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Scanning electron microscopy of the surfaces of ion implanted SiC

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ABSTRACT

This paper gives a brief review of radiation damage caused by particle (ions and neutrons) bombardment in SiC at different temperatures, and its annealing, with an expanded discussion on the effects occurring on the surface. The surface effects were observed using SEM (scanning electron microscopy) with an in-lens detector and EBSD (electron backscatter diffraction). Two substrates were used, viz. single crystalline 6H-SiC wafers and polycrystalline SiC, where the majority of the crystallites were 3C-SiC. The surface modification of the SiC samples by 360 keV ion bombardment was studied at temperatures below (i.e. room temperature), just at (i.e. 350 °C), or above (i.e. 600 °C) the critical temperature for amorphization of SiC. For bombardment at a temperature at about the critical temperature an extra step, viz. post-bombardment annealing, was needed to ascertain the microstructure of bombarded layer. Another aspect investigated was the effect of annealing of samples with an ion bombardment-induced amorphous layer on a 6H-SiC substrate. SEM could detect that this layer started to crystalize at 900 °C. The resulting topography exhibited a dependence on the ion species. EBSD showed that the crystallites forming in the amorphized layer were 3C-SiC and not 6H-SiC as the substrate. The investigations also pointed out the behaviour of the epitaxial regrowth of the amorphous layer from the 6H-SiC interface.

1. Introduction

Many of present applications and future applications of SiC is based on two of its key properties – it is one of the hardest natural materials, with a hardness of around 9.2–9.3 Mohs [1], and it has the ability to retain most of its properties at high temperatures – it decomposes significantly in vacuum only at about 1700 °C [2]. There are two applications of SiC, where particle bombardment is a fundamental step in their operation. SiC is a wide band-gap semiconductor and can, therefore, be used in high temperature electronic devices. Doping of the SiC is usually done by ion bombardment and annealing. SiC is also used as a coating layer covering the fuel elements of the next generation of nuclear power plants because SiC is diffusion barrier for radioactive fission products [3,4]. This diffusion barrier property will prevent the escape of radioactive fission products into the environment during an accident in such a nuclear reactor.

For both these two applications the radiation damage properties of SiC need to be investigated. Because there is a large difference between the covalent radii of the silicon and carbon atoms, SiC has, for the common semiconductor materials, a fairly large iconicity value of 0.475 on the Garcia-Cohen scale (Phillips 0.177 and Pauling 0.11) with charge transfer to the carbon atom [5]. Because its iconicity is larger than most of the common semiconductors, it is more radiation resistant than those semiconductors. This results in potential electronic and sensor applications in a radiation environment [6,7]. However, SiC is still mainly a covalent bonded material (88% covalency and 12% ionic). This means that it is hard to anneal radiation damaged SiC, with only very high temperatures (1500 °C and higher) being reported as successful to anneal a completely bombardment-induced amorphous SiC layer on 6H-SiC [3,8,9].

Most of the radiation damage and annealing studies of SiC have employed either transmission electron microscopy (TEM) or Rutherford backscattering/channeling. In this paper we report on ion bombardment-induced radiation damage of 6H-SiC and polycrystalline SiC (predominantly 3C-SiC crystallites) using SEM (scanning electron microscopy). This means that the results mainly pertain to processes occurring on the surface of these materials. We shall report on the effect of ion bombardment at room temperature and at 350 °C and 600 °C on these surfaces, and also annealing of

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6H-SiC with a top amorphized layer caused by ion bombardment at room temperature.

2. Experimental

6H-SiC (from *Intrinsic Semiconductors*® and from *Pam-Xiamen*) and polycrystalline SiC – predominantly 3C-SiC crystallites – (from *Valley Design Corporation*®) samples were investigated by field emission scanning electron microscopy (FEG-SEM) employing a *Zeiss Ultra 55* instrument fitted with the usual SEM detectors and an in-lens detector. In this paper, unless specifically mentioned, mostly images taken at 2 kV in the in-lens mode are shown.

Various ions, all with an energy of 360 keV, were implanted into the SiC samples at an incidence angle of 7° to fluences of either 1×10^{16} or 2×10^{16} cm^{-2}. A dose rate of about 5×10^{12} cm^{-2} s^{-1} was used. Notwithstanding this relatively low rate, temperature of the samples implanted at room temperature increased to about 50 °C during the bombardment process, which will be denoted as bombarded at room temperature. To investigate the effect of substrate temperature during ion bombardment, some samples were implanted at 350 °C and some at 600 °C. Some samples were vacuum annealed in a computer controlled *Webb* graphite furnace for different periods at temperatures ranging from 800 up to 1600 °C. The base pressure prior to annealing was in the range 10^{-6}–10^{-7} mbar. During annealing, the pressure sharply increased to a maximum of 5×10^{-5} mbar and then decreased to the low to middle 10^{-6} mbar range.

3. Results and discussion

As was mentioned in the previous Section 2in this paper only SEM images taken in the in-lens mode, are shown. The in-lens detector gives SEM images showing defects present in the samples but at the expense of topographic detail, which is the *forte* of the conventional SEM detector. Crystallographic detail (an important aspect in this paper) also becomes more distinguishable in the in-lens mode. In Fig. 1 a normal secondary electron SEM detector image (Fig. 1(a)) and also an in-lens SEM image (Fig. 1(b)) of the same area of a polycrystalline SiC sample are shown. In the normal SEM image the topography of the sample is clear to see. In contrast, the topography is much less pronounced in the in-lens image but many lines appear in the crystallites. These defect lines are due to twins and stacking faults, which are extremely common in SiC due to its plethora of polytypes [10]. The reason for the polytype formation in SiC (and consequent line and plane defects) lies in the very small differences in the total energy of formation between the common polytypes, viz. of the order of O(1) meV/atom, or even less [11].

Radiation damage caused by particle bombardment in SiC has recently been reviewed [3]. It has been reported that the radiation damage occurring in SiC at fluences below the critical fluence for amorphization is similar between ions and neutrons at the same dpa (displacements per atom) value [12,13]. At low temperatures and low irradiation fluences the main defects are point defects (called black spot defects due to their appearances in weak beam dark field TEM images) and small interstitial clusters in various configurations. Increasing the temperature and/or fluence result in the “black spot” defects to pass into dislocations and dislocation loops. At higher temperatures and/or fluences Frank faulted loops of the interstitial type appear with 1/3<111> Burgers vectors. Voids start to appear in the SiC at temperatures above 1000 °C and

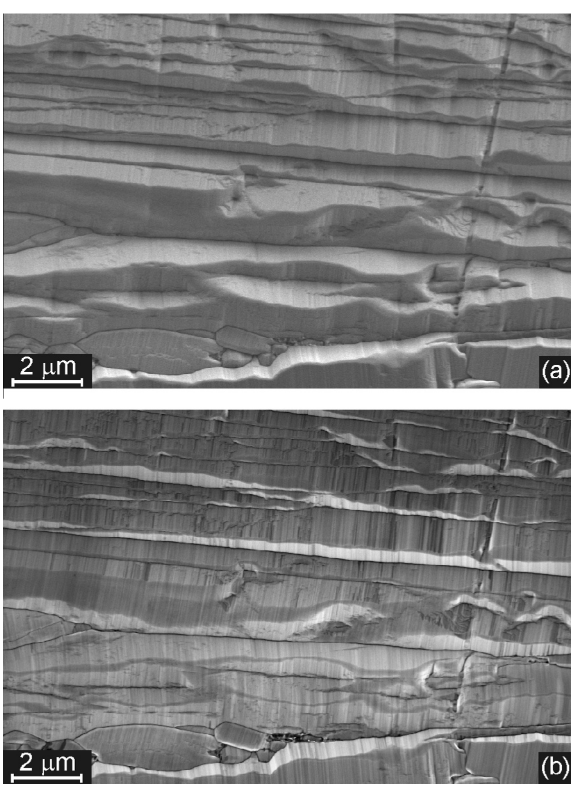

Fig. 1. (a) A normal SEM image showing the facets of the different crystallites and (b) an in-lens image showing stacking faults and microstwins in the crystallites, of the same area of a polycrystalline SiC sample.

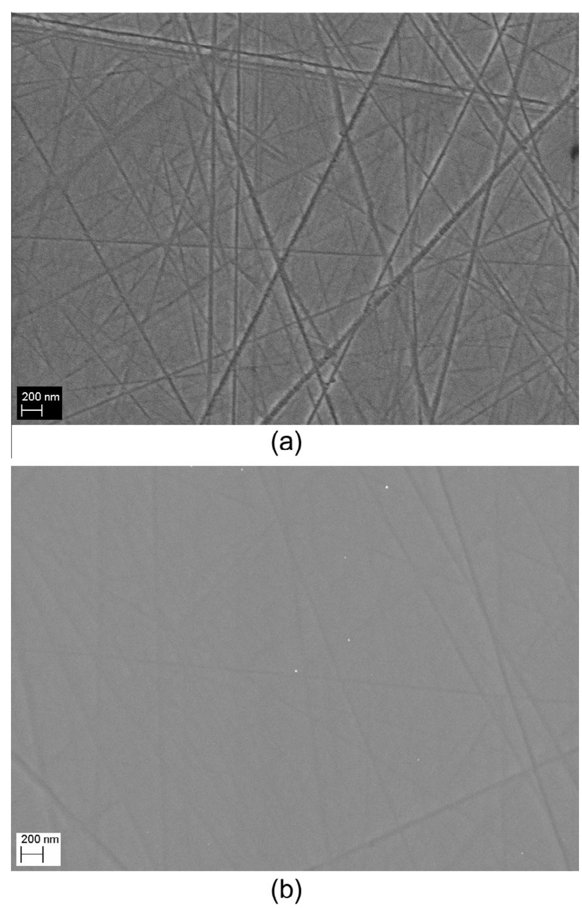

Fig. 2. SEM images of (a) as-received 6H-SiC, and (b) 6H-SiC after bombardment at room temperature by 360 keV Cs^+ ions to a fluence of 2×10^{16} Cs^+ cm^{-2}.