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In Situ TEM Probing of Ferroelectric Switching under Electrical Bias

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Ferroelectric materials are useful in a broad range of electronic, optical, and electromechanical applications and hold promise for the design of future high-density nonvolatile memories and multifunctional nanodevices. The utilities of ferroelectrics are derived from electrically switchable polarization states (domains), domain wall characteristic, and the coupling of polarization to other ordering parameters. In recent years, advanced imaging techniques based on *in situ* TEM have become a powerful method to characterize the dynamic process of ferroelectric oxides, allowing the polarization switching process to be resolved in real time. Here, we report some of our recent progress on probing the dynamics of ferroelectrics under electrical stimulus with *in situ* TEM. We have developed a proto-type electrical *in situ* TEM holder with double-tilt functionality and ultra-high stability. As an example for its applications, we show the real-time observation of various ferroelectric switching behaviors, including domain nucleation, domain-wall motion and polarization-defect interaction.

Fig. 1a shows our prototype *in situ* holder, which is designed to be compatible with most advanced JEOL transmission electron microscopes with a pole piece gap of at least 2.2 mm. A schematic of the detailed design inside the holder is shown in Fig. 1b. The ultra-high stability and multifunctionality are realized by a unique design of seal-bearing components, which provide superior vibration damping and electrical insulation while still maintaining excellent vacuum sealing and small form factor. In addition, a wide variety of *in situ* TEM applications including electrical measurement, scanning tunneling microscopy (STM) mapping, photovoltaic studies, and cathodoluminescence (CL) spectroscopy can be performed on this platform with high spatial resolution imaging and ultrahigh electrical sensitivity at the pA scale. An example of applying the probe-type *in situ* holder to exploring the effects of nanoscale impurity defects on the polarization switching is shown in Fig. 2. Epitaxial tetragonal-like $BiFeO_3$ (BFO) films were grown on $TbScO_3$ substrates by molecular-beam epitaxy with the insertion of a $La_{0.7}Sr_{0.3}MnO_3$ (LSMO) bottom electrode. Arrays of planar defects with a length of 5 to 30 nm are observed just above the BFO/LSMO interface (Fig. 2a). A HAADF image of a region containing a planar defect in the $BiFeO_3$ matrix is shown in Fig. 2b, with the overlaid polarization vectors mapped out depicting a head-to-head polarization configuration. A plot of the area of the switched domain as a function of time and applied voltage measured by *in situ* TEM, in Fig. 2c, shows that although a large domain can be written with an applied voltage, the created domain shrank back to a much smaller metastable size after the voltage was removed. All these results indicate the existence of strong built-in fields pointing to the defects, which can dramatically affect the polarization structure and domain stability in the film.

In conclusion, we have developed a probe-type *in situ* TEM holder with a unique design of seal-bearing components that realizes ultra-high stability and novel multifunctionality (including double tilting). Using the probe-type holder, we studied a wide range of complex switching phenomena. The results pave the roads not only for understanding the microscopic mechanisms underlying the polarization switching dynamics but also for guiding the development of practical ferroelectric-based devices [2].

References:
[1] Li, L.Z. *et al,* Nano Lett. **17** (2017), p. 3556.
[2] The author gratefully acknowledges the financial support by the DOE under grant DE-SC0014430.

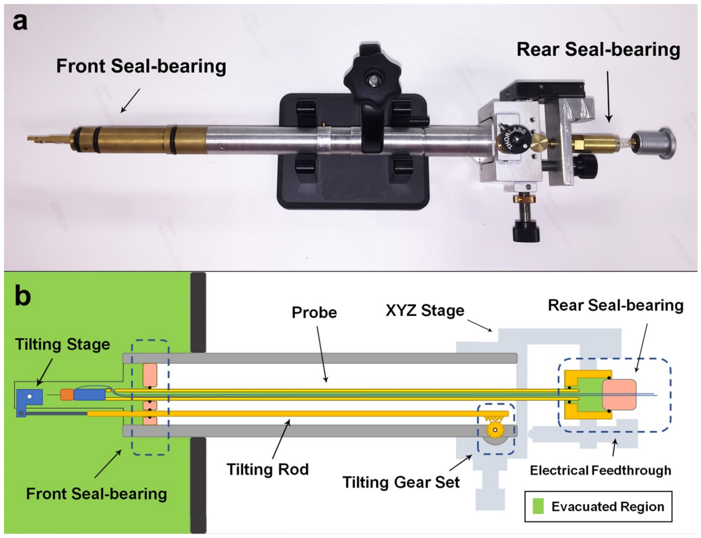

Figure 1. Photograph and schematic showing the design of double-tilt *in situ* TEM holder. **(a)** Side view photograph of the in-situ TEM holder. **(b)** Side-view schematic of detailed holder design. The evacuated area is marked by green color.

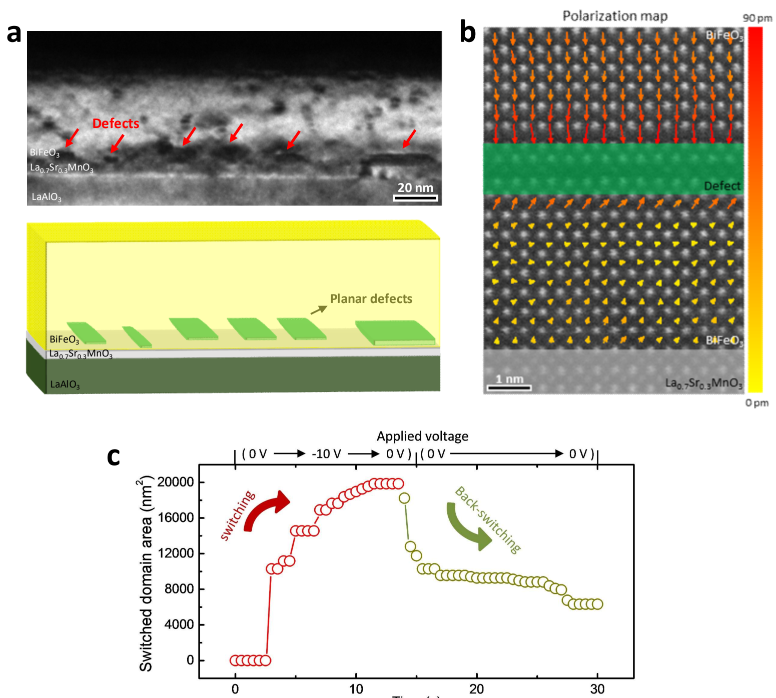

Figure 2. Polarization-defect interaction. **(a)** Cross-sectional dark-field TEM image showing an array of planar defects just above the $BiFeO_3/La_{0.7}Sr_{0.3}MnO_3$ interface, and the corresponding schematic of the heterostructure. **(b)** Polarization vectors overlaid on a HAADF STEM image, in which one planar defect is located above the $BiFeO_3/La_{0.7}Sr_{0.3}MnO_3$ interface. **(c)** Plot of the area of the switched domain as a function of time (bottom axis) and applied voltage (top axis) measured by *in situ* TEM.

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