



On the threshold of damage formation in aluminum oxide via electronic excitations



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ABSTRACT

This work is aimed to determine the threshold of dense ionization induced damage formation and their morphology in sapphire single crystals irradiated with 1.2 MeV/amu Xe ions. Cross-sectional TEM examination of r-oriented Al₂O₃ specimens irradiated to fluences of 2×10^{12} and 2×10^{13} cm⁻² has revealed discontinuous ion tracks visible from the irradiated surface up to a depth of 7.6 ± 0.1 μm. According to the SRIM code calculation, the threshold electronic stopping power for track formation in Al₂O₃ is within the range $9.8 \div 10.5$ keV/nm. This value agrees with those predicted by both inelastic and analytical thermal spike models.

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1. Introduction

Radiation damage formation in Al₂O₃ due to electronic energy losses has been observed in many works [1–11]. The first estimate of the corresponding electronic stopping power threshold undertaken by Canut et al. using channelling Rutherford backscattering gives $S_{et} = 21$ keV/nm [2]. As a result of the TEM studies of sapphire irradiated with 10–30 MeV fullerenes, values in the range of $S_{et} \approx 18$ –21 keV/nm values were obtained [3]. It was found that tens of MeV fullerenes create amorphous tracks with diameters of 13 ± 0.4 nm for $S_e = 76.2$ keV/nm, in for example [3,4]. However, no amorphization or crystalline-to-crystalline phase transition in the ion trajectory region is observed under monoatomic particle irradiation (Bi, 710 MeV) at the specific energy loss of 41 keV/nm [7]. Using the strongly diffracting region in the TEM images for estimating the Bi ion track size, the average TEM track diameter was found to be in the range of 3–4 nm.

Szenes [12] analysed the TEM data of Aruga et al. [5] on amorphization of polycrystalline alumina by using 85 MeV iodine ions in the framework of the analytical thermal spike model (ATSM) [13] and deduced $S_{et} = 9.8$ keV/nm. It should be noted that authors of work [5] estimated this value as 4–5 keV/nm taking the electronic stopping power at the amorphous–crystalline phase boundary (around 5 μm) at an ion fluence of 1.2×10^{15} cm⁻². At lower ion fluences of 1.2×10^{14} and 2.8×10^{14} cm⁻², the stripe structures along the ion beam direction were observed up to depths of around

2.5–3 μm. At this depth the iodine ion energy is ~ 0.5 MeV/amu, which suggests $S_{et} \sim 9$ keV/nm. Different thresholds may be explained by the “velocity effect” (see, for example, [14]) and agree with values predicted by calculating the dependence of the minimum electronic energy loss needed to create tracks in sapphire on the ion beam energy as presented in [15]. In this work a systematic analysis of experimental data on lattice disorder in Al₂O₃ via electronic excitations has been undertaken recently using techniques such as channelling Rutherford backscattering, atomic force microscopy and profilometry. Corresponding S_{et} thresholds deduced from these experiments and the inelastic thermal spike model (ITSM) [16], was found to be equal to 9.5 ± 1.5 keV/nm. However, the threshold value is not yet confirmed by direct electron microscopy observations. Moreover, the TEM examinations of swift heavy ion irradiated Al₂O₃ were done for several electronic stopping power levels only, and microstructural information about damage morphology in the ion tracks is very limited. The aim of this report is to determine the threshold of dense ionization induced damage in sapphire single crystals irradiated with 1.2 MeV/amu Xe ions.

2. Experimental

The material used was r-oriented single crystal sapphire wafers, with a thickness of 0.5 mm, purchased from MTI. The samples were irradiated with 167 MeV Xe ions to fluences of 2×10^{12} and 2×10^{13} cm⁻² at the IC-100 FLNR JINR cyclotron. Ion beam homogeneity over the irradiated specimen surface was controlled using

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beam scanning in the horizontal and vertical directions and was better than 5%. The average Xe ion flux was less than $10^9 \text{ cm}^{-2} \text{ s}^{-1}$ to avoid significant heating of the targets mounted on water-cooled copper holders maintained at 25–30 °C.

Cross-sectional TEM (XTEM) specimens have been prepared using an FEI Helios Nanolab 650 FIB. Initial milling was done with 30 keV Ga ions with final thinning at 5 keV ion energy. Polishing was performed at 2 keV and 500 eV producing a near damage free TEM foil. All specimens were analyzed using a JEOL JEM 2100 LaB₆ transmission electron microscope operating at 200 kV. Since the typical depth of FIB prepared lamella is around 5 microns, several FIB lamellae were produced with lengths of about 13 μm, exceeding the calculated 167 MeV Xe ion projected range in sapphire $R_p = 11.1 \mu\text{m}$, from the edge of the irradiated surface of the specimen as shown in the schematic drawing in Fig. 1.

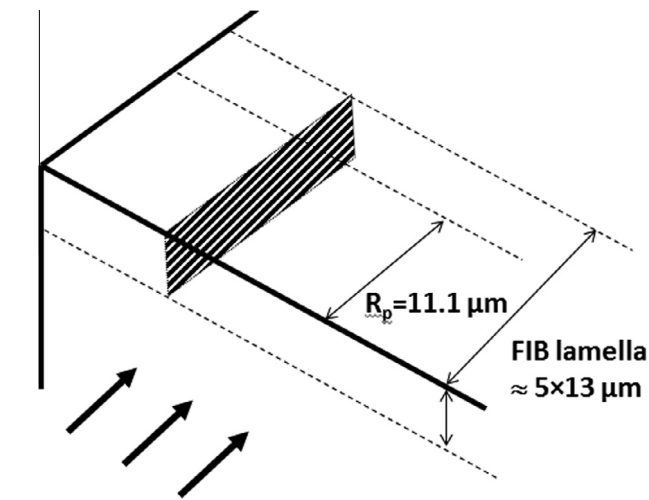


Fig. 1. Schematic drawing of XTEM target preparation. The arrows indicate the Xe ion beam direction. The size of FIB lamella, shown as shaded rectangle, is about 5×13 microns.

3. Results and discussion

Fig. 2 shows a bright field (BF) XTEM micrograph of $2 \times 10^{12} \text{ cm}^{-2}$ 167 MeV Xe ion irradiated Al₂O₃. The selected area diffraction (SAD) inset shows the diffraction condition and a higher magnification inset in the bottom corner shows the ion tracks appearing as discontinuous lines of strain contrast along the ion trajectory. The specimen in this image was not prepared in the geometry shown in Fig. 1 but rather by milling down into the implanted surface, yielding a lamella parallel to the ion trajectory with the top surface of the lamella corresponding to the implanted surface. These tracks extended through the depth of the FIB lamella ($\sim 4 \mu\text{m}$) with no significant change in morphology. The number of ion tracks per 1 cm^{-2} estimated from cross-section images is about 0.1 times that of the Xe ion fluence in the subsurface region and gradually decreases with ion penetration depth.

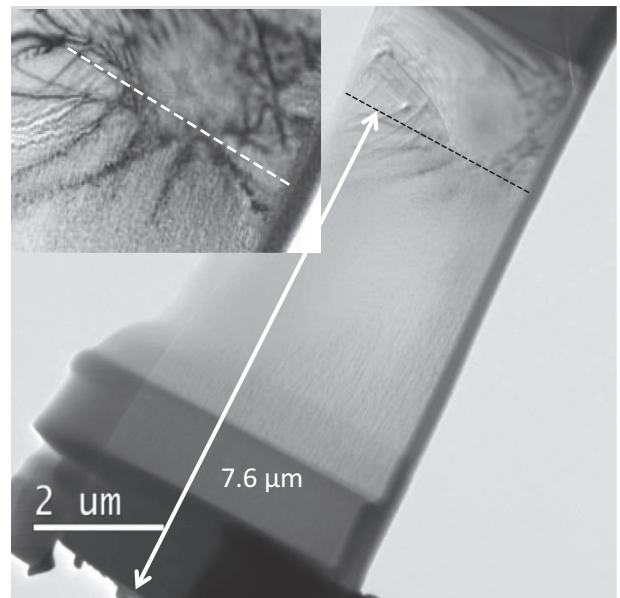


Fig. 3. The micrograph of FIB lamella used for Xe ion track range estimate. Ion fluence is $2 \times 10^{13} \text{ cm}^{-2}$. Ion tracks are registered within a range indicated by arrow.

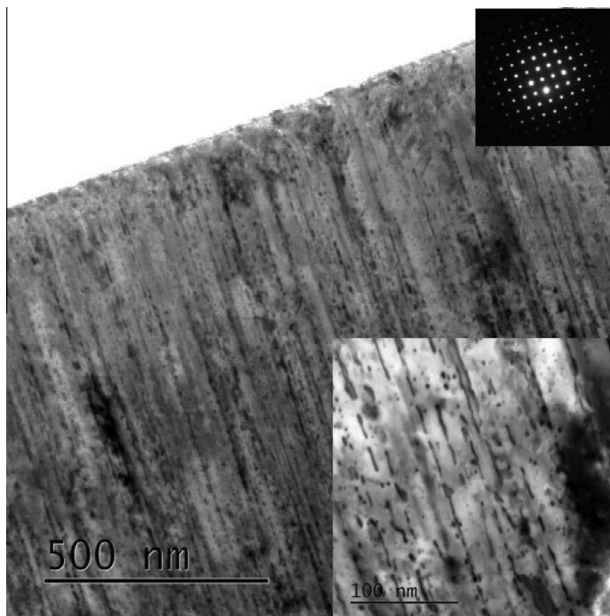


Fig. 2. XTEM image of 167 MeV xenon ion induced tracks in Al₂O₃. Ion fluence is $2 \times 10^{13} \text{ cm}^{-2}$.

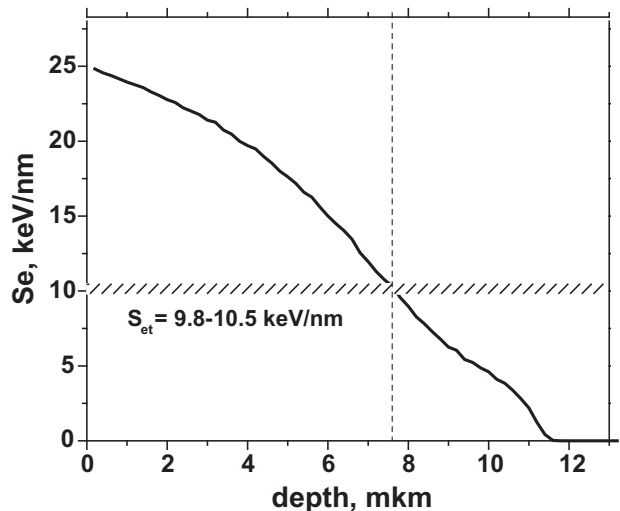


Fig. 4. Electronic stopping power profile of 167 MeV xenon ions in Al₂O₃. Dashed line indicates the maximal depth where ion tracks are visible.

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