doi:10.1017/S1431927614009532
Microsc. Microanal. 20 (Suppl 3), 2014

Live Imaging of Reversible Domain Evolution in $BaTiO_3$ on the Nanometer Scale Using in-situ STEM and TEM

Alina Schilling[1], Bastian Barton[2], Joerg R. Jinschek[2], Luigi Mele[2], Pleun Dona[2], Jan Ringnalda[2], Miryam Arredondo[1], Joshua F. Einsle[1], Marty Gregg[1]

1. Department of Physics and Astronomy, School of Mathematics and Physics, Queen's University Belfast, UK, BT7 1NN
2. FEI Company, Europe NanoPort, Achtseweg Noord 5, 5651 GG Eindhoven, The Netherlands

There is an increasing interest in novel ferroic materials, especially in device applications such as transistors, memory devices, tunneling barriers, etc.. The functionality of such materials is enabled by the reversible switching between equivalent states (or domains) that form to minimize the system's free energy. This switching behavior depends strongly on the domain structure pattern and their mobility under external stimuli (electrical, mechanical, temperature, etc.). There is a strong need to study this switching in detail. Nanoscale domain structures and their specific switching behavior strongly influence the material responses and properties such as dielectric permittivity, piezoelectric coefficients and remnant polarization. Fortunately *in-situ* (scanning) transmission electron microscopy (S/TEM) represents a powerful technique for studying ferroic materials and their switching behavior with resolutions down to the atomic scale. Here, the domain pattern evolution in $BaTiO_3$ (BTO) has been investigated *in-situ* under applied temperature -from room temperature to the critical transition temperature *(Tc).* In case of BTO, laminate domains advanced in two groups perpendicular to each other, intersecting and forming intricate square patterns (see Fig. 1). We have investigated transverse focus ion beam (FIB) prepared lamellae (~ 100 nm) of a BTO single crystal in both high-angle annular dark field (HAADF) STEM and phase contrast TEM mode, using a FEI Tecnai TF20 TEM at 200 keV electron energy. For *in-situ* imaging at elevated temperatures, the lamellae were suspended over a 1 µm hole of the thin amorphous SiN membrane of an FEI MEMS-based heating holder. The temperature of the SiN membrane can precisely be varied between room temperature and >1000°C. At room temperature, two perpendicular groups of domain lamellar structures (80 and 40 nm width) could be clearly observed in both TEM and STEM modes (Fig. 1a). When the sample was cycled from room temperature up to a temperature of 120°C ± 10°C (Tc), a gradual decrease in the contrast and number of domains was observed, until the pattern has completely vanished at temperatures above 120°C (Fig. 1d). The reversed process could be observed, when cooling the sample back, with a transition temperature ~10-20°C lower than in the case of heating. The physical background of this hysteresis effect and the origin of this pattern have to be further explored. At constant temperature, the patterns showed to be stable, so that no change was observed after 60 mins at ~80°C (Fig. 2)

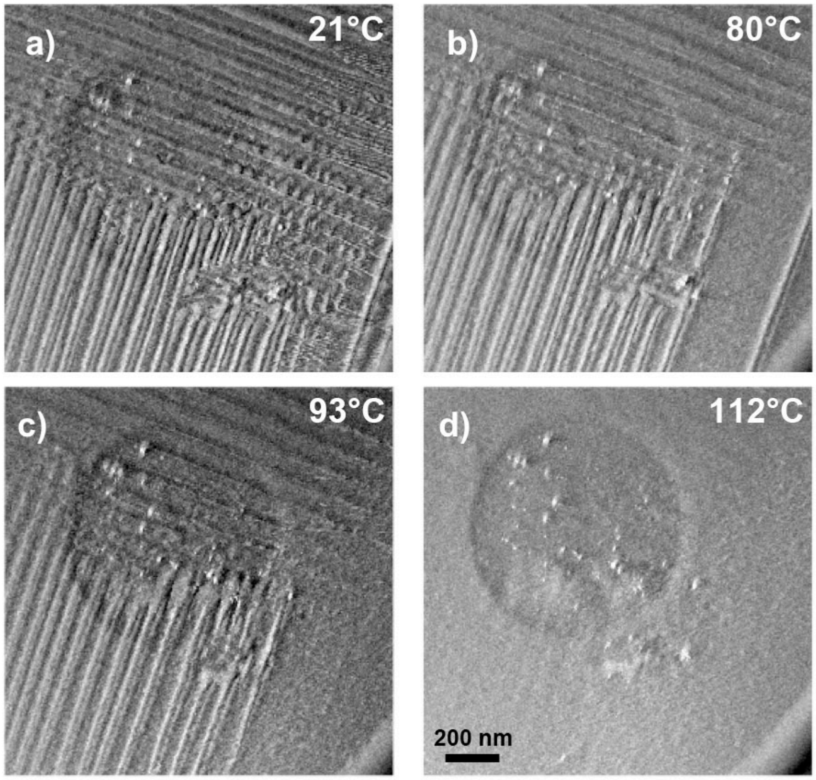

Figure 1. HAADF-STEM image. BTO - domain evolution as a function of temperature: (a) 21°C, (b) 80°C, (c) 93° and (d) 112°C.

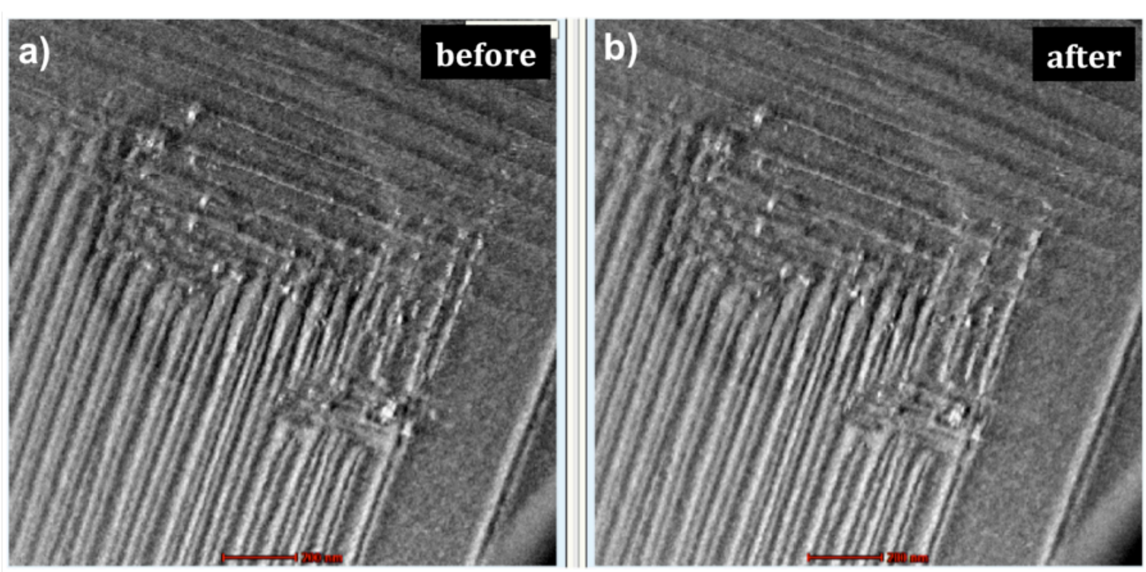

Figure 2. HAADF-STEM image of BTO domains at a constant temperature of) 80°C over 1 hr. (a) before and (b) after. Domains are stable and domain evolution is completely reversible under applying heating (see Fig.1).

https://doi.org/10.1017/S1431927614009532 Published online by Cambridge University Press