Domain Structure of BiFeO₃ Ceramics Determined by the Transmission Electron Microscopy

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Bismuth ferrite (BiFeO₃) has recently been subject of intensive research, driven primarily by its ability to exhibit both ferroelectric and antiferromagnetic ordering. In spite of many efforts, the basic electromechanical properties of BiFeO₃ ceramics are still not well characterized, the main reason being probably the difficulties related to the high electrical conductivity of this compound. We recently showed that in BiFeO₃ ceramics, which can withstand large electric fields (140 kV/cm) at low frequencies (0.1 Hz), it is possible to achieve large bipolar electric-field induced strain that can reach 0.36% (peak-to-peak value). This strain is comparable to that achievable in highly efficient Pb-based perovskite ceramics, such as Pb(Zr,Ti)O₃ and Pb(Mg,Nb)O₃–PbTiO₃. The large strain is likely associated with switching of predominantly non -180° domain walls, which, however, needs to be confirmed [1]. In order to make further steps in the understanding of the electromechanical response, a detailed microscopic analysis of the domain structure of BiFeO₃ ceramics is needed.

In the present study we investigated the domain structure of BiFeO₃ ceramics using transmission electron microscopy (TEM). BiFeO₃ ceramics was prepared by sintering a mechanochemically activated Bi₂O₃–Fe₂O₃ powder mixture at 760°C for 6h [2]. For the TEM investigation, the specimens were prepared by polishing using tripod polisher to reduce the sample damage associated with conventional ion milling.

Typical 50 nm sized domains in a BiFeO₃ grain are shown in Fig.1. In the rhombohedral BiFeO₃ (SG: R3mR, ICSD #20288) three different ferroelectric domain walls, which are labeled according to the angle formed between the polarization vectors on either side of the wall, are possible, i.e., 71°, 109° and 180° domain walls [3]. In order to identify the type of the domains, selected area diffraction (SAED) patterns were taken from different areas in the sample. The SAED patterns of the grains oriented in the [110] zone axes, taken on the domains from an area of ~700nm, showed splitting of the {111} reflections (see Fig. 2b, marked region). Such patterns can be experimentally observed only in the case in which the polarization vectors are inclined to each other by an angle of 71° and/or 109° (in the case of 180° domains there is no splitting). Using EMS program package [4] the peaks splitting were simulated for all types of domains, i.e., 71° and 109° (Figs. 2c and 2d). Fig. 2a and 2b represent experimental SAED patterns in [110] zone axis. In Fig. 2a no splitting can be observed, which indicate the presence of 180° domains in the sample. The splitting of (111) reflections in Fig. 2b can be explained by the presence of 71° domains as shown in simulated SAED pattern in Fig. 2c. At present, the splitting characteristic for 109° domain walls (Fig. 2d) was not found. These results confirm the presence of non-180° domain walls in the BiFeO₃ ceramics; switching of these domains can lead to large electric-field induced strain as reported recently [1].

References

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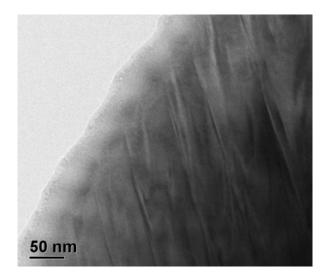


FIG. 1. TEM BF image of the domains in BiFeO₃ ceramics.

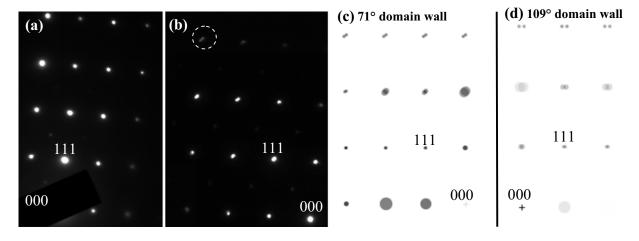


FIG. 2. Experimental SAED patterns from the domains in a BiFeO₃ grains in [110] zone axes where no splitting was observed (a) and where the splitting of the marked (333) reflections was observed (b). Splitting simulations using EMS program package where one domain is in [-110] and another in [-1-10] zone axes (c) and in [1-10] and another in [-110] zone axes (d).