all of the images corresponded to those predicted for non-edge \(\pm \frac{1}{2}[111]\) and \(\pm \frac{1}{2}[111]\) loops whereas for the specimen irradiated at 535\(^\circ\)C a majority of the images corresponded with those predicted for pure edge loops again having \(\mathbf{b} = \pm \frac{1}{2}[111]\) or \(\pm \frac{1}{2}[111]\). It is emphasized that no defects were found which gave images with a well-defined \(\mathbf{l}\) vector in both the \(\langle 211 \rangle\) reflections. Thus no evidence was found for the presence of the inclined perfect loops having \(\mathbf{b} = \pm \frac{1}{2}[111]\) or \(\pm \frac{1}{2}[111]\), or of any imperfect loops of the type \(\mathbf{b} = \frac{1}{2} \langle 110 \rangle\).

§ 4. Discussion

Considering first the question of material purity, the present results have demonstrated that it has an important influence on the collapse of displacement cascades to form visible vacancy loops both in terms of defect yield and cascade efficiency. Results obtained by Capp et al. (1975) showed that the main effect of varying the number of zone passes in the electron beam zone refiner is to change the level of substitutional impurities. Thus, the results summarized in table 2 indicate that interstitial nitrogen is more effective than substitutional impurities in reducing defect yield and cascade efficiency. Before discussing possible mechanisms, it should be noted that the present results are consistent with those obtained by Haussermann (1972 a). He studied two sets of molybdenum specimens having resistivity ratios of 130 and 430, respectively, irradiated with 60 keV Au++. In both cases Haussermann measured values for the defect yield which are lower than that found for material A but comparable to the values for materials B and C. His measured size distributions and mean defect sizes were also in close agreement with those measured for materials B and C.
Micrographs of material A irradiated at 400°C with $7.4 \times 10^{11}$ 60 keV Mo$^+$ ions/cm$^2$. Imaged with $\varphi = 0$ and in (a) $g = [211]$ and in (b) $g = [211]$. 
With regard to the mechanisms involved, there are three possibilities to consider. Firstly, the impurities could defocus replacement collision sequences, thus depositing interstitials closer to the cascade centres and increasing the probability of their recombination with vacancies. This mechanism implies that the reduction in defect yield and cascade efficiency is a consequence of a reduction in the point defect survival rate during irradiation. Secondly, the impurities might trap vacancies in the cascade centres, thus inhibiting their migration and collapse to vacancy loops. Thirdly, the impurities could directly influence the collapse process resulting in the vacancies aggregating to form sub-microscopic voids rather than loops. The second and third proposed mechanisms imply that the reduction in defect yield and cascade efficiency is a consequence of the damage not being visible rather than an enhancement in interstitial–vacancy recombination.

Although the existing evidence does not allow any of the proposed mechanisms to be positively eliminated, it is considered that the second and third mechanisms are less likely. More specifically, apart from tungsten, other impurities are present at the few p.p.m. level and therefore the number included in any given cascade will be much lower than the number of vacancies generated. Thus, the probability of impurities trapping vacancies is low. Moreover, although nitrogen, being a gas, may promote void rather than loop formation in the cascades, it is not thought generally that substitutional impurities are effective in this respect.

Turning now to consider the observed variation in visible vacancy defect numbers with irradiation temperature, a particularly striking feature of the results is the marked decrease over the lower temperature range. These observations are consistent with results obtained from neutron irradiated molybdenum (Maher, Loretto and Bartlett 1971, Bentley, Eyre and Loretto 1976), although in this latter case the decrease in vacancy loop numbers is accompanied by the formation of a fine distribution of voids at temperatures \(\leq 300^\circ\text{C}\). On the other hand, the present results are not consistent with observations made on self ion irradiated copper (English et al. 1975) in which the vacancy loop population remains constant up to \(\sim 300^\circ\text{C}\) and then decreases sharply at higher temperatures. In this case the results can be explained by a model in which the loop generation rate by cascade collapse is temperature independent and this is counter-balanced at the higher temperatures by thermal vacancy emission controlled shrinkage of the loops.

Considering possible explanations to account for both the present results and the observations on neutron irradiated molybdenum, we can rule out any model involving cascade collapse to loops followed by thermal vacancy emission since the loop population would then be expected to remain constant up to \(700^\circ\text{C}–800^\circ\text{C}\). This is supported by results from annealing experiments in which specimens irradiated at room temperature were subjected to a 1 hour anneal at \(400^\circ\text{C}\). No significant changes were observed in the vacancy loop population following this anneal. A second possible explanation of the present observations is that an increasing fraction of the loops are lost by glide to the incident surface as irradiation temperature increases. However, as we shall discuss later, analysis of loop geometry showed that a majority belong to one of the edge-on sets having \(b\) lying in the (011) crystal surface and thus, there is no resolved image force attracting the loops out of the
specimen. It is considered that any inclined loops having \( b = \pm \frac{1}{2}[111] \) or \( \pm \frac{1}{4}[111] \) glide out of the specimen during irradiation irrespective of temperature. In any case, such an explanation does not account for the observations on neutron irradiated molybdenum and in particular, the replacement of the loop population by voids. This observation also rules out the possibility that the reduction in loop numbers is a consequence of increased vacancy-interstitial recombination in the cascades as irradiation temperature increases due, for example, to thermal defocusing of replacement collision sequences.

We are led finally to the conclusion that the process of cascade collapse to form vacancy loops is strongly temperature dependent in molybdenum. The mechanism of collapse is not well understood and an important question concerns the processes involved in the nucleation and growth of loops, particularly at temperatures below that for long-range vacancy migration. In this discussion we shall limit ourselves to identifying in general terms some of the important factors involved in cascade collapse. As has already been mentioned, the evidence from the geometric analysis of the loops shows that they nucleate on \( \{110\} \) planes presumably in the faulted configuration with \( b = a/2\langle 110 \rangle \). It is generally accepted that molybdenum, along with other b.c.c. metals, has a high stacking fault energy which means that the activation barrier to the nucleation of faulted loops is high. On the other hand, there are two important contributions to the driving force for the aggregation of vacancies in the cascade. First, the local supersaturation in the cascades is initially extremely high. Second, the interstitials injected into the surrounding crystal will exert a pressure on the vacancy rich cascade centres which will favour loop formation in particular. These two factors could result in a reduction in the effective vacancy migration energy within the cascade by decreasing the activation volume component of the migration energy. The energy balance, therefore, lies between the energy of a critical sized faulted loop nucleus, which is dominated in b.c.c. metals by the stacking fault energy, and the chemical and elastic strain energy terms in the uncollapsed cascade. Clearly, as temperature increases and vacancies can migrate into surrounding crystal, the driving force for cascade collapse will be reduced and this could account for the observed reduction in loop numbers in molybdenum at temperatures as low as 200°C. In f.c.c. metals, such as copper, on the other hand, the energy barrier to faulted loop nucleation is much lower which could account for why cascade collapse to loops appears to be temperature independent. A more detailed quantitative discussion of these ideas will be deferred to a later paper.

With regard to loop geometry, the present analysis results have established a number of important features regarding Burgers vector directions and habit planes. First, evidence was only obtained for loops belonging to the two edge-on sets having \( b \)’s lying in the \( (011) \) foil plane. Second, the results of comparing image symmetry with that predicted theoretically was consistent with all of the loops being non-edge in the specimens irradiated at 20° and 200°C and with there being an increasing fraction of pure edge loops in the specimens irradiated at 400° and 535°C. Third, although the analysis was not exhaustive, it indicated that the habit plane for a majority of the non-edge loops was close to the perpendicular \( (011) \) plane. The results are therefore consistent with loops nucleating in the faulted configuration with
\( b = \frac{1}{2}\langle 110 \rangle \) and unfauling at an early stage to \( a/2\langle 111 \rangle \) as predicted by Eyre and Bullough (1965). The absence of loops belonging to the two inclined sets with \( b = \frac{1}{2}\langle 111 \rangle \) or \( \frac{1}{2}\langle 111 \rangle \) is consistent with those gliding out of the foil during irradiation due to surface image forces. More importantly, the absence of any evidence for loops lying on the inclined \( \langle 110 \rangle \) planes suggests that loops nucleated on these planes always shear to one of the \( \frac{1}{2}\langle 111 \rangle \) Burgers vectors and then glide to the surface. This is consistent with shear always occurring in a direction towards the free surface as predicted by Haussermann et al. (1972). Thus, if we assume that loops nucleate on all six \( \langle 110 \rangle \) planes with equal probability only a sixth of them remain in the foil. Clearly this should be taken into account when deriving defect yields from observed defect numbers. In low stacking fault energy, f.c.c. metals such as copper and gold on the other hand, the vacancy loops remain faulted, at least during room temperature irradiation, and none escape by glide to the specimen surface.

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**References**


