Direct observation of ferroelectric domain wall motion under mechanical stress in the ESEM

Angelika Reichmann[1], Klaus Reichmann[3], Armin Zankel[2], Peter Poelt[1,2]

1. Austrian Centre for Electron Microscopy and Nanoanalysis, Steyrergasse 17, 8010 Graz, Austria
2. Institute for Electron Microscopy, Graz University of Technology, Steyrergasse 17, 8010 Graz, Austria
3. Christian Doppler Laboratory for Advanced Ferroic Oxides, Graz University of Technology, Stremayrgasse 9, 8010 Graz, Austria

Introduction

A domain structure within single cystrals, 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 [1]. The formation of domains is a result of the minimization of electric or magnetic stray field energy and/or elastic energy. Ferroelectric materials respond to mechanical stress with a polarization. This polariaztion 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 (Fig.2). The direct observation of domain wall motion under mechanical stress provides a tool for evaluating the extrinsic contribution to the materials polarization with respect to reversible an irreversible parts.
The samples were imaged by orientation contrast (Fig.4), an easy and quick method for investigating the microstructure of ceramic materials, enabling the observation of respective changes during *in situ* experiments [2].

Experimental

Tensile stage in the ESEM

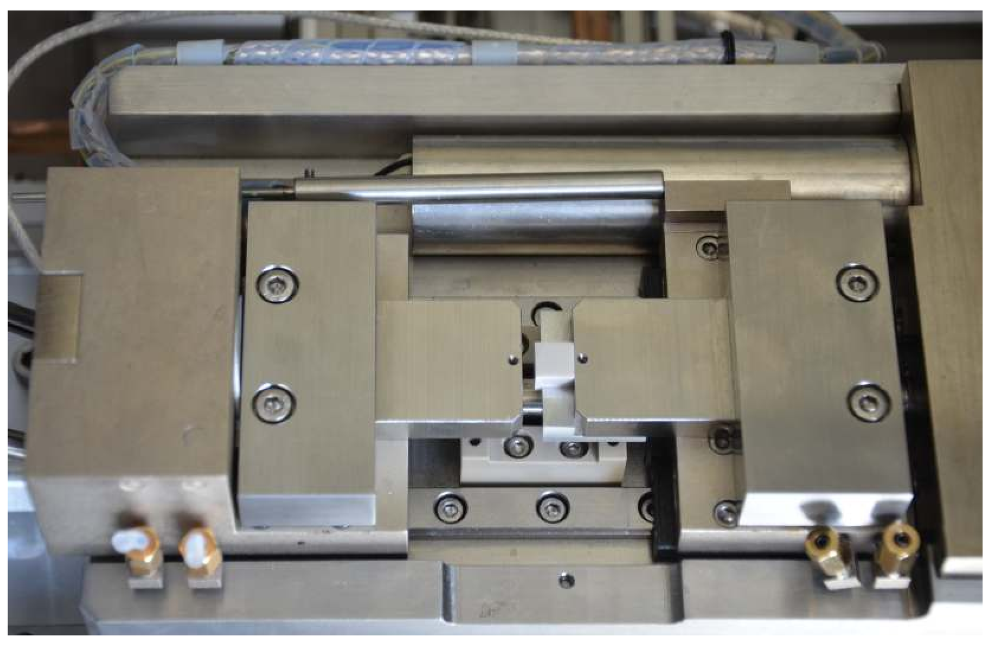

Fig.1: Photograph of the tensile stage

Sample preparation

- sintering
- sawing
- grinding
- polishing
- final polishing with Silicagel

SEM detector and parameters

- large electron probe current
- annular semiconductor detector below the polepiece
- low working distance
- accelerating voltage around 10 kV

Stress-strain diagram of ferroelastic materials

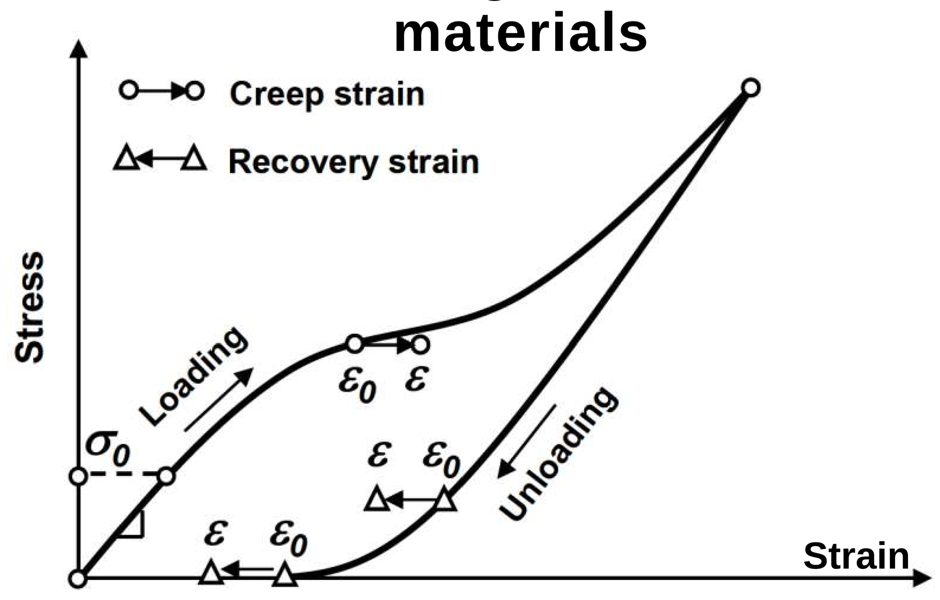

Fig.2: Stress-strain diagram of ferroelastic materials

Lit: E.H.K.Salje(1990) Phase transistion in ferroelectric crystals, Cambridge

The $BaTiO_3$ powder was pressed into cylindric tablets with a diameter of 13 mm and a thickness of about 10 mm. After sintering at 1380°C for 6h the cylinder was diced into a block of 7x7x5mm^3. The surface was grinded, polished and finally treated with Silicagel. The compression test was carried out using an MT5000 Tensile Stage (load cell: 1250N) from Deben mounted on the stage of an ESEM Quanta 600 equipped with a Schottky emitter.

Results

Domain wall motion in $BaTiO_3$ during compression

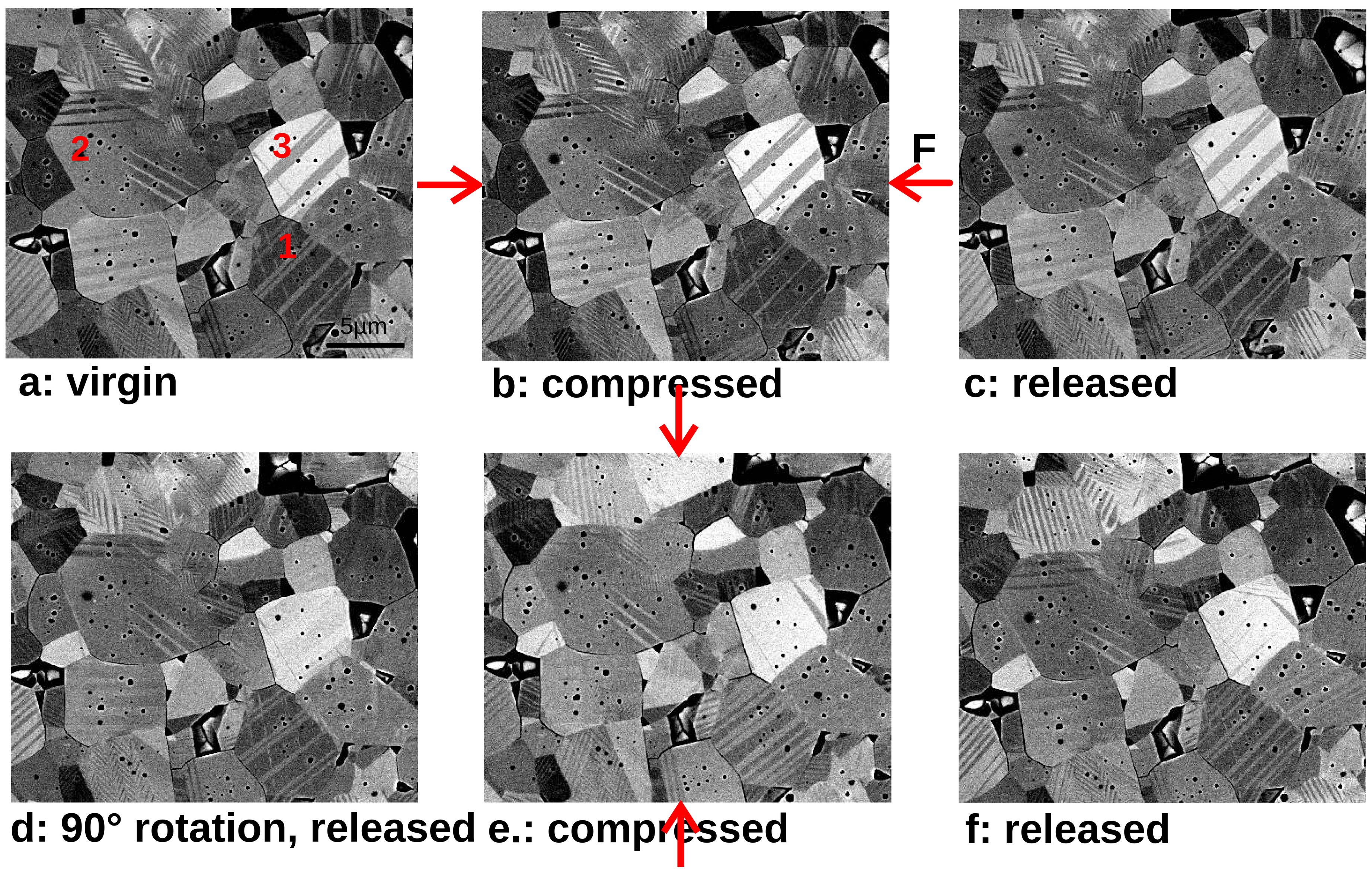

Fig.3: Domain wall switching during compression

The image series in fig.3 starts with the domain structure of the virgin sample (a). Image (b) shows the changes due to compressive stress applied in the direction of the red arrows. In some grains the domain size alters (grain 1) and some domains disappear (grain 2). In Fig.5 one can see a schematic representation of 90° domain structures and the domain wall motion induced by compressive stress [3]. After stress release (c) the virgin state is recovered to a major extent. Some changes are not reversible (grain 2). Rotation of the sample by 90° (d) and reapplied compression (e) causes in some cases domain rotation (grain 3).

Orientation contrast

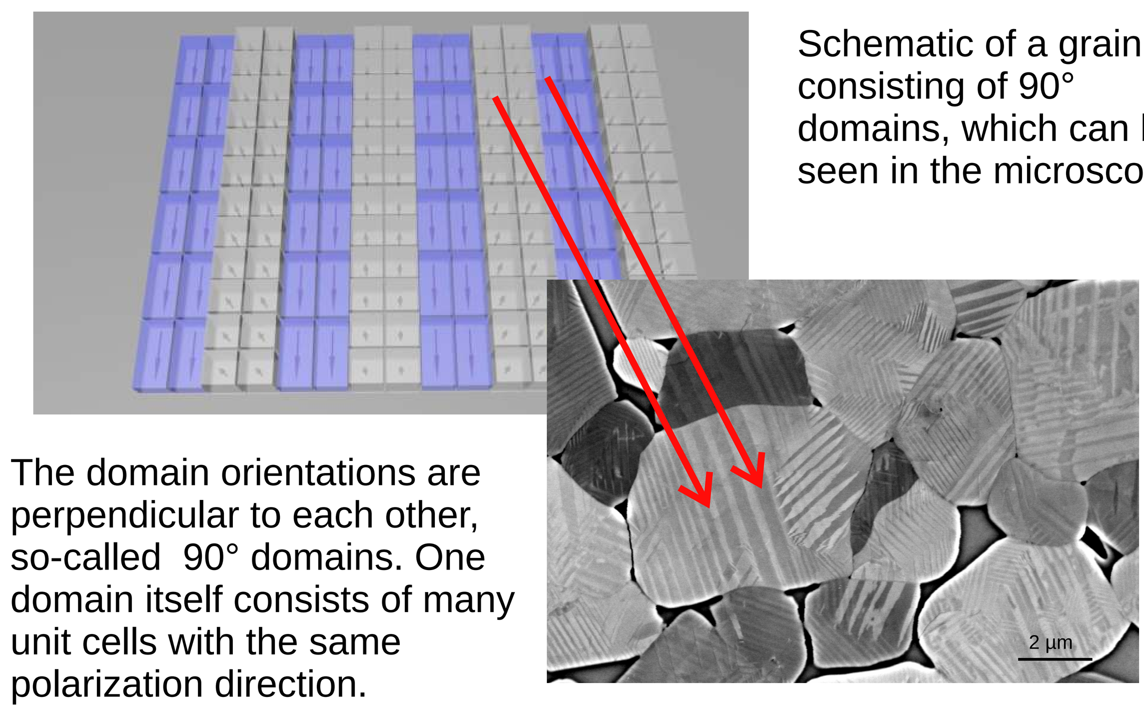

Fig.4: Imaging of 90° domains in the SEM

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.

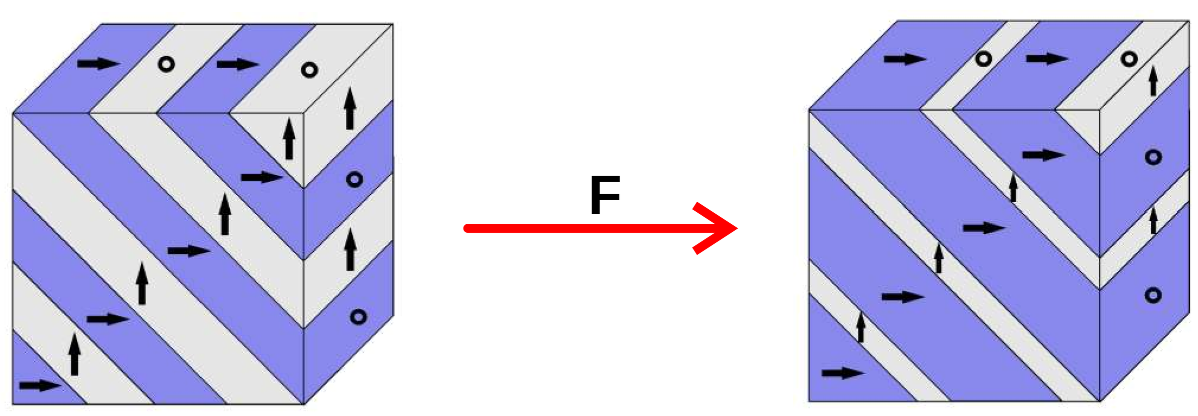

Fig.5: Schematic of domain wall motion during compression

The small arrows indicate the direction of spontanouspolarization in each domain.

References

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Conclusions

- ☺ Orientation contrast enables the imaging of the microstructure of ceramic materials in an easy way
- ☺ Visualization of domains
- ☺ In situ studies of domain wall motion are possible
- ☹ Not all grain and domain boundaries can be imaged

Contact

World Wide Web: www.felmi-zfe.at
email: angelika.reichmann@felmi-zfe.at

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