In situ electric field transmission electron microscopy: Sample preparation & Experiment

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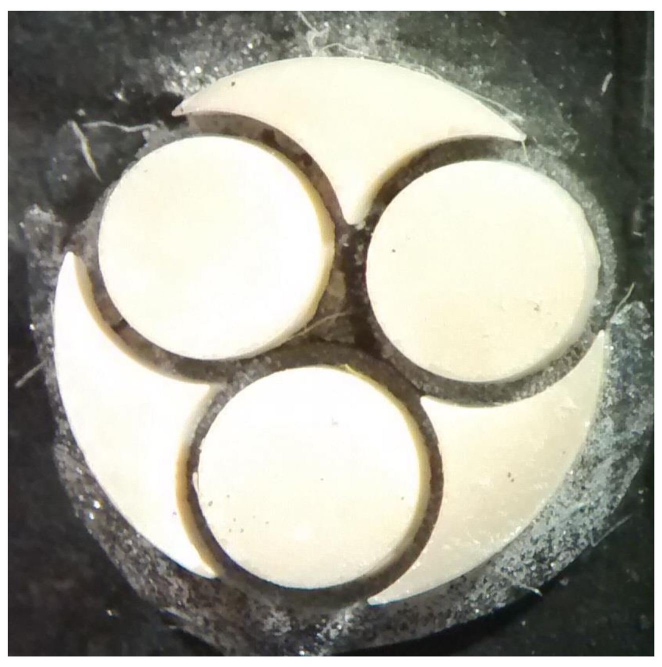
Introduction *In situ* transmission electron microscopy (TEM) of ferroelectric materials under an applied electric field allows insight in the processes that a material is undergoing on the microscopic scale. Performed sample preparation included standard processes like disc cutting, polishing and ion thinning. Subsequently, gold coated specimens were mounted into a modified *in situ* electric field TEM sample holder. With the chosen experimental setup electric fields perpendicular to the electron beam / viewing direction could be realized [1]. The microstructural evolution as a function of an applied electric field was studied in $Bi_{1/2}Na_{1/2}TiO_3$-0.25$SrTiO_3$ core-shell piezoceramic [2].

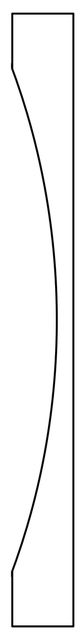
Sample preparation

Mechanical preparation



After cutting 3 mm diameter disks from the bulk material, the samples were mechanically ground down to a thickness of 120 μm and polished on one side.

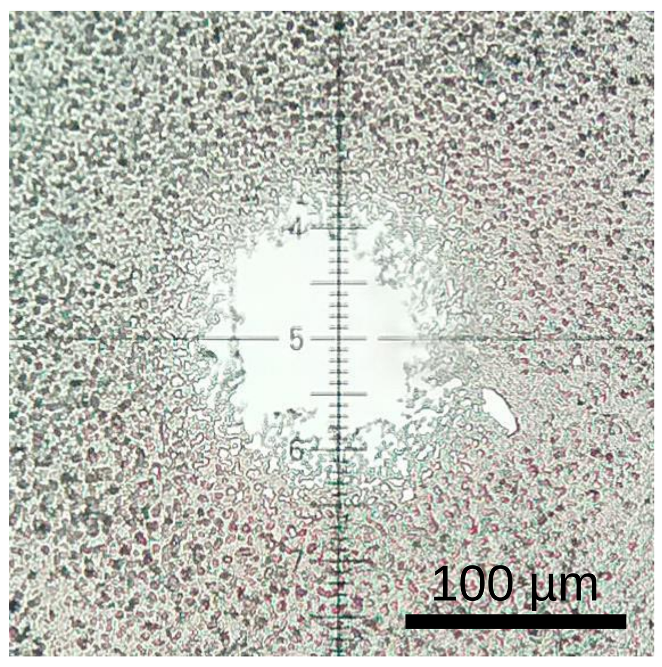
The other side was dimpled to retain one planar surface for later contacting. The dimple depth was adjusted to keep 20 μm of material in order to minimize bending of the sample.



Ion thinning

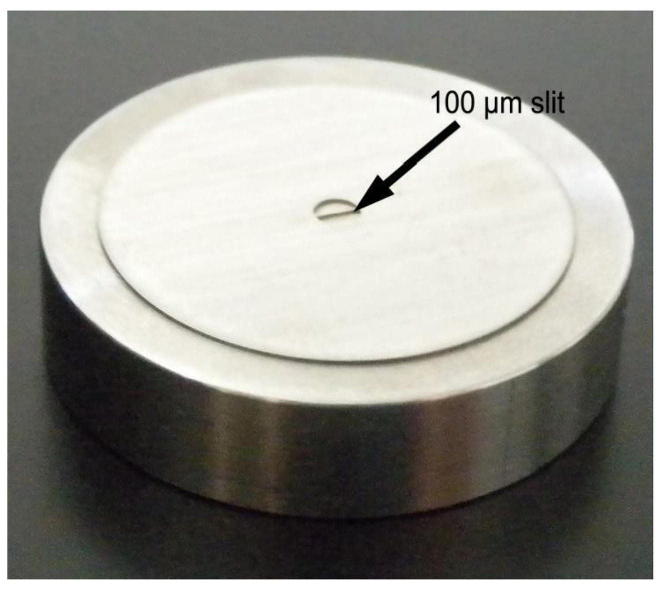
Depending on their composition the specimens were annealed above Curie temperature in order to relieve stress induced by the mechanical preparation processes.

Argon ion thinning was applied until electron transparency and a hole opening of 100 μm was reached.



In situ sample holder

Gold plating

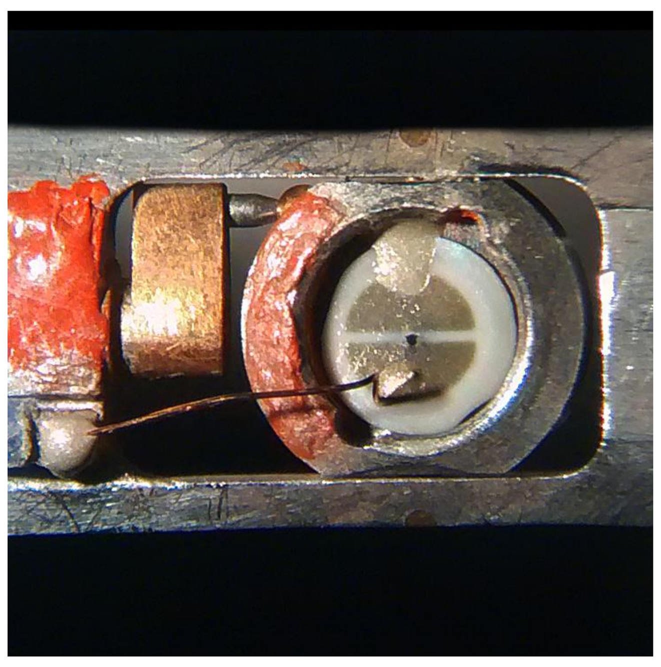


Gold electrodes were evaporated on the even side of the sample. Therefore, coating masks with different slit widths, ranging between 100 μm – 150 μm, were designed. A parallel electrode geometry was achieved with the electric field oriented perpendicular to the propagation direction of the electron beam.

Contacting

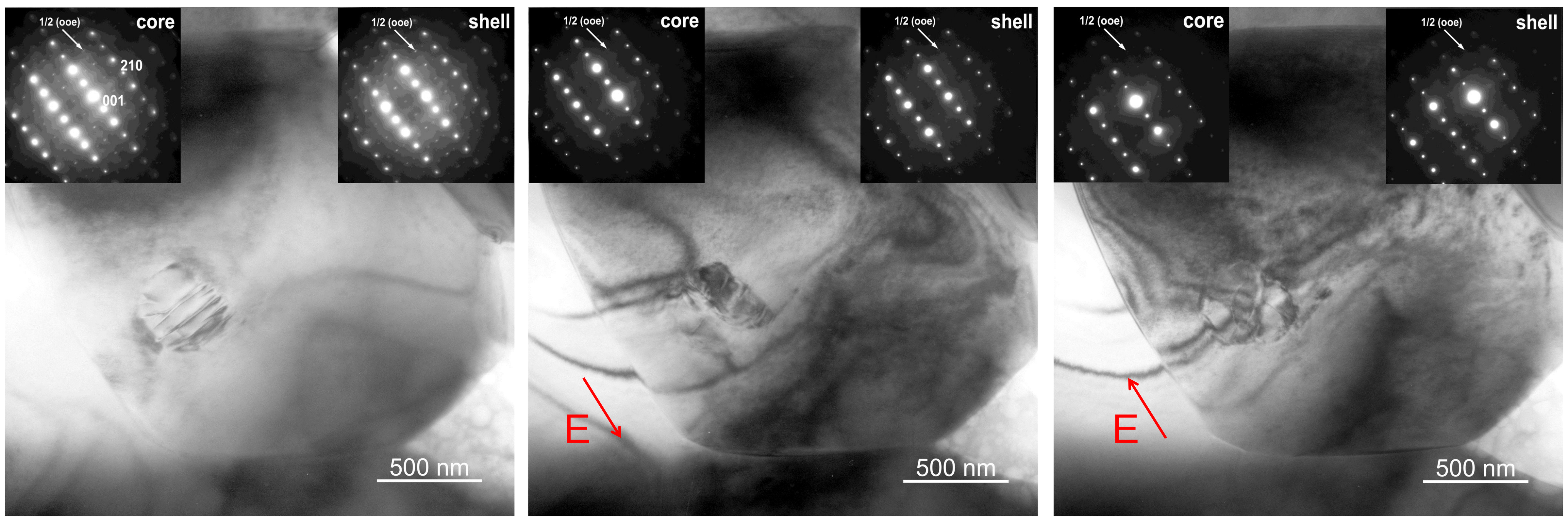
Electrical wiring was provided through the rod to the tip of the *in situ* TEM specimen holder.

The sample is mounted using an insulating varnish. The electrodes are then contacted with a copper or platinum wire to maintain β-tilting. Electric contacts were provided with the use of conductive epoxy glue.



Experiment

E = 0 kV/mm | **+4 kV/mm** | **-6 kV/mm**



Bright field imaging series of a $Bi_{1/2}Na_{1/2}TiO_3$-0.25$SrTiO_3$ sample along <120> zone axis. Corresponding selected area electron diffraction (SAED) patterns of core and shell region are shown in the inset. In the initial state (0 kV/mm) the core is composed of (001)-domain walls, whereas the shell features homogenous contrast, both showing the presence of ½ (ooe) superlattice reflections (SR). Under an electric field along <001>-direction a reorientation of domain walls within the core region is visible. The corresponding SAED patterns show a decrease in SR intensity.

Summary The shown TEM sample preparation technique and the *in situ* electric field characterization allows the observation of the impact of an electric field on various functional materials. In this study, a perpendicular electrode geometry was realized. The *in situ* experiment showed a distinct response of the core-shell structure as a function of electric field. The irreversible reorientation of (001)-domain walls within the core was monitored, whereas the shell featured reversible changes in contrast.

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[2] M. Acosta, M. C. Scherrer, Wook Jo, L. A. Schmitt, M. Deluca, H.-J. Kleebe, J. Rödel, W. Donner, J. Am. Ceram. Soc., submitted (2014).

Acknowledgements: Financial support by the Deutsche Forschungsgemeinschaft, Sonderforschungsbereich 595 is gratefully acknowledged