ESRF	**Experiment title: Proposal Title Structural characterization of barium titanate/manganite multiferroic heterostructures**	**Experiment number:** **25-02- 664**
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Report:

We studied two samples corresponding to layers of LCMO deposited by magnetron sputtering on BTO (001) substrates (dimensions: 5 x 5 x 1 mm^3). On each sample, the thickness of the manganite layer was determined by reflectometry yielding, repectively, 245 and 120 Å.

Our study aimed at the structural characterisation at room temperature of the epitaxial manganite layer when the ferroelectric BTO substrate is actuated electrically. For this purpose, the samples were mounted on the goniometer using a special sample holder which allowed good electrical contact between a silver paint electrode placed at the bottom of the BTO substrate and the top manganite conductive layer. Electrical contact to the manganite was ensured by four silver paint electrodes placed at the top corners of the samples, so as to maximise the accesible angular range on the goniometer.

For grazing incidence in-plane x-ray diffraction mesaurements, an orientation matrix for each sample was determined on the basis of the stronger reflections from the BTO substrate, which were confirmed to arise from ferroelectric domains aligned with the polarisation vector parallel to the c crystallographic axis. Measurements were taken near the folloing reciprocal space points: (102) for BTO and LCMO (102); (1.5 -0.5 0.5), (1.5 -0.5 1.5) and (1 0 0.5) for LCMO. DC electric fields were applied raging from 0 to 300 V (and subsequently decreasing values down to -300 V and back to 0 V in order to complete a full ferrolectric hysteresis cycle). Diffraction scans for the above mentioned reflections were continually collected. Near the coercive field (which for our samples correponds to ±80 V on each branch) we monitored sample evolution during a period of 10 hours. Otherwise data collection for each applied electric field value corresponded typically to a 45 min period.

In order to ensure good magnetic and electrical transport properties for the manganite layers, our samples are grown well above the ferroelectric Curie temperature for barium titanate and then slowly cooled to room temperature in an oxidising atmosphere. This results in different ferroelectric domain patterns which evolve under the applied electric field. Structural changes in the BTO substrate are evident in Figure 1 as well as their manganite counterpart (Figure 2), with strains along the c direction smaller than the domain switching limit (1 %). Finally, Figure 3 illustrates the evolution of the managanite layer throughout the ferroelectric cycle.

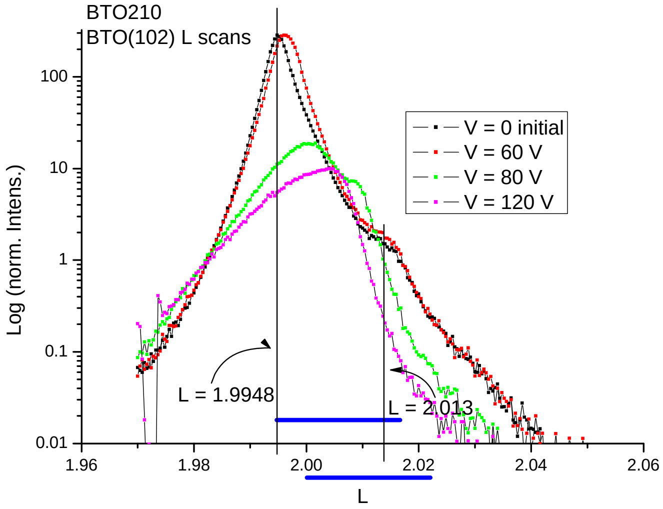

Figure 1. Evolution of the thin sample with applied electric field along the L direction. The vertical lines indicate the presence of different ferroelectric domains. Strong lineshape modification demonstrates changes brought about in the electromechanical balance.

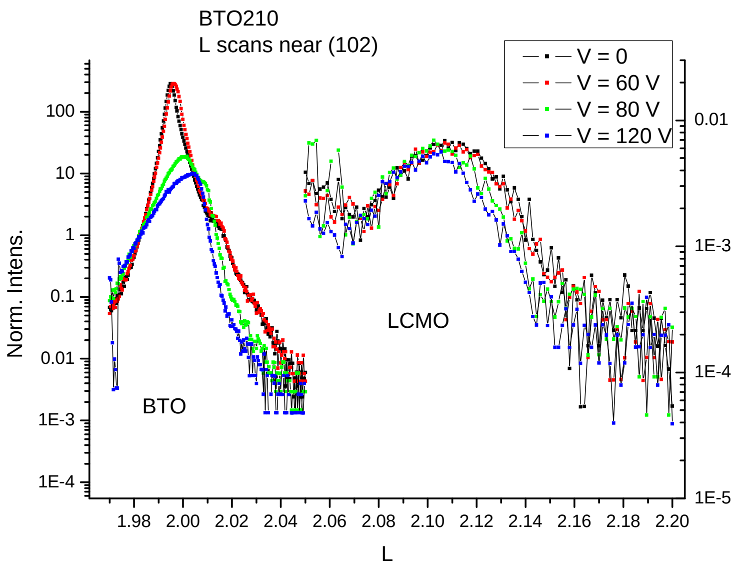

Figure 2. Effect (along the L direction) on the thin LCMO layer of the structural changes induced by the electric field on the BTO substrate.

Figure 3. Evolution with field and time of the thin layer (0, -80 V, -80 V after 10 hours, from left to right) followed through the LCMO (1 0 0.5) (left and center) and (1.5 -0.5 0.5) (right) reflections.