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Property of P-Induced Graphite Cluster in SiC During Irradiation

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Abstract. Raman scattering spectroscopy was used to study the effect of a phosphorus (P) impurity on the aggregation of displaced carbon (C) atoms into graphite clusters. The P-doped silicon carbide (SiC) samples at concentration of $1.0 \times 10^{20} \text{ cm}^{-3}$ were produced by ion implantation. The P-doped and P-free SiC single crystals were irradiated with two fluences of 1×10^{16} and $2 \times 10^{16} \text{ cm}^{-2}$ at room temperature. The Raman peaks at 1385 and 1562 cm^{-1} are observed for P-containing SiC. They originate from the breathing vibration of six-membered C rings and stretching vibration of C (sp^2)-C (sp^2) bonds. This reveal that the irradiation resulted in the formation of graphite clusters. The irradiated samples were isochronally annealed up to 500°C to investigate the thermal stability. The thermal stability of graphite cluster is approximately $300\text{-}400^\circ\text{C}$.

1. Introduction

SiC has attracted much attention as a promising nuclear material for use in fusion and fission reactors [1]. During the service process, SiC-based materials are subjected to the neutron irradiation which can produce transmutation product. For example, Si atom can be transmuted into P atom. It has been reported that the P transmutation rates in SiC reach 8.2 and 6.1 appm/dpa for the MPBR and HFIR [2]. The incorporation of P atoms into SiC lead to modifications in physical, chemical, and mechanical properties. This might deteriorate the radiation resistance of SiC materials. Therefore, it is necessary that some effort is made to investigate the effects of the P transmutation product on irradiation behavior. In the present work, we designed an ion irradiation experiment to study the contribution of P atoms and reveal the characteristic of irradiation-produced graphite clusters.

2. Experimental

The (0001)-oriented semi-insulating SiC single crystals were purchased from the TankeBlue Semiconductor Co., Ltd, China. Si ion irradiation was carried out at the 320 kV Multi-discipline Research Platform for Highly Charged Ions of the Institute of Modern Physics, Chinese Academy of Sciences. P ion implantation was carried out at room temperature. The P ion energies were 60, 120, 200, and 300 keV, and the corresponding fluences were 3.72×10^{14} , 6.2×10^{14} , 7.44×10^{14} , and $1.3 \times 10^{15} \text{ cm}^{-2}$, respectively. The P concentration was approximately $1.0 \times 10^{20} \text{ cm}^{-3}$. These samples were annealed at 1300°C for 20 min to remove the implantation damage and then irradiated with 1.25 MeV Si^{5+} at fluences of 1×10^{16} and $2 \times 10^{16} \text{ cm}^{-2}$ at room temperature. The damage levels are calculated by



SRIM 2013 software. The threshold displacement energies of C and Si atoms were used. Fig. 1 show the depth profile of damage levels. The post-irradiated samples were isochronally annealed in air at the temperature range from 200 to 500°C for 20 min. A Renishaw inVia Raman microscope was used with 532 nm laser excitation to record Raman spectra at room temperature.

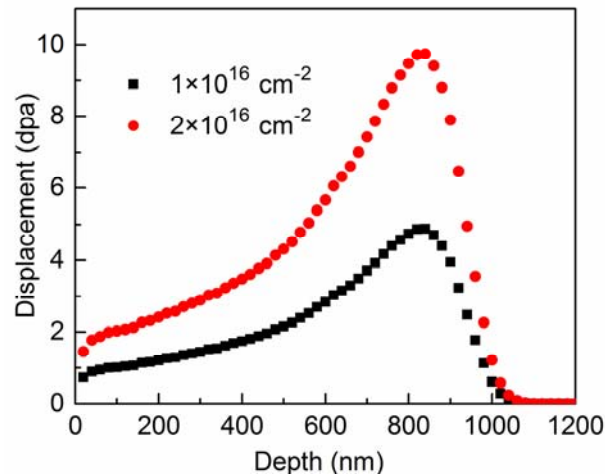


Fig. 1 Depth profiles of damage levels of SiC irradiated at fluences of 1×10^{16} and $2 \times 10^{16} \text{ cm}^{-2}$.

3. Results and discussion

Fig. 2 shows the Raman spectra of pristine and as-irradiated SiC samples. The Fig. 2(a) present the Raman spectrum of pristine SiC. The pristine SiC gives a number of intrinsic Raman peaks. The peaks at approximately 149 and 505 belong to acoustic phonon peaks, the peaks at approximately 767, 788, and 966 cm^{-1} correspond to optical phonon peaks, and the peaks at approximately 1516, 1620, and 1713 cm^{-1} arise from second-order Raman scattering [3]. The Raman peaks, which originate from the vibrations related to C-C bonds, are usually located in the range of 1000-1800 cm^{-1} . Ion bombardment leads to chemical disorder and topological disorder.

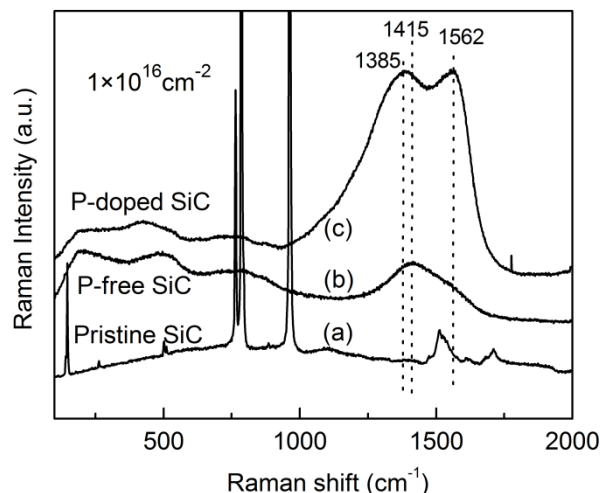


Fig. 2 Raman spectra of pristine and as-irradiated SiC. (a) is the pristine SiC. (b) and (c) are P-free and P-doped SiC irradiated at a fluence of $1 \times 10^{16} \text{ cm}^{-2}$.

The Raman spectra for as-irradiated SiC samples are very different from those for the pristine SiC sample. The Figs. 2(b) and 2(c) are the p-free and p-doped SiC samples irradiated at a fluence of $1 \times 10^{16} \text{ cm}^{-2}$. For the as-irradiated P-free SiC, a well-defined peak at 1415 cm^{-1} and a weak peak 1562

cm^{-1} are recorded. The 1415 cm^{-1} peak originates the mixed sp^2/sp^3 C clusters, and the 1562 cm^{-1} corresponds to the vibration of C (sp^2)-C (sp^2) bonds. However, for the as-irradiated P-doped SiC samples, two peaks at approximately 1385 and 1562 cm^{-1} are simultaneously present in addition to the 1415 cm^{-1} peak. They originate from the breathing vibration of six-membered C rings and stretching vibration of C (sp^2)-C (sp^2) bonds [4, 5]. This result signifies that the ion irradiation led to the formation of graphite clusters. The comparison demonstrates that the P impurity can effectively assist graphite clusters during irradiation.

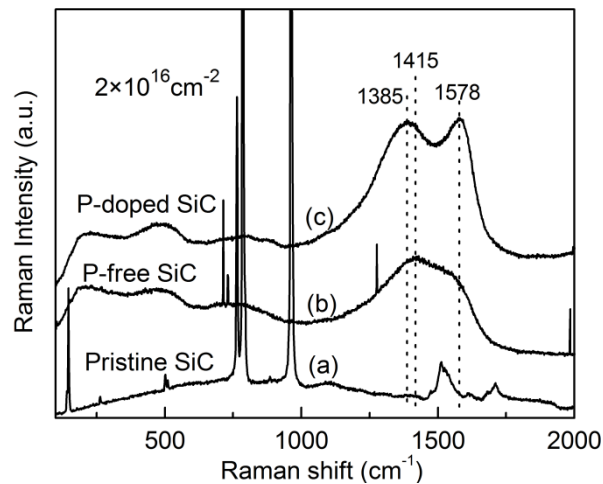


Fig. 3 Raman spectra of pristine and as-irradiated SiC. (a) Is the pristine SiC sample. (b) And (c) are P-free and P-doped SiC samples irradiated at a fluence of $2 \times 10^{16}\text{ cm}^{-2}$.

Fig. 3 show the Raman spectra pristine and as-irradiated SiC samples. The Fig. 2(a) present the Raman spectrum of pristine SiC. The Figs. 2(b) and 2(c) are the p-free and p-doped SiC samples irradiated at a fluence of $2 \times 10^{16}\text{ cm}^{-2}$. When the irradiation fluence increased to $2 \times 10^{16}\text{ cm}^{-2}$, the damage level also increase. These samples contains larger irradiation damage. For the P-free SiC samples, the two 1415 and 1562 cm^{-1} Raman peaks were recorded. Compared to the fluence of $1 \times 10^{16}\text{ cm}^{-2}$, the 1562 cm^{-1} became more distinct. For the P-doped SiC samples, the 1385 and 1562 cm^{-1} peaks are also recorded simultaneously although the peak intensities slightly decrease. This irradiation still led to the formation of graphite clusters.

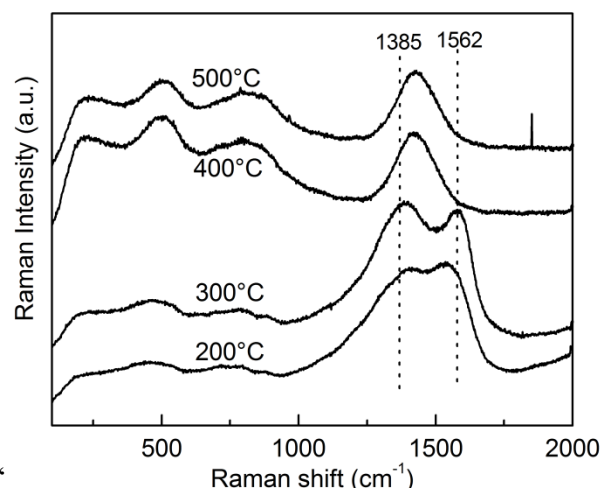


Fig. 4 Raman spectra of post-irradiation annealed SiC at the temperature range from 200 to $500\text{ }^{\circ}\text{C}$.

To analyze the thermal stability, the P-doped SiC samples irradiated at a fluence of $1 \times 10^{16} \text{ cm}^{-2}$ were isochronally annealed in the temperature range from 200 to 500 °C. Fig. 4 shows the Raman spectra of samples annealed at different temperatures. After the 200 and 300 °C annealing, the 1385 and 1562 cm^{-1} almost remain unchanged. When the annealing temperature increases up to 400 °C, the two Raman peaks completely disappear, and only the 1415 cm^{-1} peak is detected. This reveal that the annealing at 400 °C can completely remove the graphite clusters. Thermal stability of graphite cluster is very poor compared to other types of defects.

4. Conclusion

The P-doped and P-free SiC single crystals were irradiated with 1.25 MeV Si^{5+} ions at room temperature. The Raman peaks at 1385 and 1562 cm^{-1} are observed for P-containing SiC. They originate from the breathing vibration of six-membered carbon (C) rings and stretching vibration of C (sp^2)-C (sp^2) bonds. This reveal that the irradiation resulted in the formation of graphite clusters. The thermal stability of graphite cluster is very poor. Annealing at 400 °C can completely remove the graphite clusters.

Acknowledgements

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