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Deliverable 6.2

Report on protocols and standards developed in ESTEEM2

Deliverable leader 6 – Max Planck Institute for Intelligent Systems, Stuttgart

Protocol contributed by the Jožef Stefan Institute, Ljubljana

TEM sample preparation of cross-sections of platelet crystals

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Platelet crystals are often added to mixture of powders in order to trigger anisotropic and/or textured growth during sintering of materials. However, due to its morphology, it may be difficult to prepare cross-sections of platelet crystals along the crystallographic axis in which the growth rate is reduced. In view, of this the present protocol describes the procedure how to prepare cross-sections of such platelet crystals. As a test material $Sr_3Ti_2O_7$ Ruddlesden-Popper platelet crystals were used which were prepared by the molten salt synthesis (MSS). Due to crystallographic anisotropy $Sr_3Ti_2O_7$ forms anisotropic platelets as shown in Figure 1. Special attention was given to prepare thin foils of these platelets in the [001] crystal orientation.

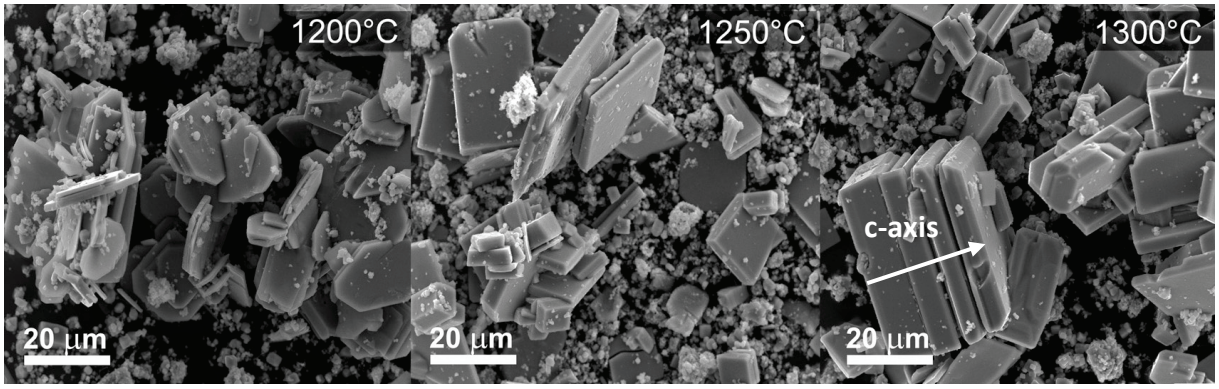

Figure 1: $Sr_3Ti_2O_7$ platelet crystals synthesized at different temperatures. The c-axis is marked in the last micrograph.

The first step in preparation procedure is to mix platelet crystals with an epoxy (G1) resin (Figure 2a) and to press the mixture between Si supports in order to align as many as possible platelets parallel to the Si supports. To avoid eventual damage caused by ion-milling, specimens are prepared by the mechanical tripod polishing method using an automatic tripod polisher (*Allied Multiprep System*). The platelets specimens are cut into slabs of ~1.5 mm wide and 2 mm long and attached to a pyrex specimen holder using *Crystal Bond* thermoplastic wax. The specimen is first polished using a diamond-lapping film (DLF) with 15-µm grain-size to provide a planar surface. This is followed by polishing with 6-, 3-, 1-, 0.5-, and 0.1-µm grain DLFs. A final polishing step is done on a polyurethane cloth using a silica solution with 50-nm particle size (*Allied Colloidal Silica Suspension*) in order to avoid

wedge chipping and scratches on the polished surface (Figure 2b). Prior to polishing the other side, the sample is removed from the pyrex by heating the polishing block on a hot plate. In order to polish the second side, the specimen is turned upside down and glued onto the pyrex specimen holder again. The specimen is thinned down to a thickness of app. 200 µm using 15-µm DLF. A wedge angel of 1° is introduced. The specimen is polished down to a thickness of 70, 50, 30, 10 µm using 15-, 6-, 3-, 1- µm DLFs, respectively. Subsequently, the final polishing is performed with 0.5-µm DLF and 50-nm colloidal silica to further thin the specimen wedge. After finishing all the polishing steps, the sample is glued on 3 mm Cu support half-grid (Figure 2c). The wedge-shaped specimens are additionally cleaned and Ar^{+} ion-beam thinned in a Gatan PIPS at 3.5 keV for 60 minutes and at 0.6 kV for 20 min (Figure 2d). During the ion-milling process the specimens are cooled using liquid nitrogen (L-N_2).

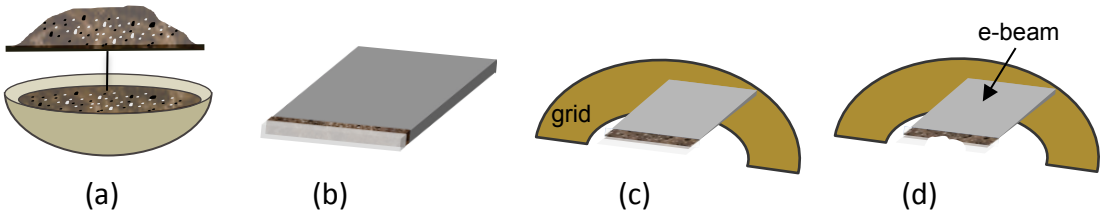

Figure 2: Schematic diagram showing different steps of tripod polishing with subsequent ion-milling.

Figure 3 shows electron transparent, epoxy-free regions of $Sr_3Ti_2O_7$ platelets crystals in cross-section geometry (c-axis parallel to the electron beam). Described procedure is suitable for the preparation of TEM cross-sections of any platelet-like crystals. The advantage is that described procedure enables to view a large number of crystals in the zone axis of lesser growth rate.

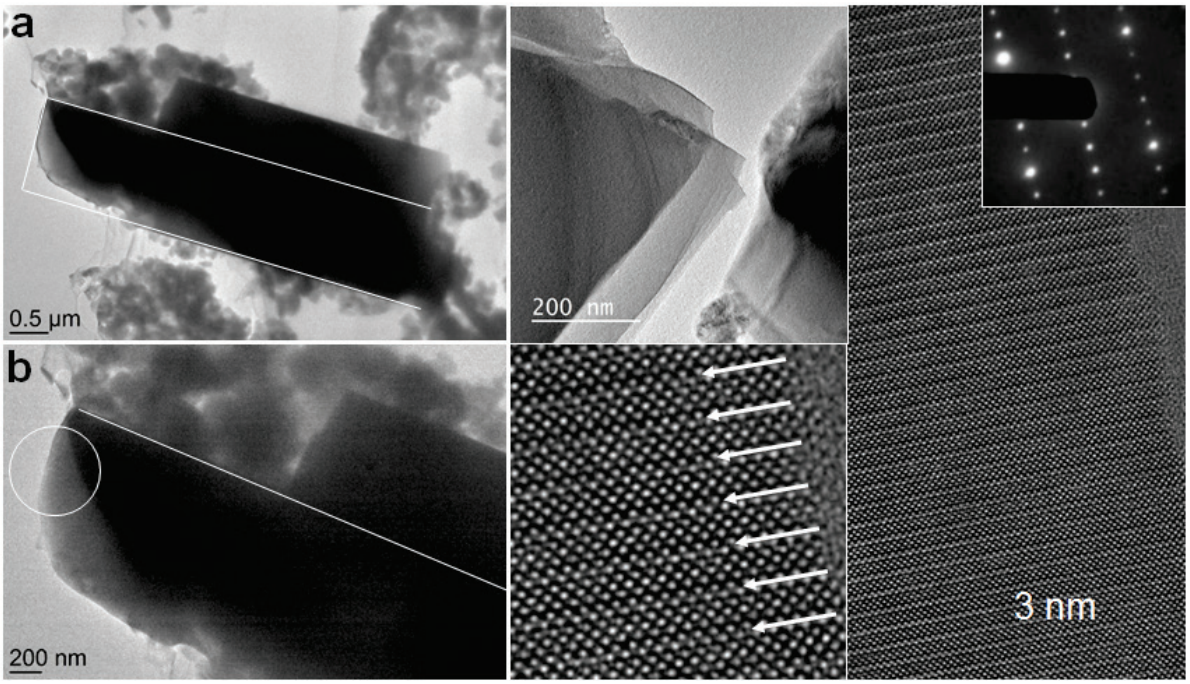

Figure 3: (a) Bright-field TEM micrograph of a cross-section of a platelet crystal. (b) Higher magnification showing thin region for HRTEM. (c) HRTEM showing perfect ordering of SrO layers between two perovskite blocks with the corresponding diffraction pattern.