Summary of the full doctor dissertation

Microscopy study of correlation between residual elastic strain and defects in He^+ ion irradiated 4H-SiC

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Chapter 1 Introduction

Silicon carbide (SiC) has drawn great attention for electronic and optical applications owing to its excellent mechanical, optical and electronic properties, which has also been proposed as structure and cladding materials in nuclear or aerospace environment. However, for its extensive applications, there are still lots of problems to overcome, especially for the irradiation-induced strain in SiC.

In this chapter, the excellent properties of SiC, including superior electronic properties, high thermal conductivity and good mechanical properties, were described, which made SiC a promising material both as semiconductor materials for electronic devices and structure materials in nuclear environment. Besides, the problems SiC would meet during application were also introduced, and the need for the investigation of irradiation-induced strain or swelling in SiC is reviewed. For SiC-based electronic devices fabrication, ion implantation is widely used for selected-area doping into SiC, which would inevitably induce great strain or swelling. This strain has been found to significantly degrade the mechanical properties and change the electronic properties of the SiC devices. Therefore, an accurate non-destructive evaluation of the residual strain in SiC devices has become increasingly important, in particular with the development of the advanced micro/nano SiC-based devices. However, it is a significant challenge for common strain measurement techniques like X-ray diffraction (XRD) due to their spatial resolution limitation. A non-destructive technique with a high spatial and strain resolution is needed.

Besides, the irradiation-induced strain/swelling has been found to be driven by the various defects in SiC. Hence, clarifying the contribution to the strain/swelling of each defect type is important to accurately evaluate the strain or swelling. Moreover, the investigation of irradiation-induced strain/swelling in SiC is also important for its application in nuclear reactor or aerospace, where SiC would also be subjected to various irradiation. In the following chapters, those issues mentioned above were targeted on the basis of a series of experiments.

Chapter 2 Non-destructive evaluation of the strain distribution in selected-area He+ ion irradiated 4H-SiC

To investigate the strain distribution in the whole selected-area ion-implanted SiC, selected-area He^+ ion irradiation was performed on n-type 4H-SiC (0001) single-crystal substrates with an energy of 100 keV at room temperature to a fluence of 5×10^{16} cm^{-2}. The swelling state in this sample was confirmed by atomic force microscopy, and the surface of sample after irradiation was observed by field emission scanning electron microscope. The residual strain is this sample was evaluated by non-destructive techniques of electron backscattering diffraction (EBSD) and confocal Raman microscopy (CRM).

A detailed normal strain distribution in the ion-irradiated 4H-SiC was first provided using EBSD and CRM, whose results showed good agreement. This result validated the application of EBSD and CRM to evaluate the detailed strain distribution in selected-area ion-implanted SiC. Besides, In the selected-area He^+ ion-irradiated 4H-SiC, strain is shown to not only be introduced into the irradiated area, but also to extend into the unirradiated substrate owing to irradiation-induced swelling. Furthermore, a higher compressive strain is concentrated around the interface between the irradiated and unirradiated areas. An anisotropic strain distribution in selected-area He+ ion irradiated 4H-SiC were detected by EBSD. The relative strain degree shows correlation with the crystallographic orientation, and the strain distribution around the interface may be determined by the correlation between the strain and interface direction. Variation of the A1(LO) mode peak was observed by CRM in the strain-introduced substrate, which might be attributed to the effect of strain and defects on the carrier density in this region. Comparison of the results EBSD and CRM indicates that EBSD is more convenient to explore the correlation between the strain and the orientation, but CRM spectra can provide more information about variations in microstructure and electronic properties.

Chapter 3 anisotropic defects distribution in the He+ ion irradiated 4H-SiC with anisotropic swelling

In chapter 2, the anisotropic strain was found in selected-area ion irradiated 4H-SiC, the anisotropy strain would induce crack in the material. However, the mechanism of this anisotropic strain is not clear. In this chapter, the mechanism was investigated. For a comparison, samples with different irradiation conditions were made at room temperature, including selected-area He^+ ion irradiated 4H-SiC with fluence of 1×10^{15} and 5×10^{16} cm^{-2}, non-selected-area He^+ ion irradiated 4H-SiC with fluence of 5×10^{16} cm^{-2} and electron irradiated 4H-SiC TEM foils. TEM samples were made using a focused ion-beam system. The defect distribution in these irradiated 4H-SiC was explored using various transmission electron microscopy (TEM) techniques. The stress in the selected-area He^+ ion irradiated samples was also measured using EBSD. In addition, the defect distribution after annealing was also explored to investigate the stress effect on defect distribution.

An anisotropy of defects distribution was found that interstitials type defects preferentially redistribute into the free expanding direction ([0004] direction) with more negative volume defects locating in the constraint swelling direction ($[10\bar{1}0]$ and $[11\bar{2}0]$ direction), which might account for the anisotropic strain or swelling. This anisotropy decreased upon annealing at 873 K. The anisotropy of defect distribution was also observed in the non-selected-area He^+ ion irradiated 4H-SiC and electron irradiated 4H-SiC, but it is quite lower than that in the selected-area ion irradiated samples. It was found that great compressive stress was introduced in the lateral direction ($[10\bar{1}0]$ and $[11\bar{2}0]$) in the selected-area He+ ion irradiated 4H-SiC due to the constraint against lateral expansion, and these compressive stresses were introduced at the beginning of ion irradiation. The compressive stress introduced during irradiation is supposed to inhibit the interstitials types defects formation, enhancing the anisotropic defect distribution.

Chapter 4 Electron energy-loss spectroscopic evaluation of depth-dependent swelling of He^+ ion-irradiated 4H-SiC correlated with defect type

The irradiation-induced strain or swelling is driven by irradiation-induced various defects, but the knowledge of the role of specific defect on selling is still limited. In this chapter, various defects, including point defects or tiny clusters, defects clusters, amorphous transition and helium bubbles, were introduced into 4H-SiC by He+ ion irradiation with different fluence (1×10^{15} to 1×10^{17} cm^{-2}), and these defects were identified by various TEM techniques. The local swelling induced by in different defect region was measured by scanning transmission electron microscopy electron energy-loss spectroscopy (STEM-EELS).

The correlation of the swelling with various defects, such as point defects or tiny clusters, defects clusters, amorphous transition and helium atoms, in the He+ ion irradiated 4H-SiC at room temperature was separately analyzed. These results provided the volume swelling range for the different defect regions, which is helpful for the prediction of SiC swelling in actual application. A saturation of the volume swelling in the near-surface region, where only point defects and tiny clusters were introduced, was observed over the fluence of 1×10^{15} to 1×10^{17} cm^{-2} at room temperature. This saturation of swelling has already reached with a value of about 2-3% at great low dose of about 0.02 dpa (displacement per atom). The swelling of region containing defect clusters ranges from about 3%–7% at room temperature. The amorphization transition could result in a swelling of SiC as high as about 13%. Helium atoms in the form of helium bubbles increase the volume swelling at relatively high irradiation fluences. However, decreasing effect of He^+ ion irradiation on volume swelling also seems to be possible as below a certain irradiation fluence.

Chapter 5　Conclusions and Future Work

In this chapter, the main conclusions and most relevant findings of this work are presented and summarized.