Ceramics, Oxides, Geomaterials

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In situ mechanical polarization of polycristalline $BaTiO_3$ in the ESEM with subsequent EBSD investigations

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$BaTiO_3$ belongs to the class of ferroelectric materials, the electric analogs of ferromagnetics. It crystallizes in the simple perovskite structure, where the barium ions are positioned at the corners of a cube. The centre of this cube is occupied by the smaller titanium ion, which is coordinated by six oxygen ions forming an octahedron. For temperatures above 127°C the unit cell is cubic (unit cell parameter a=0,401 nm at 150°C) and $BaTiO_3$ is paraelectric. On cooling below 127°C it undergoes a first order phase transition and the central titanium ion, the surrounding oxygen ions and the barium ions move away from the centre of the unit cell. This causes a tetragonal distortion of the unit cell (a=0,399 nm and c=0,403 nm at room temperature) "Figure 1". As a result the positive and negative charges separate and a permanent dipole moment is created, which leads to a spontaneous polarization along the c-axis of the unit cell.

The symmetry changes from cubic to tetragonal and $BaTiO_3$ becomes ferroelectric. In ceramics these dipoles are not randomly oriented within the grains [1]. To minimize energy due to mechanical stresses inside the grain and between individual grains (most of them caused by the phase transition at 127°C), unit cells with the same orientation of the polar axis form so-called domains. Two different types of boundaries between two domains are possible in tetragonal $BaTiO_3$: 90° und 180° domain walls. Only the 90° domains have the capability to reduce the elastic energy for the tetragonal structure [2]. There are two types of domain structures in coarse grained $BaTiO_3$, one called herringbone, which is the most common in unpoled ceramics, and the other square net pattern [3].

The ferroelectric behavior is based on the fact that the direction of the polar axis (and the direction of the c-axis) can be changed by an external electric field, so called poling. Also mechanical stress can induce a preferred orientation of the overall polarization. In this work domain wall motion of polycrystalline $BaTiO_3$ was recorded under mechanical stress in situ in the ESEM (Environmental Scanning Electron Microscope). The samples were imaged by crystal orientation contrast, an easy and quick method for the investigation of the microstructure of ceramic materials, enabling the observation of respective changes during in situ experiments. Crystal orientation contrast caused by orientation anisotropy of backscattered electrons can generate images in which grains of different orientations in polycrystalline material have different grey levels [4]. Even ferroelectric domains can be observed because of their differing polarization axes giving rise to twin boundaries.

Commercially available samples of $BaTiO_3$ ceramics were used for this investigation. From such sintered pellets rectangular blocks of 7x7x5 mm^3 were cut with a diamond wire saw and then one of the 7x7mm^2 faces was ground on silica carbide paper, finished with 4000 grit and then polished using a 0.25 µm diamond paste. The surface was mechanically fine polished using 0.04 µm SiO_2 emulsion to obtain the smooth surface required for good-quality OC (orientation contrast) images. The compression tests were carried out in an ESEM Quanta 600 FEG from FEI using the low vacuum mode and equipped with a slightly modified tensile stage from Deben (load cell: 1250 N, "Figure 2"). To determine the orientation of the grains before and after the compression experiments, EBSD (Electron Backscattered Diffraction) measurements were performed. Domain wall motion was observed at a stress exceeding 33 MPa resulting in mechanical poling "Figure 3".

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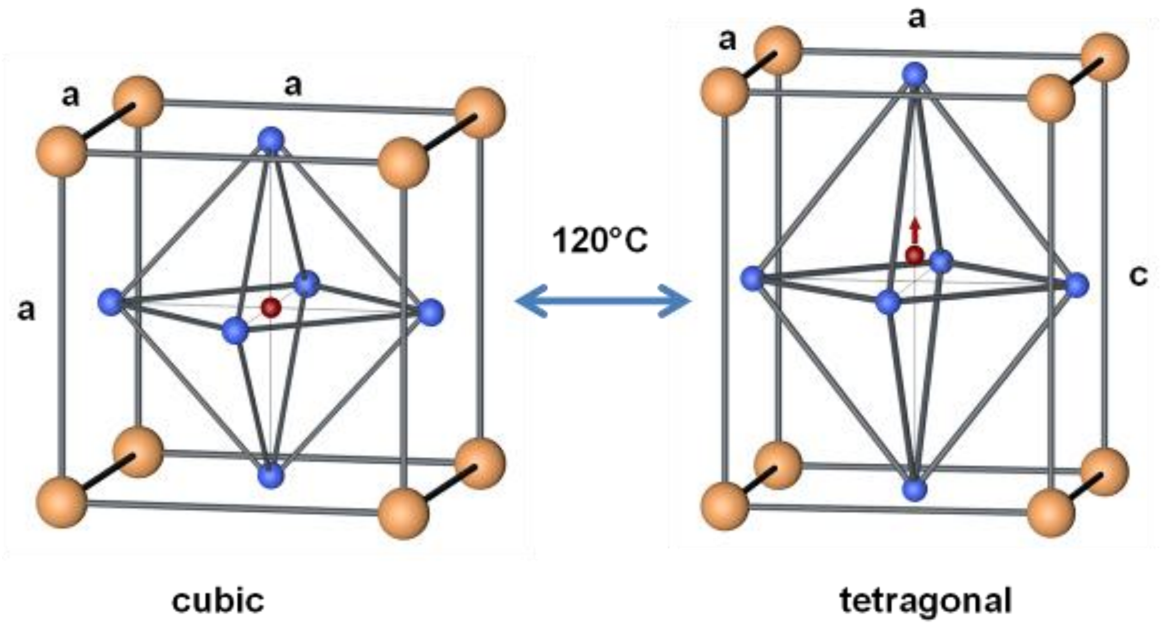

Figure 1. Unit cells of cubic and tetragonal $BaTiO_3$, yellow: Ba^{2+}, blue: O^{2-}, red: Ti^{4+}

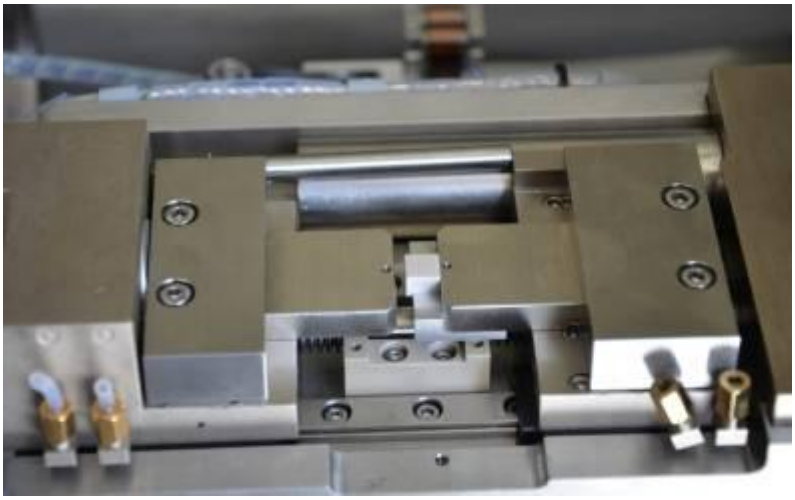

Figure 2. Tensile stage from Deben, with the ceramic sample mounted on it

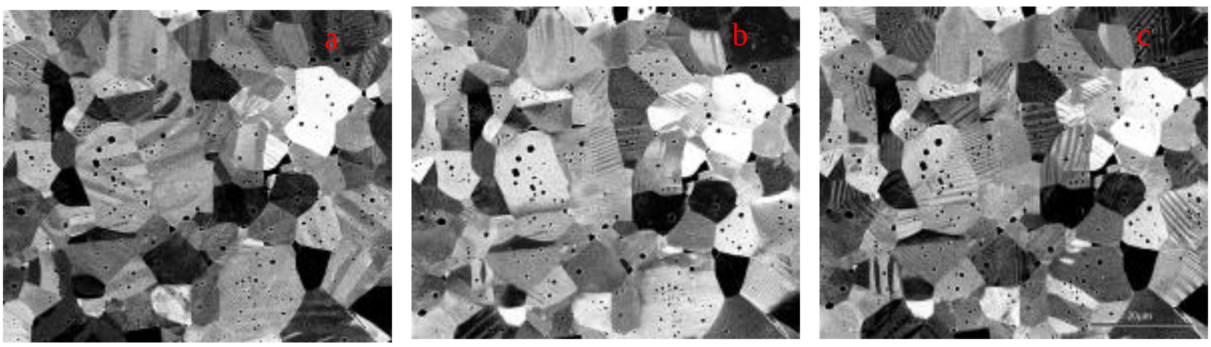

Figure 3.SEM-images of $BaTiO_3$ before (a), during (b) and after (c) compression