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Direct Polarity Determination of Ferroelectric $Ca_{0.28}Ba_{0.72}Nb_2O_6$ Single Crystal by Combined Defocused Convergent Beam Electron Diffraction and Simulation

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The performances of ferroelectrics and related devices are largely dependent on configurations and dynamic behavior of ferroelectric domains which are characterized by electron polarization (polarity). Therefore determining the ferroelectric polarity in ferroelectric domains is of central importance from a viewpoint of not only scientific research but also technological applications. In this abstract, we show that defocused convergent beam electron diffraction (CBED) can be used for polarity determination in ferroelectrics. The advantage of combination of the shadow image and diffraction intensity in diffraction disks allows facile and fast link between polarity and specimen morphology, which would help to understand evolution of ferroelectric domains under various external fields.

$Ca_{0.28}Ba_{0.72}Nb_2O_6$ (CBN) is a typical relaxor ferroelectric material of tetragonal tungsten bronze structure with its polarization vector along c axis [1]. TEM specimen was prepared through mechanical polishing, dimpling and Ar-ion milling. An FEI Tecnai F20 microscope with an accelerating voltage of 200 keV was used. CBED patterns were obtained near [100] zone axis (about 3 degrees off, toward [010] direction) in a systematic row diffraction condition.

Two defocused CBED patterns were recorded at different specimen thicknesses, since CBED is susceptible to specimen thickness due to dynamic scattering. Fig. 1 (a) and (b) show two patterns recorded at thinner and thicker specimen respectively. It can be seen that the bright and dark regions of banded domains in (002) and $(00\bar{2})$ disks are reversed (as marked by "B" and "D" in Figure 1), as observed in (001) and $(00\bar{1})$ disks. This indicates that they are 180 degree ferroelectric domains, as revealed by Lu *et al*. [2]. However, the image intensities of the same domain in (001) and (002) disks are different at thinner specimen, while they become the same at thicker specimen, giving us ambiguity in determining the polarity according to the diffraction intensities in (001) and (002) disks. To correlate polarity with diffraction intensity in diffraction disks, CBED simulations were carried out [3]. Regarding the calculated CBN structure as a single domain, we explored the thickness dependence of the (001) and (002) diffraction intensity. Fig. 2 shows the simulated patterns along with the averaged intensity profiles along the dotted line rectangles. Specimen thicknesses corresponding to Fig. 2(a) and (b) are 20 nm and 35 nm respectively. From the intensity profile, it is found that when specimen is thin (20 nm), (001) diffraction intensity is lower than that of $(00\bar{1})$, which becomes stronger when the thickness increases. However, the (002) stands stronger than $(00\bar{2})$ all the time. The thickness dependence of (001) and (002) diffraction intensity stated above is in good consistency with the experimental patterns. The fact that (002) diffraction is less influenced by specimen thickness can be easily understood since (002) diffraction possesses a larger dynamic extinction distance ξ_g than (001) diffraction. Based on the discussions above, the polarity of each domain can be visualized by the red rows, as shown in Fig. 3.

In summary, we showed that the polarity of ferroelectric domain can be determined by defocused CBED patterns and simulations. This is highly beneficial in observation of dynamic evolution of ferroelectric domain under external fields. Our results suggest that care must be taken when accessing the direction of the polarization vector based on the diffraction intensities [4].

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[4] The authors gratefully acknowledge Prof. H. J. Zhang in Institute of Crystal Materials of Shan Dong University for supplying high-quality CBN single crystals.

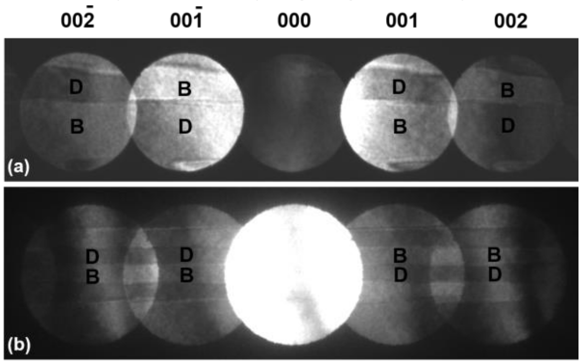

Figure 1. CBED patterns recorded at (a) thin region and (b) thick region.

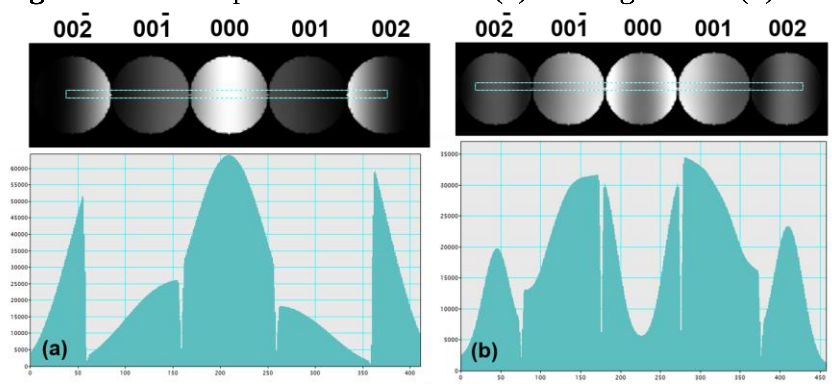

Figure 2. Simulated CBED patterns and the intensity profiles integrated along the dotted line rectangles at different sample thicknesses: (a) 20 nm; (b) 35 nm.

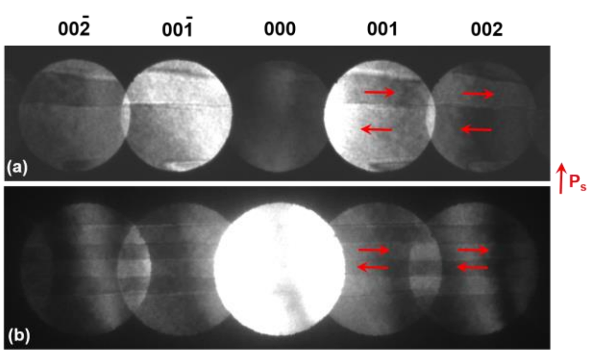

Figure 3. Experimental defocused CBED patterns with polarization vectors indicated in the domains.

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