Synchrotron X-ray Microdiffraction Analysis of Proton-irradiated Polycrystalline Diamond Films

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Introduction

Diamonds possess many desirable properties, including extreme hardness, high wear resistance, chemical inertness, optical transparency, and a wide band-gap. Polycrystalline diamonds produced by techniques such as chemical vapor deposition (CVD) continue to show great promise for insertion into advanced technologies, such as advanced electronics, microsystems, and microelectromechanical systems.

The fact that diamonds are extremely resistant to radiation damage is a very important attribute, especially for space-related applications where high radiation levels are expected. The effects of radiation on the properties of diamonds have been studied for decades. However, the majority of these investigations examined electronic or microstructural changes in monocrystalline diamonds, usually by using heavy ions [1, 2]. Fewer studies examined the effects of light ions, such as protons, on the microstructural changes of irradiated CVD diamonds, particularly as a function of ion implantation depth [3]. We have used the 3-D polychromatic x-ray microscope at beamline 34-ID at the APS to make preliminary measurements of the depth-resolved strain in protonirradiated CVD diamond films. Differential aperture x-ray microscopy [4] was used to resolve the strain tensor as a function of depth below the sample surface.

The elastic strain tensor elements are extracted as a function of depth along the penetration direction of the microbeam [4].

Figure 1 presents a schematic of the experimental setup needed to perform micro x-ray diffraction (μ -XRD) analysis. A broad-bandpass microbeam intercepts the sample and illuminates a small number of crystal grains. The overlapping Laue patterns from the grains are recorded on an area detector. If a wire is moved near the surface of the sample, the difference between the pattern before and after a submicron movement can be used to determine the origin of the various Laue reflections. After of series of differential moves, the Laue patterns from along the incident beam can be reconstructed. Each pattern is then analyzed to determine the reflection indices. The texture and orientation and strain tensors are then determined from the location and angular differences of the various reflections. Strain measurements of less



FIG. 1. Schematic sample and instrumental geometry for μ -XRD experiment.

than 10^{-4} are possible by using this technique. For a detailed example of this procedure, see Ref. 4.

Methods Materials

A polycrystalline diamond that was approximately 20 μ m thick was prepared on a 2-in. single-crystal silicon substrate by using microwave plasma-assisted CVD (MPACVD) at a temperature of 800°C and pressure of 110 torr. The samples were implanted with 600-keV protons at a dose of 2×10^{17} H⁺/cm² at 1.2 to 6×10^{-7} torr. According to implant modeling software, 600-keV protons would be implanted to a depth of approximately 6 μ m.

The sample was not annealed after the irradiation exposure. Polychromatic microdiffraction measurements were performed at beamline station 34-ID-E at the APS to determine the stress tensor as a function of depth. Both implanted and as-deposited films were studied.

Results

The strain tensor was measured as a function of depth at six sites for each sample. Both normal strain and shear strain were investigated. Normal strain for the asdeposited film was relatively small, with fluctuations of $<5 \times 10^{-4}$. The normal stresses for the implanted films are much larger and peak at about 5 µm into the film (Fig. 2). The shear component behavior is similar. The shear



FIG. 2. Normal components as a function of depth for the deviatoric strain tensor for the $2 \times 10^{17} \text{ H}^+/\text{cm}^2$ implanted diamond specimen.

components for the as-deposited film are relatively small at approximately $\pm 1 \times 10^{-3}$. The shear components for the implanted film (Fig. 3) fluctuate wildly within the first $5 \,\mu m (\sim 6 \times 10^{-3})$ and then are more similar to those of the as-deposited film. The large shear components are assumed to arise from elastic mismatch between the misoriented grains in the film.

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FIG. 3. Shear components as a function of depth for the deviatoric strain tensor for the $2 \times 10^{17} \text{ H}^+/\text{cm}^2$ implanted diamond specimen.

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