THE EFFECT OF POST-IRRADATION ANNEALING ON STACKING FAULT TETRAHEDRA IN NEUTRON-IRRADIATED OFHC COPPER - D. J. Edwards (Pacific Northwest National Laboratory)*, B. N. Singh and M. Eldrup (RISØ NATIONAL LABORATORY, DENMARK)

OBJECTIVE

Evaluate the annealing behavior of stacking fault tetrahedra (SFT) formed during neutron irradiation in OHFC copper irradiated at 200 and 250°C.

SUMMARY

Two irradiation experiments have been completed wherein two sets of tensile specimens of OFHC copper were irradiated with fission neutrons, one set at 200°C and the other at 250°C. Post-irradiation annealing in vacuum was then used to evaluate the change in the defect microstructure, including vacancy-type SFT, voids, and dislocation loops. Individual samples within each set were given one annealing exposure at 300, 350, 400, 450, 500, or 550°C for 2 hours. The fine-scale defect microstructure was characterized by transmission electron microscopy (TEM) to compare the defect size and spatial distribution at each annealing temperature and reference the results to that measured in the as-irradiated condition.

Based on the change in the SFT size distributions, post-irradiation annealing led to a preferential removal of the smaller sized SFT, but did not lead to a general coarsening as might be expected from an Oswald ripening scenario. The issue of whether the SFT produced during irradiation are all structurally perfect is still being investigated at the time of this report, however, the images of the SFT appeared more perfect after annealing at 300°C and higher. Further analysis is being performed to determine whether intermediate stages of SFT formation exist in the as-irradiated condition.

PROGRESS AND STATUS

Introduction

Post-irradiation annealing is a useful tool to explore the stability of defect agglomerates in irradiated materials. One of the issues that has arisen recently is how do stacking fault tetrahedra interact with mobile dislocations generated during deformation. This issue arose during attempts to model the clearing of defects produced during irradiation when cleared channels form inside neutron irradiated copper deformed in tensile tests. Analysis of such interactions by Wirth et al. [1] and by Hiritani et al. [2] revealed that a mobile dislocation interacts differently with an SFT depending on whether the SFT is in a perfect configuration or is truncated. These efforts have pointed to the importance of understanding more about the nature of the SFT produced during irradiation.

By annealing neutron irradiated samples of pure copper, insight can be gained into the relative stability of SFT and how they interact with other defects within the irradiated material. An earlier post-irradiation experiment by the authors [3] revealed that the SFT were very stable, with a large fraction of the defects surviving after 50 hours of annealing at 300°C, a temperature well above recovery Stage V. Above recovery Stage V the vacancy-type defects become increasingly unstable and emit vacancies into the lattice. From this experiment it was observed that the images of the SFT became more easily discernible after annealing, partly in response to a slight reduction in density and increase in average size. However, an alternative, additive reason for this observation is that the more perfect triangular appearance of the SFT after annealing may have been aided by the preferential removal of SFT that were not structurally perfect or were partially dissociated loops. The release of vacancies from these defects may have been

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partially absorbed by surrounding, closely-spaced SFT. This question of whether non-perfect SFT exist in the as-irradiated material is an important issue for describing the annealing kinetics of SFT in irradiated copper as well as how the defects interact with mobile dislocations.

To further explore this issue, an annealing experiment has been performed on samples of OFHC copper that were neutron irradiated at two different temperatures, 200°C and 250°C, in the DR-3 reactor in Denmark. These two irradiation temperatures were chosen in order to compare the effect of having an additional high density of vacancy sources/sinks (a moderate density of voids form at 250°C) present in the microstructure along with the vacancy-type SFT. An earlier semi-annual report [4] briefly described this experiment and the results obtained after annealing the samples irradiated at 200°C. In the following report the results obtained from annealing both sets of samples irradiated at the two temperatures will be presented and compared.

Experimental Procedure

Irradiation experiments have been completed wherein two sets of tensile specimens of OFHC copper were irradiated with fission neutrons in the DR-3 reactor in Denmark, one set at 200°C and the other at 250°C, to a total dose of 0.3 dpa. The post-irradiation annealing was performed by taking individual samples from each set and annealing (in vacuum) each sample at either 300, 350, 400, 450, 500, or 550°C for 2 hours. Positron annihilation spectroscopy was conducted on each annealed sample as well as on the specimens in the as-irradiated conditions. Results of those experiments will be described in later reports. After the PAS was completed, 3-mm disks were punched from each condition and electropolished using a solution of 25% perchloric acid, 25% ethanol and 50% water at 11V for about 15s at ~20°C. The microstructure of each condition was characterized in a JEOL 2000FX transmission electron microscope. The SFT and void images were taken by imaging near a <011> zone axis oriented along the 200 direction in either weak beam dark field or bright field kinematical conditions for the SFT and voids, respectively. Dislocation and loop images were obtained from the same zone axis orientation, but imaged using the [111] reflection.

Results

The specimens irradiated at 200°C contained a high density of small SFT and small SIA (self interstitial atom) loops. Those specimens irradiated at 250°C contained an additional microstructural component in the form of voids with an average size of ~15 nm and a density of 3.5 x $10^{20}m^{-3}$. The SFT microstructure formed both at 200 and 250°C proved to be very stable, with a significant density of defects remaining up to 400°C, after which their density began to decrease rapidly. The measured size distributions for the SFT (and voids at 250°C) are plotted in Figures 1 through 3 to illustrate the changes that occurred due to annealing after irradiation. Images of the SFT are shown in Figures 4 and 5 comparing the effect of annealing to the SFT imaged in the as-irradiated samples.

For samples irradiated at 200°C, a gradual decrease in SFT density occurred up to ~400°C, after which the decrease was more pronounced (see Figure 1). Annealing at 550°C for 2 hours removed all of the defect clusters produced at 200°C, including the dislocation loops, and yielded a well-annealed microstructure with only a few line dislocations. The annealing response of the SFT formed at 200°C cannot be considered as Oswald ripening, but as a reduction in the total number of vacancies contained within the SFT with only a slight coarsening that did not exceed a maximum size. The samples irradiated at 250°C possessed a reasonable density of cavities (~5 x 10^{20} m^{-3}) that seemed rather immune to the effects of annealing up to 450°C (see Figure 3). The density of SFT after irradiation at 250°C was lower than that measured at 200°C. The annealing response for samples irradiated at 250°C (Figure 2) was somewhat different than observed for samples irradiated at 200°C in that the size distributions shifted to the larger sizes when annealed at 300 and 350°C, indicating a real growth of SFTs. On the other hand, when annealed at 400°C and above, the density began to decrease substantially with little change in the

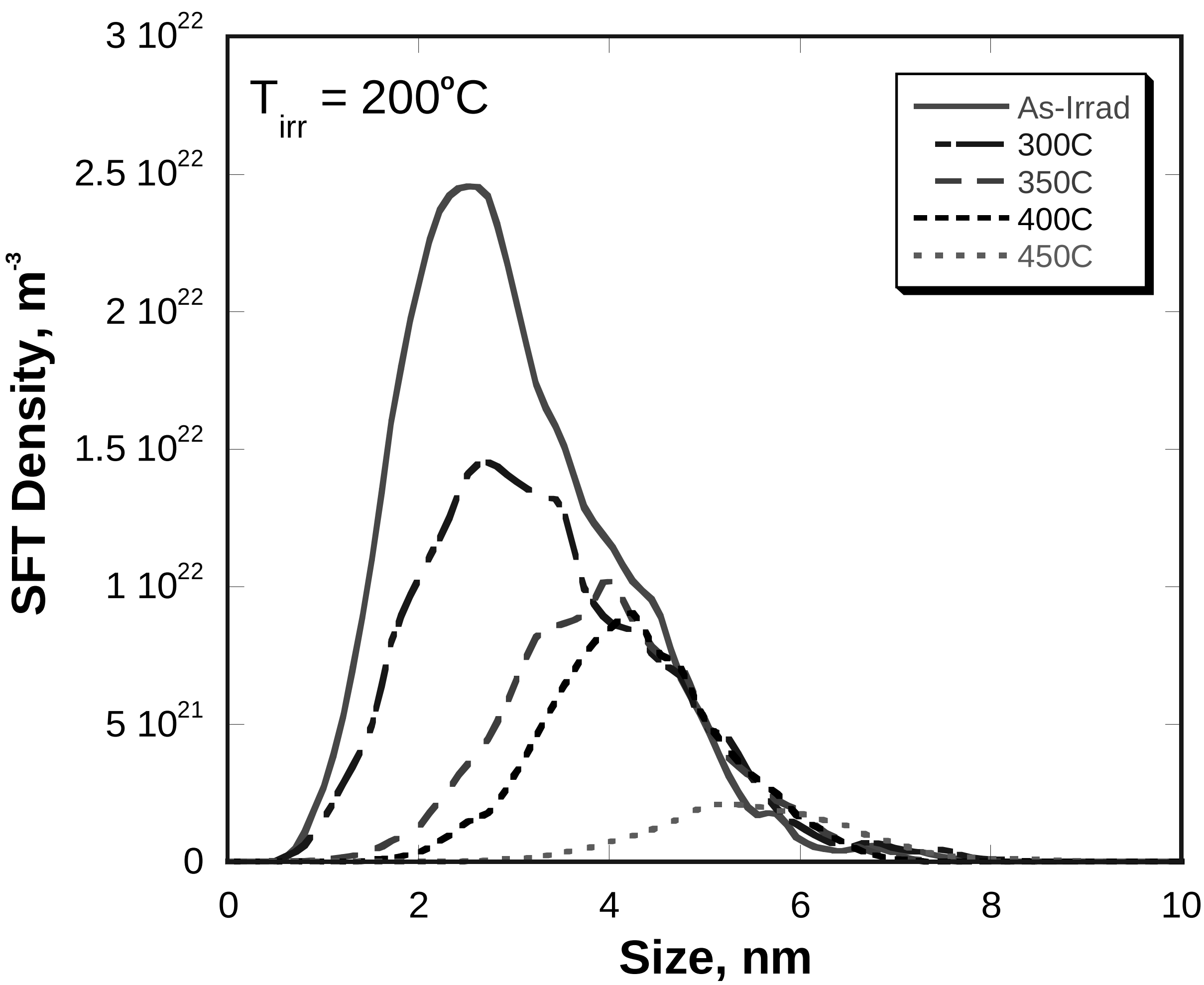

Figure 1. Size distributions for the SFT in OFHC copper irradiated at 200°C and exposed to different annealing temperatures for 2 hours. Note that the size distributions do not indicate a general coarsening such Oswald ripening, but rather the preferential removal of the smaller SFT.

peak of the size distribution. Annealing a 250°C irradiated sample at 500°C for 2 hours produced a very heterogeneous distribution of both SFT and voids, with some grains completely free of defects of either type. The role of the voids remains unclear, but their influence will have to be accounted for in the modeling of the annealing behavior of the SFT. It is interesting to note that the size of the smallest SFT survive during annealing increase with increasing annealing temperature. Furthermore, the mean size of the SFT surviving during annealing increases with increasing annealing temperature. Both of these observations are significant since in the as-irradiated condition neither the size of the smallest SFT nor the mean size measured after irradiation are affected in any substantial way by the irradiation temperature.

Unfortunately, the issue of whether the SFT are structurally perfect cannot be addressed adequately from the images obtained thus far. Higher magnification images are needed under better controlled imaging conditions to be able to reliably determine whether the small SFT are indeed perfect or not. Thickness fringes appearing in the stacking fault of the SFT complicate the images of the SFT, in some cases giving the appearance of small Frank loops with a triangular strain field when it may in fact just be a bright

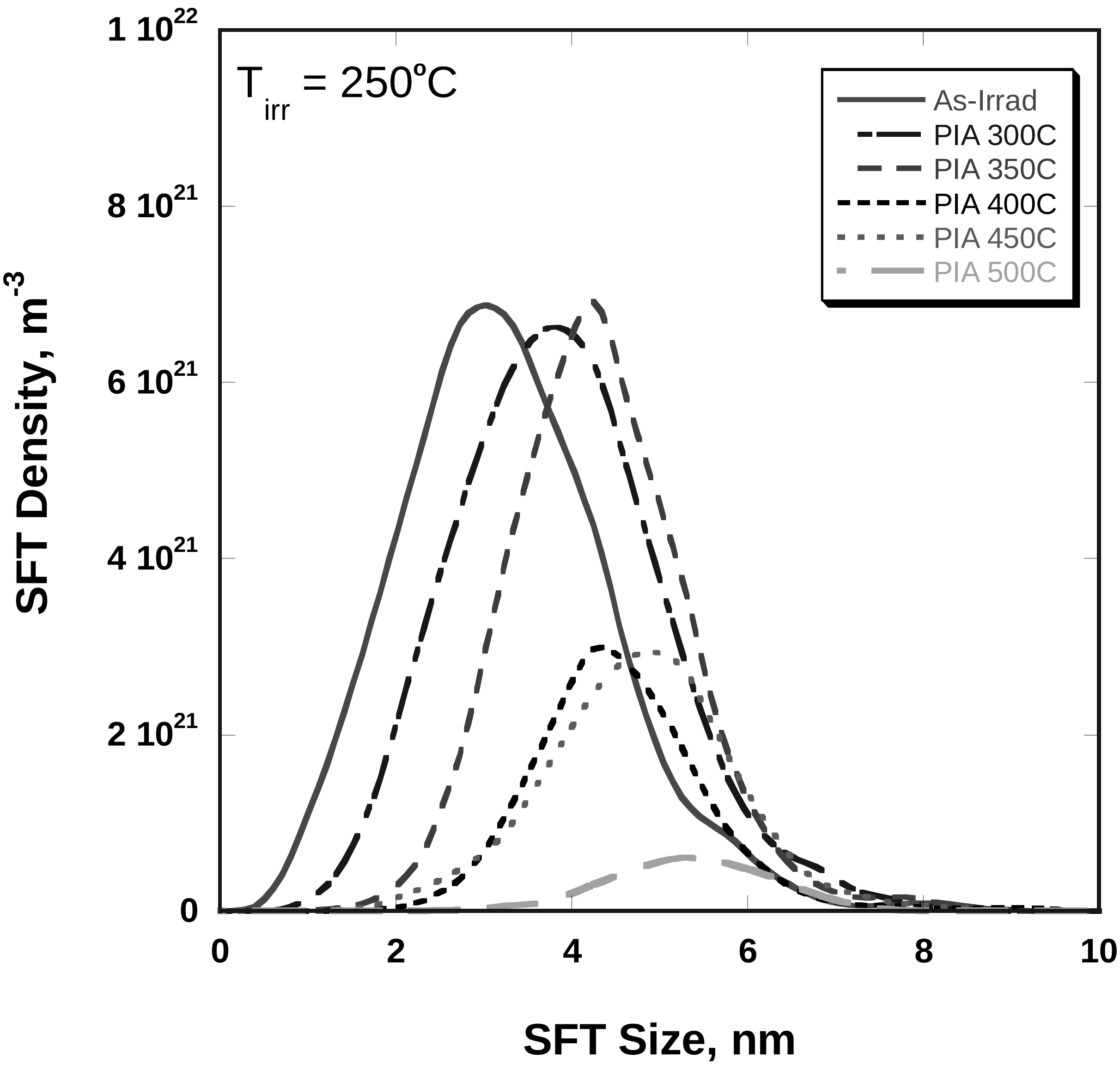

Figure 2. Size distributions for the SFT in OFHC copper irradiated at 250°C and exposed to different annealing temperatures for 2 hours. Compared to the annealing response shown in Figure 1, coarsening up to 350°C, then the smaller SFT begin to disappear at higher annealing temperatures.

thickness fringe. A more careful analysis of the SFT is needed to study this issue and find more suitable imaging conditions and criteria.

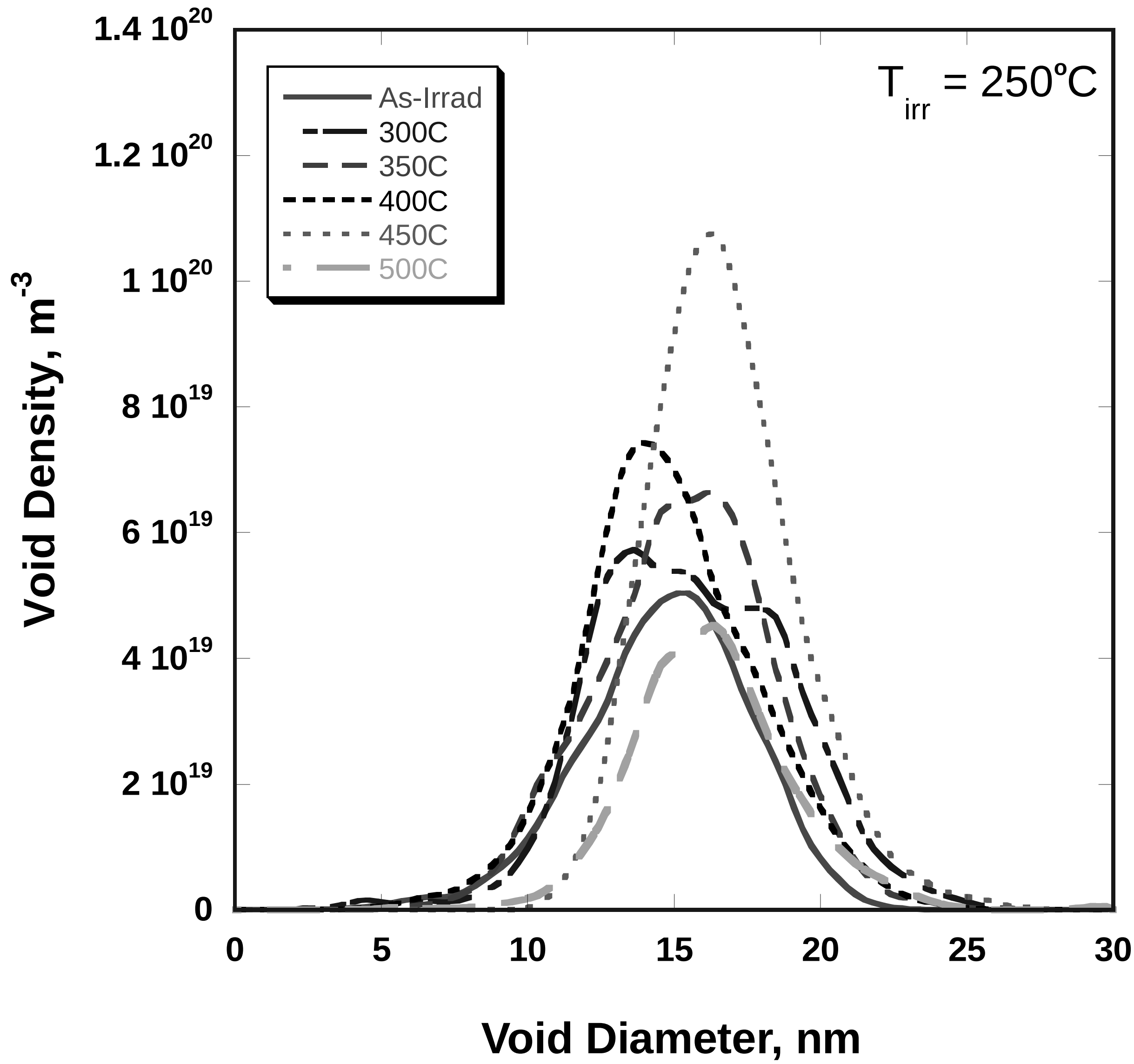

Figure 3. Size distributions for the cavities formed in OFHC copper after irradiation at 250°C and exposed to different annealing temperatures for 2 hours. The higher peak density after annealing at 450°C is being evaluated further to see if it is a real behavior or simply a scatter in the density measurements.

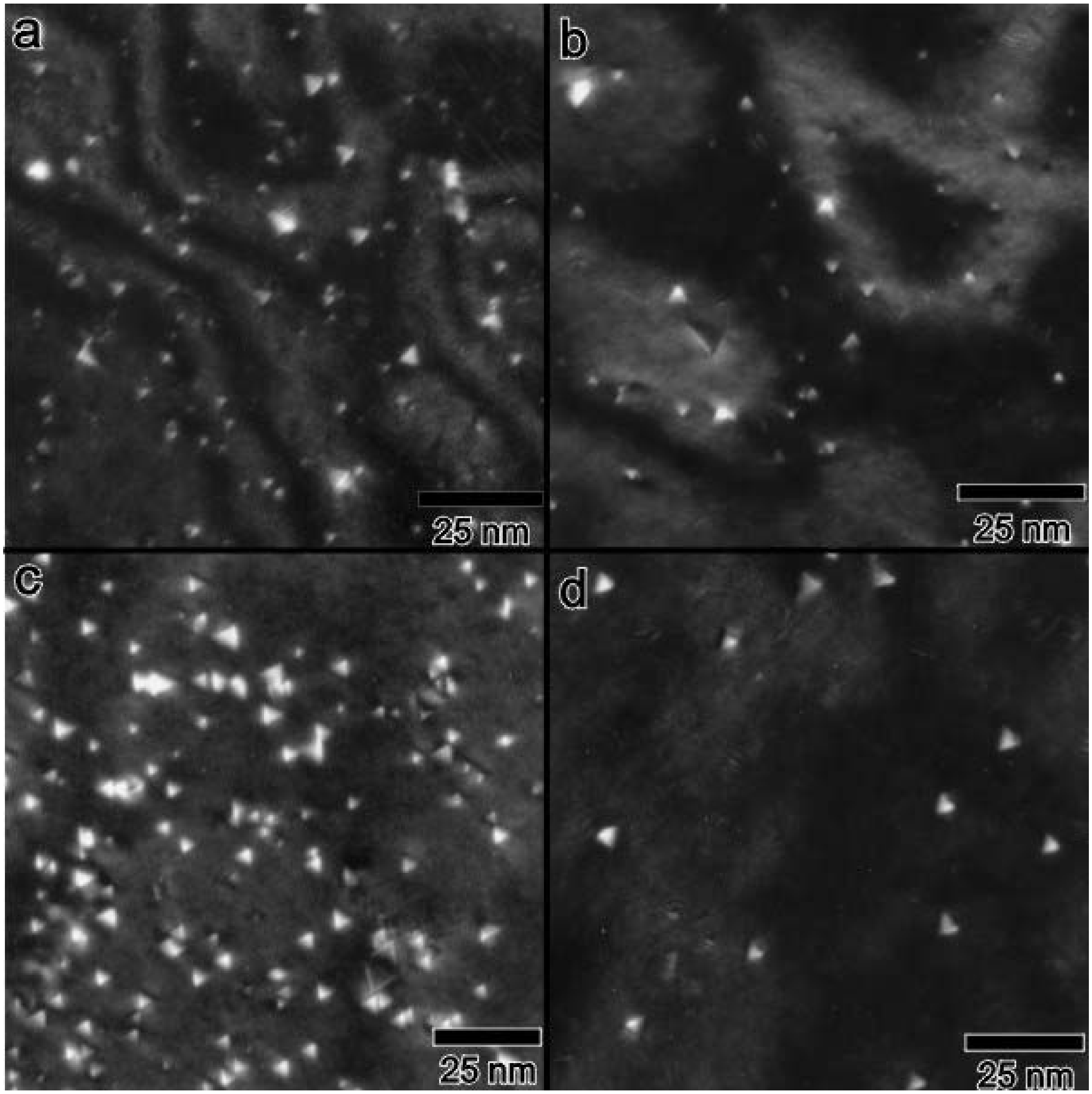

Figure 4. Images of the SFT formed in OFHC copper after irradiation at 200°C and exposed to different annealing temperatures for 2 hours. The image shown in (a) represents the as-irradiated condition, whereas the images provided in (b), (c) and (d) are from samples post-irradiation annealed at 300, 350 and 450°C, respectively. The area in (c) is thicker than the areas shown in the other images, hence the apparent higher density of SFT.

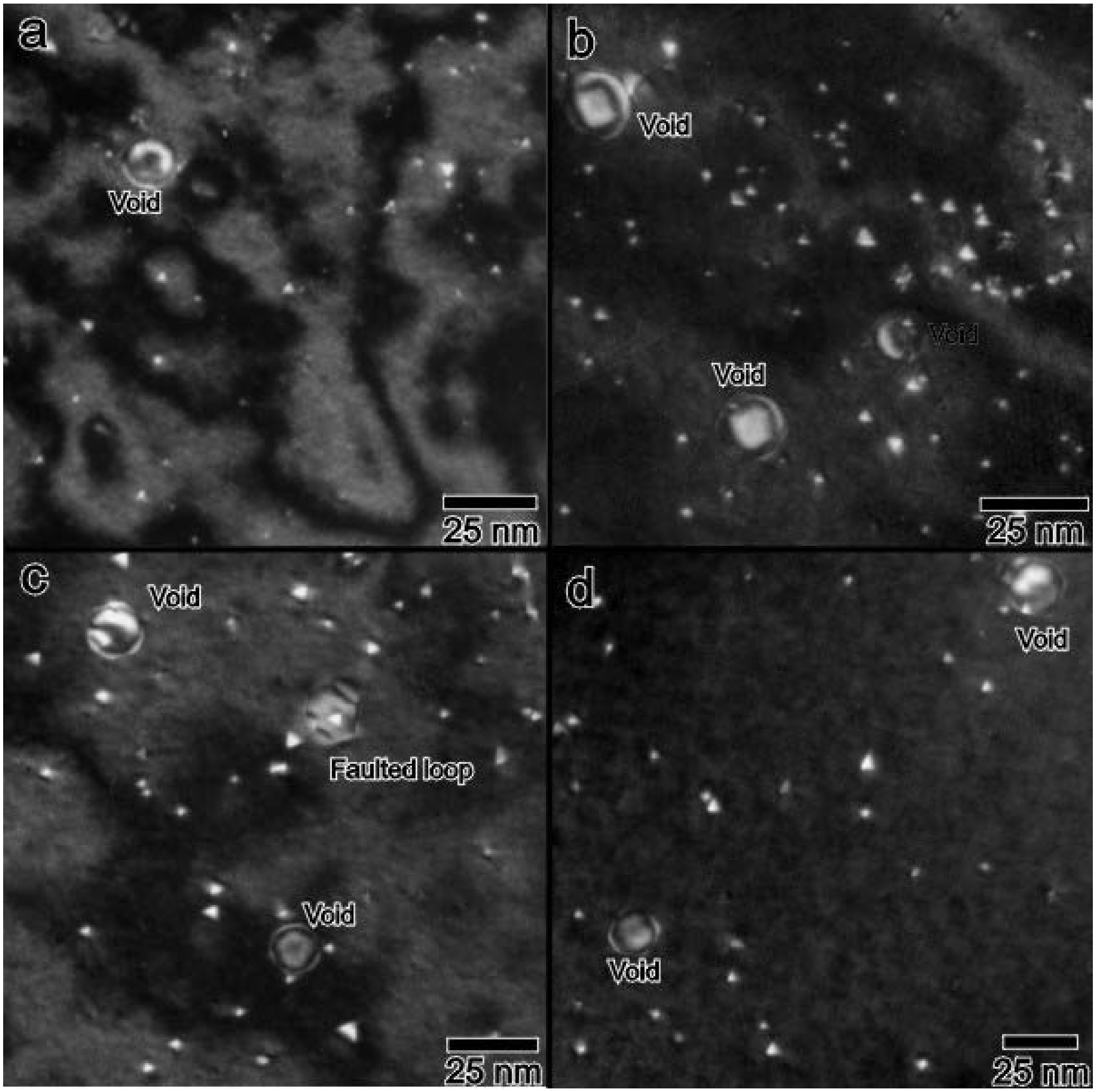

Figure 5. Images of the SFT formed in OFHC copper after irradiation at 250°C and exposed to different annealing temperatures for 2 hours. The image shown in (a) represents the as-irradiated condition, whereas the images provided in (b), (c) and (d) are from samples post-irradiation annealed at 300, 350 and 450°C, respectively. Voids and a large, faulted dislocation loop are indicated in the figure.

Future Work

Modeling of the annealing response of the SFT is in progress, and will have to address a number of issues to describe accurately the annealing response. These issues include the possibility that some fraction of the SFT are not perfect in the as-irradiated condition, that is, some of the SFT may be truncated. The presence of small vacancy-type Frank loops or intermediate defect configurations (partially-dissociated Frank loops) needs to be considered also. Another issue is that the relative structural perfection of the SFT may play a strong role on determining their annealing behavior. The spatial distribution is deemed an important factor since the relative close spacing of the defects produced at 200°C yields a small mean free path that may promote a simple exchange of vacancies between SFT for intermediate annealing temperatures and annealing times. The details of the microstructure will continue to be analyzed, including a measurement of the line dislocation and dislocation loop density. Other samples of OFHC irradiated at 200°C or less are going to be reevaluated to see if the images can provide any insight into the issue regarding the perfection of the SFT.

Acknowledgements

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