DIRECT OBSERVATION OF FERROELECTRIC DOMAIN WALL MOTION UNDER MECHANICAL STRESS IN THE ESEM

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ABSTRACT

A domain structure within single crystals, polycrystalline ceramics or thin films is a commonly observed feature of ferroic materials (ferroelectrics, ferromagnetics and ferroelastics) below a typical transition temperature usually referred to as Curie point. The formation of domains is a result of the minimisation of electric or magnetic stray field energy and/or elastic energy. Ferroelectric materials respond to mechanical stress with a polarisation. This polarisation consists of an intrinsic part due to compression of the unit cell and an extrinsic part due to domain wall motion. Such a response can be observed in piezoelectric ceramics and is described as mechanical poling. The mechanical behaviour is called ferroelastic effect. The direct observation of the domain wall motion under mechanical stress provides a tool for evaluating the extrinsic contribution to the materials with respect to reversible and irreversible parts.

The samples were imaged by orientation contrast, an easy and quick method for investigating 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. Even ferroelectric domains can be observed because of their differing polarisation axes giving rise to twin boundaries. As test sample barium titanate, $BaTiO_3$, a ferroelectric material with a Curie temperature of 127 °C was investigated. From such sintered pellets rectangular blocks of 7×7×5 mm^3 were cut and one of the 7×7 mm^2 faces was polished first with diamond paste. Due to the very low information depth of about 5 - 50 nm of the signal, a final polishing step using colloidal silica is necessary to get a fully distortion free surface.

In situ investigations were carried out in an ESEM equipped with a tensile stage using the compression mode. Domain wall motion was observed at a stress exceeding 33 MPa resulting in mechanical poling.