

Investigation of KTP and BTO Ferroelectric Domain Behavior Through *In-situ* TEM Biasing

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Ferroelectric materials, exhibiting a spontaneous and electrically reversible polarization, hold great potential for a wide range of technological advances and are an actively researched class of materials. At the micron-scale, ferroelectric switching is governed by domain nucleation and domain wall motion. These dynamic processes are difficult to measure. In this study, we utilize in-situ transmission electron microscopy (TEM) to visualize ferroelectric domain switching at high spatial and temporal resolution in two ferroelectrics: Potassium Titanyl Phosphate (KTP) and Barium Titanate (BTO).

KTP has strong nonlinear optical properties and is used for second harmonic generation (SHG). Precise control of KTP domain morphology is necessary for efficient SHG, however, poorly understood switching kinetics make domain engineering difficult [1]. Polarization switching has been characterized through several techniques, including atomic force microscopy, piezoresponse force microscopy, and digital holography, yet these methods cannot provide both the temporal and spatial resolution of in-situ TEM [1, 2]. BTO has applications in ferroelectric memory as well as strain-mediated magnetoelectrics, yet much is still unknown about BTO's domain kinetics [3, 4].

In-situ electric biasing experiments were performed at Drexel's Central Research Facilities on a JEOL 2100 LaB₆ microscope using a custom biasing holder designed by M. L. Taheri and Hummingbird Scientific. A FEI DB235 Dual-Beam Focused Ion Beam was used for all sample preparation. KTP liftouts were constructed with a [010] surface normal. Domains were imaged using dark-field TEM imaging, as shown in Figure 1. Electric contacts were attached to the c+ and c- faces of the sample (the c-axis is the polar axis) through ion-beam platinum deposition. A monodomain KTP sample was biased at fields greater than KTP's coercive field, allowing visualization of domain nucleation. Secondly, a multi-domain sample was biased; domain wall kinetics were studied as a function of applied field. BTO samples were similarly tested.

Biasing of KTP crystals was performed using an accelerating voltage of 120 kV and a small (50 micron) condenser aperture. With these beam conditions, KTP samples were stable under the e-beam and suitable for biasing experiments. However, at 200 kV and a 200 micron condenser aperture, Rubidium doped KTP samples (RKTP) were unstable after extended exposure to e-beam radiation. At high beam dosages, we observed amorphization of the RKTP. At lower dosages, we observed ferroelectric domain motion, induced by the e-beam.

In-situ TEM is a unique characterization tool capable of providing high spatial and temporal resolution unattainable through competing techniques. Our experiments provide valuable insight towards domain kinetics in both KTP and BTO. Furthermore, the techniques utilized in our work can be transferred to many other ferroelectric materials.

References:

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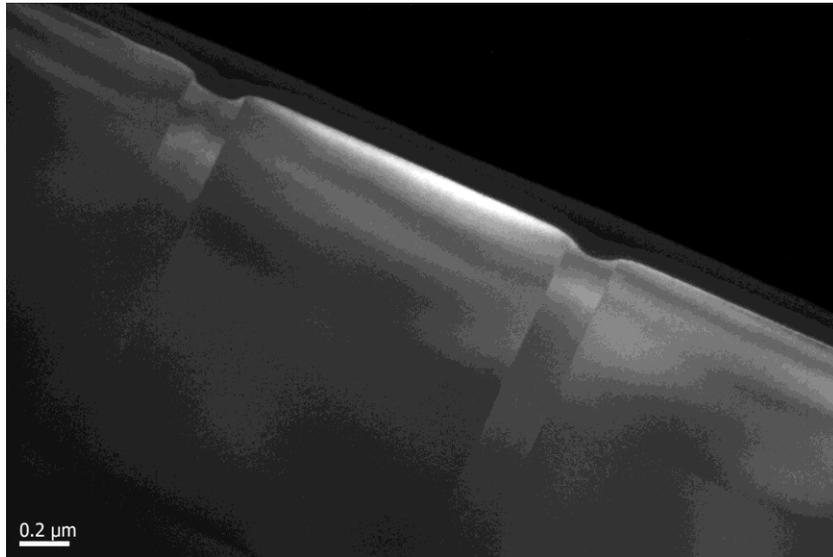


Figure 1. Dark field TEM image of KTP domains. Domains are seen extended along the crystal's polar axis – the crystallographic [001] direction. Biasing was performed along this axis.