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Grain growth anomalies and their effect on domain structure in Barium Titanate

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Barium Titanate is a classical ferroelectric material that is widely used as dielectric in multilayer ceramic capacitors. In the ferroelectric state, which is observed below the Curie-Temperature of about 127°C, each unit cell of the compound develops a dipole moment, whose direction can be changed by an external electric field. This feature is responsible for the large relative permittivity of the material but also for a typical domain pattern inside each grain. Domains are regions of parallel orientation of the dipoles. Within the tetragonal phase of Barium Titanate 90° and 180° orientations between different domains are possible. The domain structure is given by the minimization of stray field energies (180° domain walls) and elastic energies (90° domain walls) [1]. The dielectric properties of barium titanate ceramics with respect to temperature, electric field, frequency and time strongly depend on the substitution of minor amounts of other ions (sometimes impurities in the ppm range) for Ba or Ti. In some cases such dopants influence domain structure and domain wall motion, which in turn affects the permittivity and a large fraction of the dissipation factor [2]. Thus an understanding of the domain structure of ferroelectric ceramics greatly assists the control of dielectric properties. In this study we investigate the effect of Zirconium Oxide contamination on the microstructure and the domain structure of Barium Titanate. This Zirconium Oxide could be introduced as contamination from milling media that are used for milling the material after solid state synthesis. Barium Titanate powder (BESPA BT-4-A from Nippon Chemical Industrial) with an average grain size of 0.3 µm was milled in a planetary mill (Fritsch Pulverisette 4) in 250 ml beakers made of yttrium stabilized zirconium oxide with 5mm milling balls made of the same material as the beakers with water as suspending liquid for six hours. In order to exaggerate the abrasive wear of the milling media the ratio between Barium Titanate powder and milling balls was chosen out of the recommended range. 15 g of Barium Titanate powder was milled with 500 g of milling balls. The main rotor was operated at 300 /min the planetary rotors were set at - 600 /min. The powder was dried after milling, mixed with poly-ethylenglycol as binder, sieved and pressed in a 13 mm diameter pellet die with 150 MPa. After debinding these pellets were sintered at 1280°C for two hours. For comparison pellets from Barium Titanate powder as received without milling were sintered in the same run. The sintered samples were grounded 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 SiO₂ emulsion to obtain the smooth surface required for good-quality OC (orientation contrast) images. The orientation contrast imaging was done with an ESEM from FEI using the low vacuum mode. Figure 1 shows the microstructure and domain distribution of the sintered Barium Titanate without milling step. The sample exhibits a homogeneous microstructure with an average grain size of 2.1 µm. The domains show a typical 90° herringbone pattern. Figure 2 shows the microstructure and domain distribution of the sintered Barium Titanate after the milling step. Abnormal grain growth with a preferred growth direction can be observed. Many grains exhibit a “coffee bean” shape with a twin boundary dividing the grain in two halves. The domain pattern, which is much coarser than in the reference sample, usually crosses this twin boundary but exhibits a change in domain orientation.

1. R. Waser, U. Böttger, S. Tiedke, in „Polar Oxides“, Wiley-VCH Verlag GmbH, Weinheim (2005), p. 30ff.
2. A.J. Moulson, J.M. Herbert in “Electroceramics”, Second Edition, John Wiley & Sons, Chichester (2003), p. 311ff.

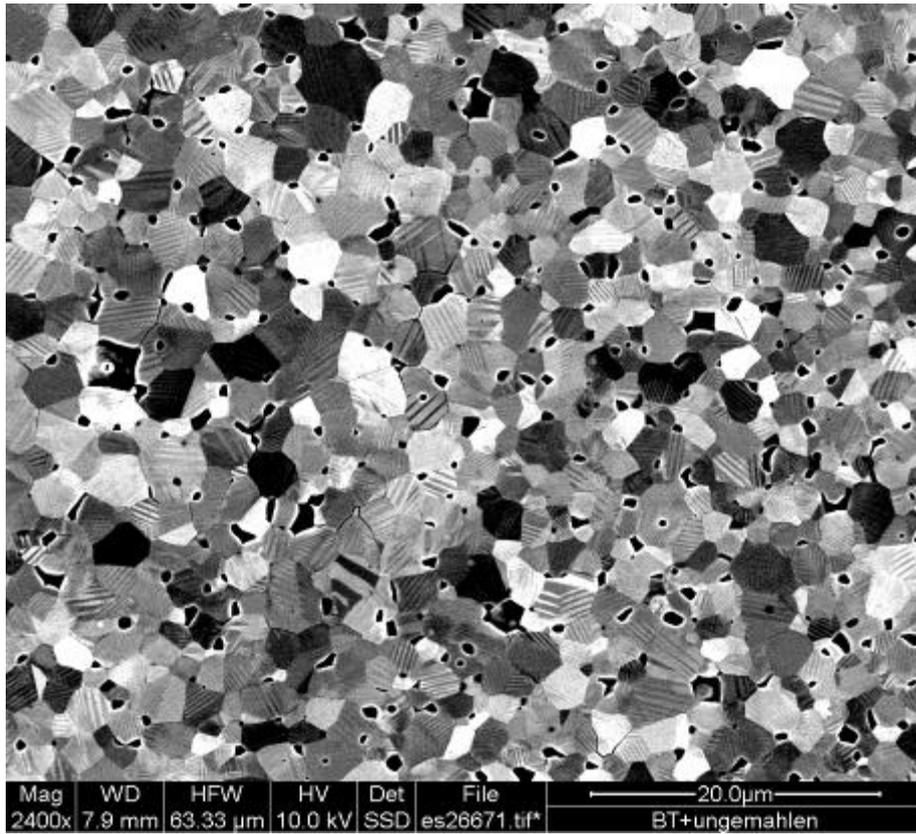


Figure 1. Microstructure and domain distribution of sintered Barium Titanate without milling step.



Figure 2. Microstructure and domain distribution of sintered Barium Titanate after the milling step.