**ID-10-P-2697 Nb-doped Sr$_3$Ti$_2$O$_7$ platelets: Preparation of cross-sections thin foils for TEM observations**

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Nb-doped SrTiO$_3$ is a promising n-type thermoelectric material. It is expected that by triggering textured grain growth one will be able to improve thermoelectric figure of merit of these materials. One of possible ways to induce textured grain growth is to add platelets seeds to the powder mixture prior to sintering. This is why we synthesized Nb-doped Sr$_3$Ti$_2$O$_7$ platelets seeds via molten salt synthesis (MSS). Due to crystallographic anisotropy, when grown in an appropriate medium and conditions, Sr$_3$Ti$_2$O$_7$ forms anisotropic platelets with ordered RP-type faults in [001] direction [1]. There have been several reports on synthesis of pure Sr$_3$Ti$_2$O$_7$ platelets with the MSS [2], but none on Nb-doped Sr$_3$Ti$_2$O$_7$. In order to study the crystallography, structure and chemical composition of synthesized platelets one has to be able to prepare thin foils for TEM observation in different orientations. This is why in our work we report on preparation of Nb-doped Sr$_3$Ti$_2$O$_7$ thin foils for TEM observation in defined orientation using two preparation procedures; namely conventional ion-milling and tripod polishing, and the comparison between them. The first step in both preparation procedures was to mix Nb-doped Sr$_3$Ti$_2$O$_7$ platelets with an epoxy (G1) resin and to press the mixture between Si supports in order to align as many as possible platelets parallel to the Si supports. The specimens prepared by ion-milling were then mechanically grinded to a thickness of 100 µm, followed by dimpling in a cross-section geometry down to a thickness of 16±2 µm. Afterwards, the specimens were Ar-ion thinned at 3.5 keV and at 8° incident angle to perforation in a Gatan, PIPS (without cooling) (Fig. 1). The specimens prepared by tripod polishing method [3] were polished on a diamond-lapping film (DLFs) at a wedge angle of 1° and afterwards mounted on a Cu grid. The specimens were additionally thinned in PIPS at 3.5 keV for 60 min and at 0.6 keV for 20 min. During the ion-milling process the specimens were cooled using liquid nitrogen (Fig. 2). The results showed that it was possible to find electron transparent, epoxy-free regions of Nb-doped Sr$_3$Ti$_2$O$_7$ platelets in cross-section geometry (c-axis parallel to the electron beam) for both specimens’ preparation procedures. However, the amorphous region in ion-milled specimens was app. 5 times as thick as compared to the specimens prepared by tripod polishing method. It was concluded that tripod polishing is far better technique for TEM specimen’s preparation of Nb-doped Sr$_3$Ti$_2$O$_7$ platelets despite its difficulty to carry out.

References


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Fig. 1: (a) SEM micrograph of Nb-doped Sr$_3$Ti$_2$O$_7$-platelets. (b,c) TEM micrographs of an ion-milled platelet. (d) HRTEM showing large amorphous layer.

Fig. 2: (a) SEM micrograph of Nb-doped Sr$_3$Ti$_2$O$_7$ platelets. (b,c) TEM micrographs of a tripod polished platelet. (d) HRTEM showing almost amorphous-free region.