

In-Situ TEM He⁺ Implantation and Thermal Aging of Nanocrystalline Fe

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A key aspect in predictively modeling the response of materials exposed to many radiation environments is understanding the role of light transmutation products. He in particular can result in the swelling and precipitation of bubbles, both of which can substantially deteriorate the mechanical properties [1]. In this study, *in situ* TEM characterization of nanocrystalline Fe samples implanted with 10 keV He⁺ is performed to understand and quantify the mechanisms underlying He diffusion and cavity nucleation under a wide temperature range

Nanocrystalline free-standing Fe thin films were produced by pulse laser deposition and annealed *in situ* at 550°C in order to stabilize the grain structure for the subsequent experiments. Two types of *in situ* experiments were performed to study defect evolution and cavity formation: (1) He implantation at room temperature followed by annealing to 600°C, and (2) He implantation at elevated temperatures up to 600°C. *In situ* He implantations were carried out utilizing a JEOL 2100 TEM and a 10 kV Colutron ion accelerator that are part of the *In situ* Ion Irradiation TEM facility at Sandia [2]. The implantation occurred in a hummingbird heating stage tilted 40° towards the He beam operated at an average flux of approximately 10¹⁴ He⁺/cm²s to a total concentration of ~4% He at the end of range.

Figures 1(a-d) illustrate bright field TEM images of He implanted Fe at (a) room temperature, (b) 200°C, (c) 400°C and (d) 600°C. It was observed that He implantation at room temperature and 200°C resulted in the formation of dislocation loops with a maximum size of approximately 3 nm. Implantation at 400°C also resulted in the formation of dislocation loops, but with much larger sizes (16 nm max). In contrast, at 600°C, first where He implantation occurred at room temperature followed by annealing to 600°C, and second where He implantation occurred directly at 600°C, Fresnel imaging revealed the presence of nanometer sized voids in both cases. Figure 2(a) shows an under-focus image of the room-temperature implanted sample after annealing to 600°C where the cavities appear to be evenly distributed through the grains. In the under-focus image of the sample implanted directly at 600°C presented in Figure 2(b), the observed behavior is drastically different since cavities are seen only along grain boundaries. The experimental observations suggest different mechanisms are active during He⁺ implantation under sequential versus simultaneous He⁺ implantation and annealing. These results will be compared to other existing experimental observations and defect evolution models in order to provide insights into the various mechanisms contributing to the different behaviors.

References:

[1] G.S. Was in “Fundamentals of Radiation Materials Science”, (Springer).

[2] K. Hattar, *et al.* Nuclear Instruments and Methods in Physics Research B **338** (2014).

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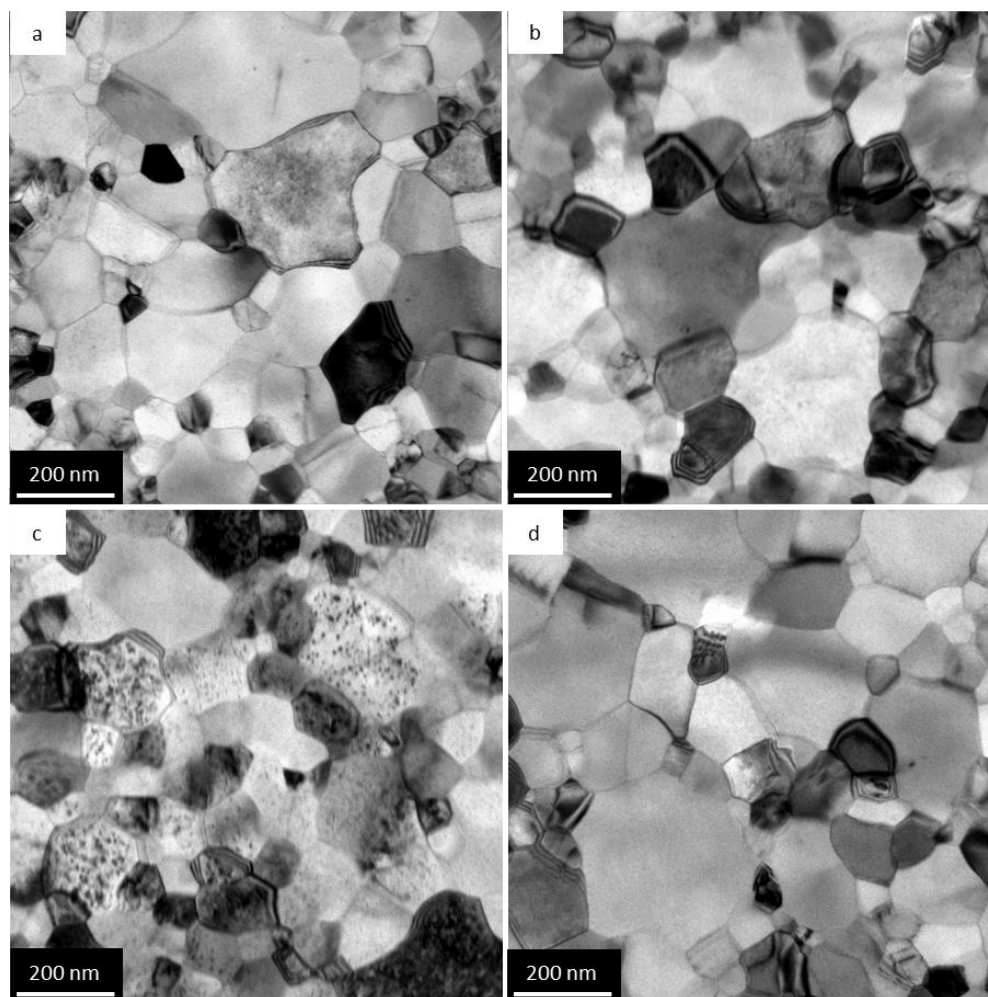


Figure 1. He⁺ implanted Fe implanted at (a) room temperature (b) 200°C (c) 400°C (d) 600°C.

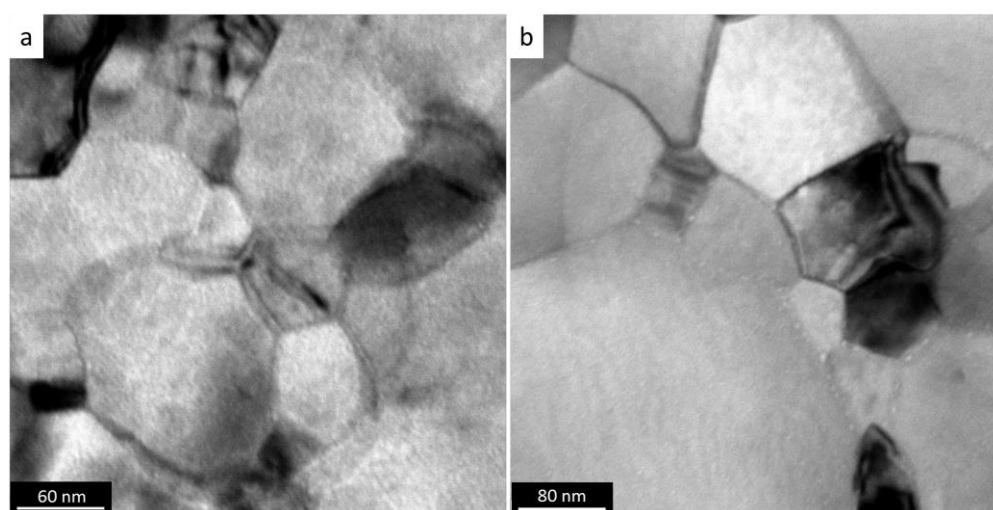


Figure 2. Under-focus images of cavities at 600°C in samples implanted at (a) room-temperature and post-implantation annealed (b) 600°C.