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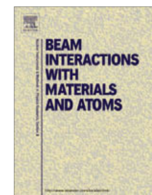

Temperature behavior of damage in sapphire implanted with light ions

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ABSTRACT

In this study, we compare and discuss the defect behavior of sapphire single crystals implanted with different fluences (1×10^{16}–1×10^{17} cm^{-2}) of carbon and nitrogen with 150 keV. The implantation temperatures were RT, 500 °C and 1000 °C to study the influence of temperature on the defect structures. For all the ions the Rutherford backscattering-channeling (RBS-C) results indicate a surface region with low residual disorder in the Al-sublattice. Near the end of range the channeled spectrum almost reaches the random indicating a high damage level for fluences of 1×10^{17} cm^{-2}. The transmission electron microscopy (TEM) photographs show a layered contrast feature for the C implanted sample where a buried amorphous region is present. For the N implanted sample the Electron Energy Loss Spectroscopy (EELS) elemental mapping give evidence for the presence of a buried damage layer decorated with bubbles. Samples implanted at high temperatures (500 °C and 1000 °C) show a strong contrast fluctuation indicating a defective crystalline structure of sapphire.

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1. Introduction

Ion beams are a reliable way to modify and create new structures far from equilibrium offering the possibility to tailor material properties. The ballistic nature of ion implantation is responsible for the creation of different microstructures associated with defects produced in the implanted region. In most cases the understanding of the physics underlying defect production and stability is still far from accomplished in a large variety of materials. In sapphire implantation damage processes are fairly well understood for implantations at room temperature (RT) with medium or heavy mass ions (mostly inert gases and transition metals) [1–4].

Sapphire is one of the most stoichiometric oxides and shows a limited solubility of impurities. The nonequilibrium introduction of implanted species by ion implantation requires the formation of second phases and/or defect-impurity complexes that compensate for unbalanced charges [5]. Implantation of inert gases at high fluences is known to cause blisters and exfoliation of the surface [6]. In a previous work, we found a dechanneling peak in the Al-sublattice at the depth of the Al recoils and suggested that it might be due to Al-interstitials [7]. According to SRIM simulations there are few recoils beyond the range of the light ions and both the displaced Al and O as well as the implanted ions exhibit a range distribution with negative skewness [8]. The calculations suggest that there should be little damage beyond the range of the implanted ions which are likely to be associated with the vacancies created by the knock-ons. Furthermore, recent studies reveal the presence of diamond like precipitates in 1 MeV C implanted sapphire [9]. However, no information was obtained about the stability of the new mixed structures formed in the implanted layer.

In this work, we report the implantation of sapphire with C and N at RT, 500 °C and 1000 °C with different fluences. The as implanted and annealed samples were studied by RBS-C, TEM, EELS elemental mapping and optical absorption in order to get a detailed picture of the evolution of the microstructures formed during the implantation.

2. Experimental procedures

High purity single crystals of α-Al_2O_3 (Crystal Systems, Inc., Bedford, MA) with optically polished surfaces parallel to {0001} planes were implanted with C and N ions to fluences in the range of 1×10^{16}–1×10^{17} ion/cm^2 (150 keV). The samples were kept at RT, 500 °C and 1000 °C during the implantation. The beam was tilted 7° from the *c*-axis to prevent channeling and the current density was kept below 1–2 μA/cm^2 to reduce beam heating effects. All the samples were annealed at 1000 °C for 1 h in vacuum ($\sim 1 \times 10^{-4}$ Pa).

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RBS-C measurements were performed with 2.0 MeV He^+ beams and silicon barrier detectors placed at 140° and 180° in the standard IBM geometry. Optical absorption measurements were made with a Varian Cary 5G IR–VIS–UV spectrometer. Cross-sectional TEM specimens were prepared by mechanical polishing and focused ion beam (FIB) thinning. The specimens were examined at 300 kV with a JEOL 3010 HR TEM with 1.7 Å point resolution equipped with a GATAN TRIDIEM post column EELS spectrometer. The TEM samples were prepared by embedding two slabs of the specimen into a 3 mm diameter Ti disk that was thinned and polished mechanically until a residual thickness of 50 µm. It was followed by ion beam thinning with 10 kV Ar ions at extremely low incidence angles (2–4°) and finally, the ion energy was decreased to 3–1 kV for reducing amorphisation [10].

3. Discussion of results

The implantation damage produced by light ions in sapphire evolves from the end of range of the implanted ions towards the surface. As we can see in Fig. 1 for the lowest fluence a high concentration of defects close to the range of the implanted ions is responsible for the dechanneling at a depth of ~270 nm. For higher fluences the damage level increases significantly towards the surface which is visible in the spectrum obtained for the sample implanted with 1×10^{17} cm^{-2}. This asymmetrical behavior follows the SRIM2006 calculations as we could see in Fig. 2. The spectra taken with the beam aligned along the ⟨0001⟩ axis in the samples implanted with 1×10^{17} C^+/cm^2 at 500 °C (not shown) and 1000 °C are very similar and show an increasing dechanneling to almost random values at depths of 200–300 nm. The high dechanneling rate is an indication for the presence of a buried layer with a high density of defects. Although the dechanneling curves are similar for the RT and high temperature implanted samples the defects produced are different according TEM analyses. While the sample implanted at room temperature show a layered damage structure with a thin amorphous layer in the middle, Fig. 3, the sample implanted at a 1000 °C only reveals a rather homogeneously damaged crystalline structure (Fig. 5).

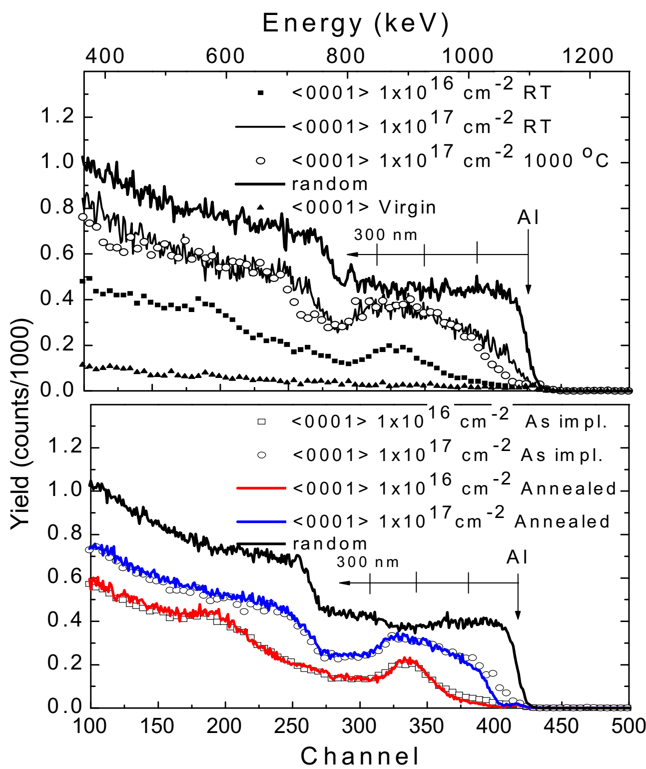

Fig. 1. Random and c-axis aligned RBS spectra obtained with 2.0 MeV He^+ for the sample implanted with 1×10^{16} C^+/cm^2 and 1×10^{17} C^+/cm^2 (150 keV) at RT and 1000 °C (top). A spectrum of a virgin sample is included for reference. At the bottom we compare the as implanted and annealed samples at 1000 °C 1 h under vacuum.

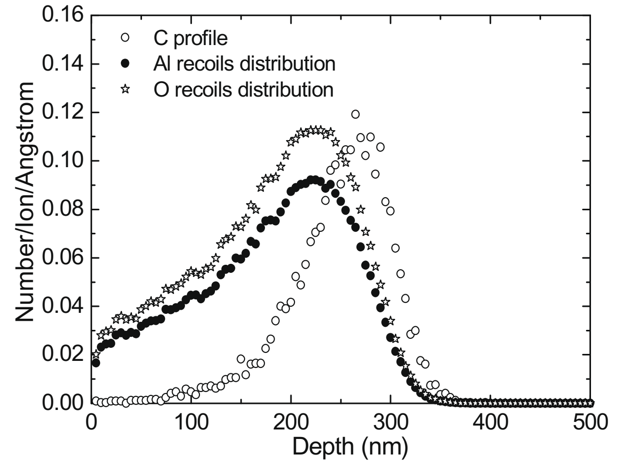

Fig. 2. SRIM2006 calculations showing the asymmetric distribution of the Al and O recoils. The asymmetry of the C profile is less pronounced (skewness = −0.962)

A similar damage evolution was found for N implanted samples. In this case the RT implanted samples show the presence of bubbles and no indication of an amorphous structure in the implanted region was found for fluences up to 1×10^{17} N^+/cm^2. The bubbles are also present in the samples implanted at 1000 °C and are aligned parallel to the surface (along the c-plane) at a depth of ~160 nm, Fig. 4. The Zero Loss Image and EELS elemental maps shown in Fig. 4 indicate the string out of the bubbles (a) a depletion of Al (b) and a high concentration of N (c) that prove the filling of the bubbles by N. Since the projected range calculated by SRIM2006 indicates a value of 247 nm it is evident that the N ions

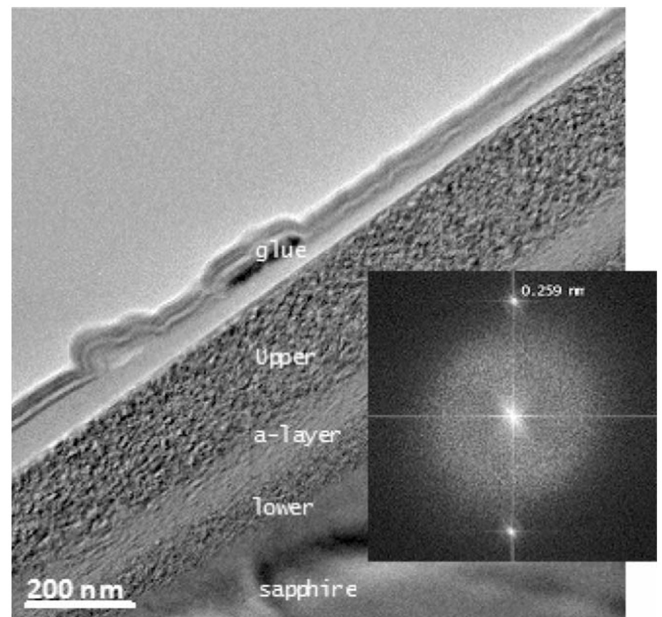

Fig. 3. Cross-sectional TEM micrograph for sample implanted 1×10^{17} C^+/cm^2 at room temperature where a damage layered structure with a buried amorphous layer is observed. The insert shows the diffraction pattern of the amorphous layer.

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