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Novel TEM Specimen Preparation Using Multi-Source Focused Ion Beams for Real-Time Electrostatic Biasing Studies

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As Transmission Electron Microscopy (TEM) technology improves, and cutting-edge, *in-situ* experiments are developed, the need for ultra-pristine, extremely high-quality TEM specimens continues to grow. One such novel and rapidly growing field within the community involves electrostatic biasing experiments performed real-time in the TEM. Two main approaches have been taken to accomplish this; one involves conventional TEM specimen preparation with a conductive probe tip being brought into contact with the surface, another requires a FIB-fabricated Microelectromechanical (MEMs) device (see Figure 1). We have applied both techniques to investigate the microscopic origins of enhanced strain-driven magnetoelectric coupling (i.e. voltage control of magnetism) in thin magnetic layers (Ni-Fe and FeCoB alloy layers) grown on ferroelectric PMN-PT ($[Pb(Mg_{1/3}Nb_{2/3})O_3]$-$[PbTiO_3]$) substrates[1].

A comparison of the conventional method with the FIB technique shows that the Ga source used in the FIB procedure introduces artifacts in the specimen (see Figure 2), similar to those observed in earlier studies [2]. A subsequent Ar clean-up operation to remove the Ga artifacts is typically used, but the MEMs device eliminated this procedure as an option due to the orientation of the specimen when it is placed on the device (see Figure 1). Therefore we developed a new improved method for removing Ga artifacts which is conducted in a multi-source FIB, through the use of various noble gas species during the final in-situ ion polishing step (which can be applied to a diverse range of applications). In this study, we summarize our efforts to achieve ultra-pristine electron transparent specimens, with little or no image artifacts due to defects or contamination, through the utilization of an advanced multi-source focused ion beam technique.

Piezo Force Microscopy (PFM) was used for comparison (see Figure 3) since it requires no specimen preparation, and therefore is the purest (artifact-free) representation of the material characteristics. The main criteria for comparison are: threshold electric field required to trigger domain switching, reproducibility in the switching characteristics under cyclic biasing, and the onset of polarization fatigue. Examples will be presented describing the effects of specimen preparation techniques on real-time electrostatic poling studies. [3]

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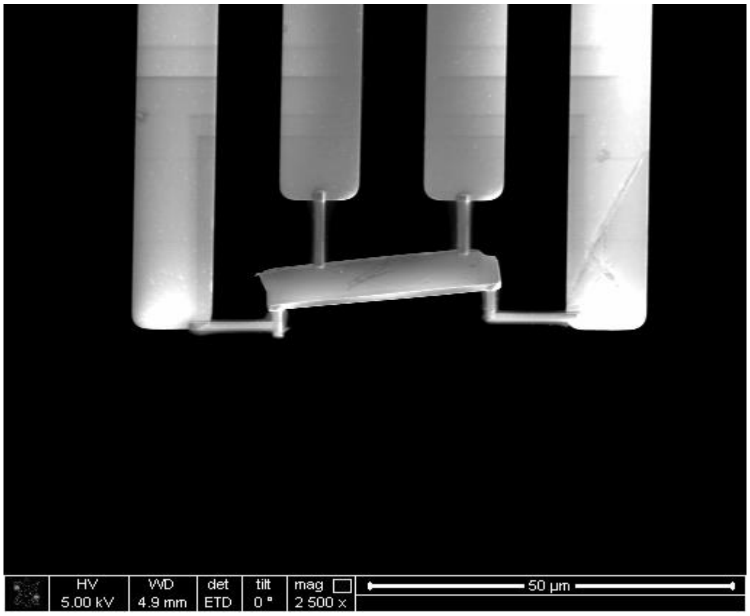
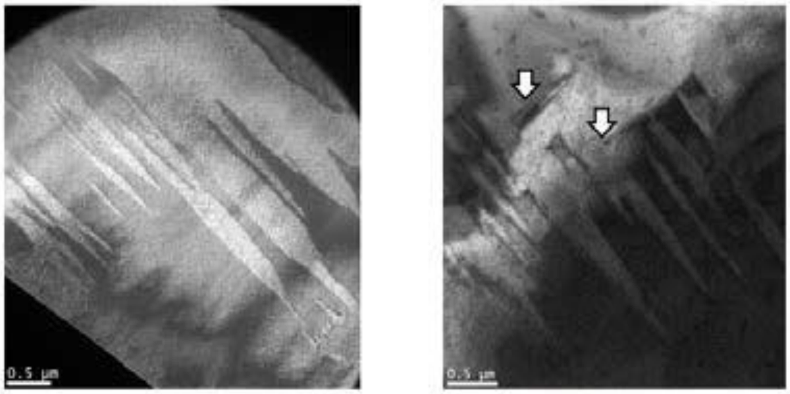


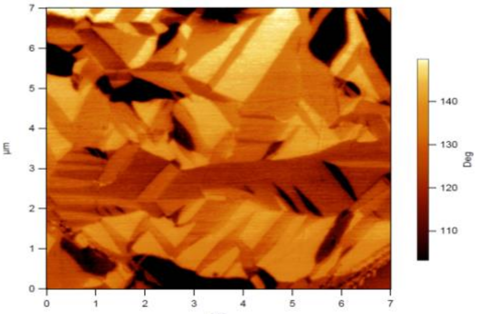
Figure 1. TEM specimen attached to MEMs device.



Conventional Ion Mill

Focused Ion Beam

Figure 2. TEM images of the domain structure in PMN-PT prepared by Argon ion-milling (left) and focused ion beam milling (right). The arrows in the image on right indicate artifacts due to FIB milling.



PFM of Unaltered PMN-PT domains

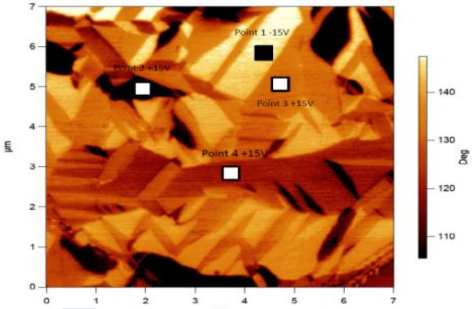
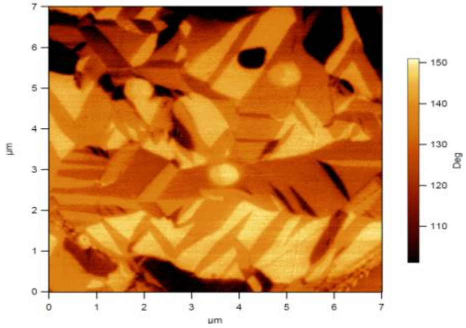


Illustration of where domains will be Electrically Biased



PFM of PMN-PT after Electrically Biased

Figure 3. PFM of PMN-PT domains before and after electrical biasing.

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