

Two-step evolution mechanism of multi-domains in BaTiO₃ single crystal investigated by in situ transmission electron microscopy

Z.H. Zhang, X.Y. Qi and X.F. Duan*

Beijing National Laboratory for Condensed Matter Physics, Institute of Physics, Chinese Academy of Sciences, Beijing 100080, China

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The electric-field-induced evolution of 90° stripe multi-domain structures in BaTiO₃ single crystal have been investigated by transmission electron microscopy (TEM). The polarization vector of the matrix is against the direction of external field at the initial state. Upon application of the electric field, a two-step domain-switching mechanism is observed. The stress produced by 90° domain switching has important effects on the evolution of domains in the thin TEM specimen. The result is very different from that observed in optical microscopy.

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Understanding the response mechanism of ferroelectric domains to an external field has been an active research area because of its technical importance [1–4]. Experimental work has focused on the study of macroscopic properties, such as the measurement of the ferroelectric hysteresis loop. Direct observations of microscopic domain-switching behavior, however, are scarce. Studies of the details of ferroelectric domain configurations and domain wall movement are helpful for understanding the basic physics involved in ferroelectric switching and thus realizing the potential of ferroelectric materials as the high-density storage devices [5]. Since the size of the ferroelectric domain ranges from nanometers to micrometers and the switching process is limited to a few micrometers, transmission electron microscopy (TEM) is ideally suited for observing this domain. A recently developed in situ TEM method has been used successfully to study the microcracking, aging and domain switching of ferroelectrics by applying an electric field perpendicular to the electron beam on the TEM sample surface [6–9]. This method makes the judgment of the direction and magnitude of the electric field driving the specimen more precise. Our group has designed and fabricated a special TEM stage which can be used to apply an electric field in situ to the TEM specimen surface. Systematic investigations of the evolution of

the ferroelectric domains under an external field have been performed. (i) The initial domains gradually disappeared; new domains oriented at 90° to the original appeared, and the polarizations were switched to the direction of the external electric field [10]. (ii) A matrix with polarization vectors along the direction of electric field expanded and the domains disappeared gradually [11]. Here, we report the results related to the third case in which the polarization vectors of the matrix are against the direction of the electric field. We will provide the intrinsic physical mechanism of domain wall movement and new domain nucleation involved in ferroelectric domain switching for thin TEM specimens. It is interesting to find that accomplishing a 180° switching occurs via two distinct 90° switching events for the thin TEM specimen. The intrinsic stress produced by 90° domain switching has important effects on domain evolution. Our result for thin TEM specimens is very different from that observed by optical techniques and can help to understand the microscopic evolution behavior of ferroelectric domain switching in ferroelectric thin films.

The specimens used here for in situ TEM are BaTiO₃ single crystal, which has a typical tetragonal perovskite structure at room temperature. The steps for preparation of the TEM specimens are similar to the procedures in our previous field-driven in situ TEM studies [10,11]. The square shape of the specimen makes it more convenient to determine the direction of the domain walls and that of external field. An improvement should result

* Corresponding author. Tel.: +86 10 82648008; fax: +86 10 62561422; e-mail: xfduan@blem.ac.cn

from the use of our homemade specimen holder, which offers a large space in which to place the specimen. A high-voltage source with voltages ranging from 0 to 5 kV was utilized, which gave rise to a static electric field on the specimen surface. The TEM experiments were performed in a Philips CM200 microscope operated at 200 kV.

Figure 1a shows the morphology of the investigated ferroelectric domain structures in BaTiO₃ single crystal before the application of an external field. There are three sets of domains besides the matrix. Two sets of domains, indexed as series A (1–9) and series B (1–3), appear as perpendicular strips, and the domain boundaries are almost parallel to the [101] and $[10\bar{1}]$ directions, respectively. All the domains have pointed tips, some of which stop at the domain boundaries. Convergent-beam electron diffraction analysis indicates that domain series A and B have opposite polarization vectors and the domain boundaries are all 90° *a*–*a* type [12]. The third set, indexed as series C (1–3), on the lower left of Figure 1a is inclined at 45° to the other two sets. These are *c*-domains surrounded by *a*-domain matrix [12]. The domain walls exhibit fringes in the image contrast due to their inclined character.

A series of bright-field TEM micrographs (Fig. 1b–f) shows the process of domain switching in BaTiO₃ single

crystal under an external field. Assuming the polarization vector of the matrix is along $[001]$, the electric field is along the $[00\bar{1}]$ direction and increased from 0 kV cm^{−1} step by step. When the electric field is maintained at 0.32 kV cm^{−1} for 6 min, the domain patterns began to change. Figure 1b shows the morphology. The matrix shrunk through domain wall movement. The domain tips tended to move readily, and stop at the intersections with the other domain series (domains A4 in Fig. 1a and b, B3 in Fig. 1a and c, C2 in Fig. 1a and c). Such a growth of domain state by means of tip motion has already been observed by Snoeck et al. [13]. This motion is energetically easier compared with the lateral displacement of walls because it implies only displacement of the ions situated near the domain point [13]. With an increase in the applied field, the intersections showed significant resistance to the tip point movement. The domains were all wider, with tip angles larger than before. In particular, the tips of some domains became asymmetric under external fields. For example, one side of the tip of domains A3 and A5 was curved with a larger inclination angle to the straight domain wall, while the other side had a smaller inclination angle. The case was same for domains B2 and B3. Figure 1c shows the morphologies when the electric field was maintained at 0.32 kV cm^{−1} for 74 min; some nuclei appeared beside the domain tips. When the electric field was increased to 0.54 kV cm^{−1}, violent changes occurred. Figure 1d and e shows the morphology corresponding to an electric field of 0.54 kV cm^{−1} for 11 and 14 min, respectively. The nucleation of many new small, narrow *a*-domains caused the matrix to divide into small strips. The matrix was annihilated by the new domains through nucleation and exploitation and coalition. When the electric field was kept at 0.54 kV cm^{−1} for 24 min, all the domains disappeared (Fig. 1f) and the whole region observed became a monodomain.

In the present case, the polarization vectors of domain series A, B and C are 90° away from the field direction, while the matrix is unfavorably oriented because its polarization vector is antiparallel to the field. The polarization vectors of domains and the direction of external field are indicated by arrows in Figure 1. From the above analysis, the whole domain-switching processes for 90° *a*-domain strips in the TEM specimen can be divided into two stages. First, the polarization vector of the matrix rotated 90°, perpendicular to the direction of electric field. During this stage, the domain wall movements are initially more important to domain switching (see Fig. 1b). The matrix shrunk as all the series A, B and C expanded. However, at relatively higher fields, nucleation of the new 90° domains dominated the evolution of domain switching (see Fig. 1d). This ferroelectric domain kinetics was also observed in the epitaxial films [14]. In the second stage of the domain switching, the polarization vectors rotated again 90°, as observed in Figure 1f, so that the polarization vectors of the whole region were along the direction of external field. The evolution in the second stage is too fast to record. For 90° *c*-domain strips, the growth progress was observed under the external field. The polarization vectors of the *c*-domain rotated to the direction same

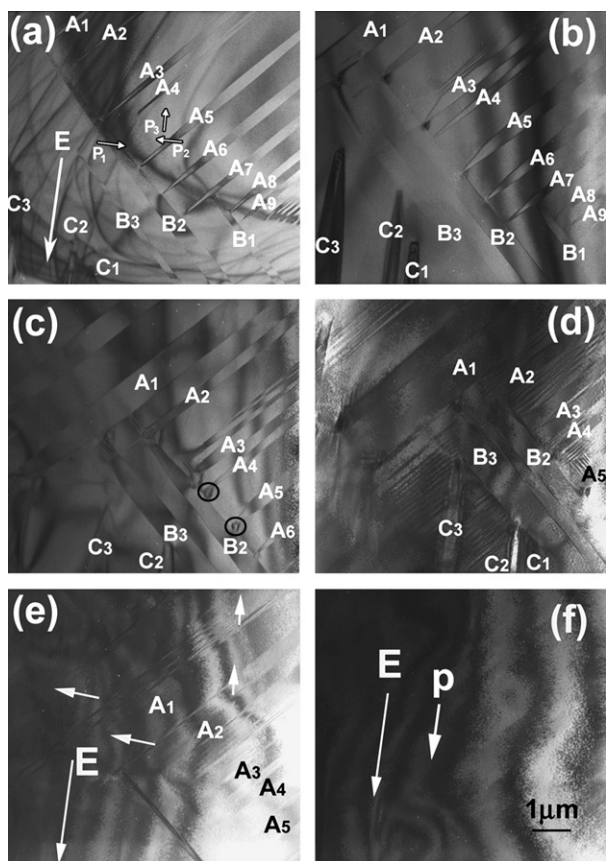


Figure 1. Bright-field TEM micrographs showing domain switching in the BaTiO₃ single crystal at different electric field levels: (a) initial morphology, 0 kV cm^{−1}; (b) 0.32 kV cm^{−1} for 6 min; (c) 0.32 kV cm^{−1} for 74 min; (d) 0.54 kV cm^{−1} for 11 min; (e) 0.54 kV cm^{−1} for 14 min; (f) 0.54 kV cm for 24 min^{−1}.

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