3- 11 Transmission Electron Microscopy Investigations of Bubble Formation along GBs in He-implanted Polycrystalline SiC

Li Bingsheng, Wang Zhiguang, Du Yangyang, Wei Kongfang,
Yao Cunfeng, Sun Jianrong and Zhang Hongpeng

Because of the low cross-section for neutron capture and its excellent structural, chemical and mechanical stability, silicon carbide (SiC) is an important material with application in the development of nuclear energy and waste technologies. The (n,α) nuclear reaction inevitably introduces numerous He in SiC. Because of the low solubility of He atoms in SiC, a certain concentration of He atoms that are trapped in the matrix in the form of helium-vacancy clusters would form bubbles upon annealing. He bubbles would perhaps lead to degradation of material properties. Especially, He bubbles along Grain boundaries (GBs) can cause embrittlement by intergranular fracture, as usually observed in metals. Therefore, it is important to investigate the nucleation and growth of He bubbles along GBs in He-implanted SiC.

Samples used in the present study are hot-pressed polycrystalline SiC supplied by the saint-gobain company, and 6H-SiC oriented < 0001 > surface supplied by the MIT company. The sample was exampled via X-ray diffraction (XRD). XRD measurements were carried out with a Bruker D8 Discovery instrument at room temperature (RT). A θ-2θ scanning mode was used with the step size of the scan angle of 0.02 °. The scan range was from 20 ° to 80 °. The result was shown in Fig. 1. This result demonstrates that the sample contains approximately 90% 6H-SiC and 10% 4H-SiC. Polycrystalline SiC wafers were implanted with 230 keV He ions to fluences of 5×10^{15}, 1×10^{16}, 2.5×10^{16}, 5×10^{16} and 1×10^{17} cm^{-2} and a 6H-SiC wafer was implanted with 300 keV He ions to a fluence of 1×10^{16} cm^{-2}. He-implantation was performed at room temperature (RT) and the current density was approximately 0.8 μA/cm^2. He-beam was scanned horizontally and vertically for lateral homogeneity. The peak values of displacement per atom (dpa) and He concentration are approximately 0.14 dpa and 0.35 at. %, respectively, for the implanted fluence of $5\times10^{15}cm^{-2}$. They were estimated via the Monte-Carlo code Stopping and Range of Ions in Matter (SRIM-2008) using t-displacement hreshold energies of 20 and 35 eV for C and Si sublattices, respectively. The projected range, R_p, is approximately 810 nm. The implantation was performed at the 320 kV Multi-disciplinary Research Platform for Highly Charged Ions of the Institute of Modern Physics, Chinese Academy of Sciences (CAS). After-implantation, samples were isochronally annealed in a tube furnace.

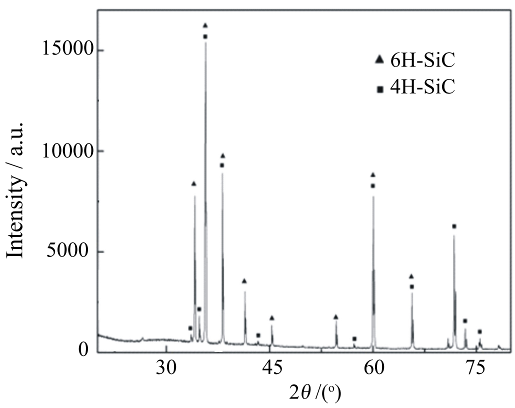

Fig. 1 XRD pattern obtained from the unimplanted polycrystalline SiC sample.

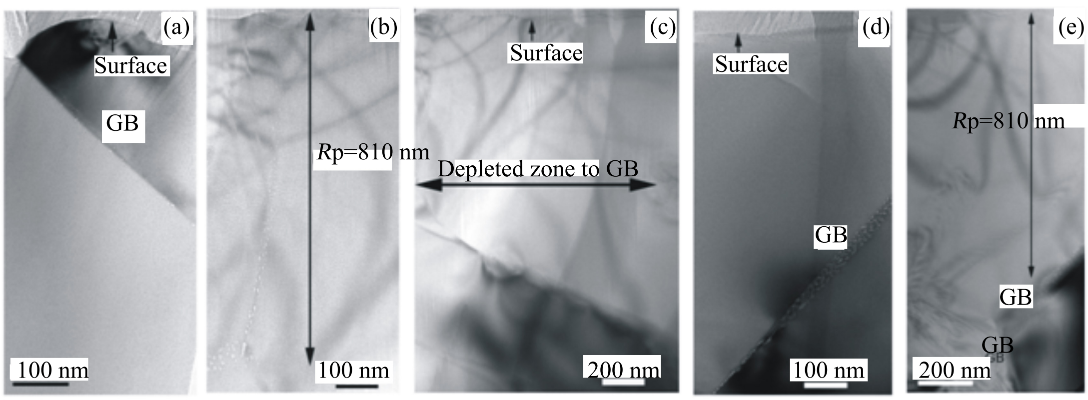

Fig. 2 Under-focused XTEM bright field micrographs of bubble formation in different locations of GBs of the He-implanted polycrystalline-SiC sample to a fluence of 5×10^{15} cm^{-2} followed by annealing at 1 000 °C for 30 min.

Fig. 2 shows under-focused XTEM bright field micrographs of the He-implanted polycrystalline SiC sample to a fluence of 5×10^{15} cm^{-2} followed by annealing at 1 000 °C for 30 min. There are three different cases for the sample implanted to a fluence of 5×10^{15} cm^{-2}. The first case is the GB is located in the near-surface region. A low number density of bubbles was found as shown in Fig. 2(a). The second case is the GB passes through the maximum damage region. He bubbles with triangular shape, oriented in the identical direction along the GBs as shown in Fig. 2(b). The sizes and number density of the bubbles increased when the GB is close to the maximum damage region. Some bubbles were found at the GB which is located in the near sample surface. Bubbles at GBs and in GIs were well separated by a depleted zone, as shown in Fig. 2(c). The width of the depleted zone was approximately 1 μm. The result is consistent with the report of Vincent et al.[1]. Numerous He atoms accumulated into GBs. He bubbles can be formed as the He concentration is greater than the threshold concentration for the formation of He bubbles. Fig. 2(b) shows numerous bubbles with triangular shape at the GB. The decrease of He concentration resulted in suppression of He bubble nucleation near the GB. After annealing at 1 000 °C, He-implantation-induced defects could be easily recovered because of no bubbles observed near the GB, resulting in a depleted zone observed. It should be noted when the grain size is smaller than 1 μm, He bubbles were only nucleated and grown at GBs rather than in GIs, as shown in Fig. 2(d). The third case is the GB is located beyond the projected range. No He bubbles were found, as shown in Fig. 2(e).

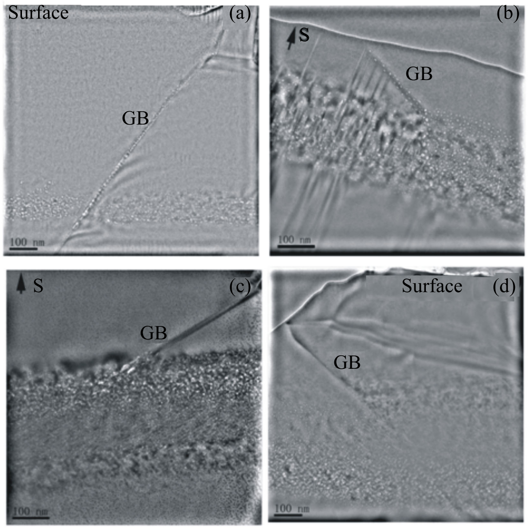

Fig. 3 Under-focused XTEM bright field micrographs of the He bubble formation at GBs of the He-implanted polycrystalline SiC samples with fluences of (a) 1×10^{16}, (b) 2.5×10^{16}, (c) 5×10^{16} and (d) 1×10^{17} cm^{-2} followed by annealing at 1 000 °C for 30 min.

Fig. 3 shows under-focused XTEM bright field micrographs of the He-implanted polycrystalline SiC samples with fluences of 1×10^{16}, 2.5×10^{16}, 5×10^{16} and 1×10^{17} cm^{-2} followed by annealing at 1 000 °C for 30 min. The considerably different microstructural morphology is that no depleted zones were found as compared to that observed in the sample implanted with a fluence of 5×10^{15} cm^{-2}. Growth in size and decrease of number density of the bubbles in GBs for the samples implanted with fluences of 1×10^{16}, 2.5×10^{16} and 5×10^{16} cm^{-2} were found, as shown in Figs. 3(a), (b) and (c). There is no considerable difference of the size and density of the bubbles at GBs and in GIs for the sample implanted with a fluence of 1×10^{17} cm^{-2}, as shown in Fig. 3(d). These results suggest that the ability of trapping interstitial He atoms at GBs is related to the He concentration. It is consistent with the report of Chen et al.[2]. The ability of trapping interstitial He atoms at GBs is inversely proportional to the He concentration.

References

[1] L. Vincent, T. Sauvage, G. Carlot, et al., Vacuum, 83(2009)S36.

[2] J. Chen, P. Jung, H. Trinkaus, Phys. Rev. B, 61(2000)12923.