

Bubble Formation in Er and ErD₂ During *In Situ* He⁺ Ion Implantation

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A fundamental characterization and understanding of the response of a material to extreme conditions is important for validation before a part is placed in service in a real radiation environment. Gases resulting from implantation or internal fission products frequently appear [1], and produce well-known detrimental effects in material performance. Near the surface, gases may precipitate out as blisters, leading to undesirable surface structures, while more deeply implanted gases may form bubbles within grains or segregate to grain boundaries, leading to swelling and embrittlement. Previous studies of He-implanted Er films have revealed bubble formation and lattice expansion behavior sensitively dependent on the implanted He dose and post-implantation anneal time [2, 3]. While these studies provided information about the overall structural changes caused by He implantation and annealing, their insight into the microstructural mechanisms involved during the implantation and coalescence processes is more limited.

One tool capable of resolving microstructure evolution on both the time and length scales needed to study He bubble growth is *in situ* irradiation transmission electron microscopy [4]. The *in situ* ion irradiation TEM (I³TEM) facility at Sandia National Laboratories permits observation of samples while under bombardment by ions from a Colutron G-1 accelerator (0.5-10 kV). In the energy range provided by this accelerator, a significant fraction of He⁺ ions stop within a TEM foil, allowing observation of the formation, coalescence, and growth processes of gas bubbles and voids. Er and Er deuteride (ErD₂) samples were prepared by a FIB lift out technique. The samples were implanted *in situ* at room temperature with 10 keV He⁺ at a flux of 2.9×10^{13} ions/cm²/s to a maximum fluence of 1.7×10^{17} ions/cm². In order to separate out the effects of the electron beam, one area was observed continuously during implantation, while a second area was imaged at doses of 1.7, 17, and 170×10^{15} ions/cm² (corresponding to irradiation for 1, 10, and 100 minutes). After implantation, the samples were subjected to *in situ* annealing to a maximum temperature of nominally 790 °C using a Gatan heating stage in a Philips CM30 TEM.

Bubble/void formation within grains and along grain boundaries was observed by 1.7×10^{15} ions/cm² (Figure 1a,b). Increasing fluence to 1.7×10^{16} ions/cm² resulted in a slight increase in bubble density, but after that point bubble size and density remained essentially stable up to the maximum tested fluence of 1.7×10^{17} ions/cm² (Figure 1c), suggesting that much of the implanted He was initially accommodated within the lattice. Implanted sample microstructures were essentially unchanged for one week at room temperature. This stability supports the critical dose and critical temperature for bubble formation determined in previous studies [5]. Post implantation *in situ* annealing revealed little bubble activity prior to a nominal temperature of 785 °C. Upon holding at this temperature, rapid bubble coalescence was observed over several minutes (Figure 1d), followed by a period of apparent stability [6].

References

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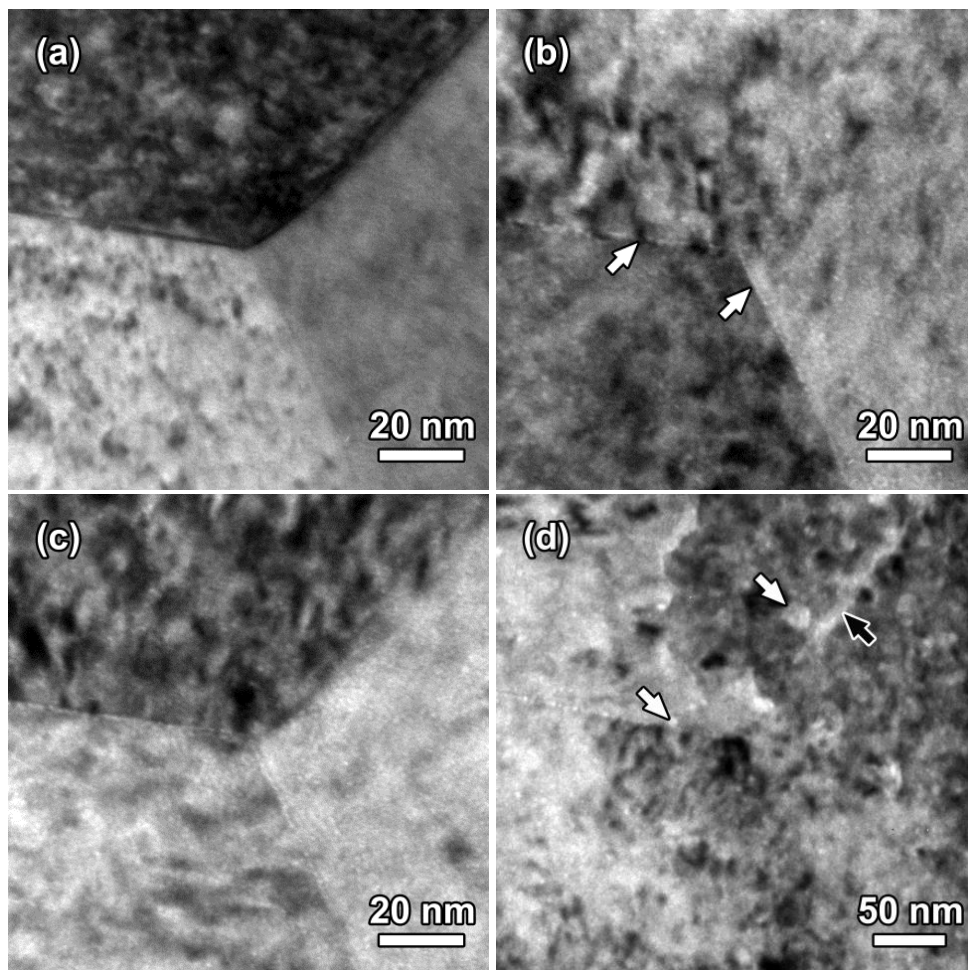


Figure 1. Transmission electron micrographs of the Er/ErD₂ sample showing (a) a triple junction before implantation. (b) In the same area after 10 keV He⁺ ion fluence of 1.7×10^{15} ions/cm², small bubbles were present in the grains and along grain boundaries (indicated by arrows). (c) The triple junction again after 1.7×10^{16} ions/cm², showing little noticeable change. (d) The same area at lower magnification after 1.7×10^{17} ions/cm² and *in situ* annealing to 785 °C, showing larger coalesced bubbles and voids within the grain (white arrows) and along grain boundaries (black arrow). Micrographs (b-d) were collected in an under focused condition to reveal the presence of bubbles and voids (white dots).