ESRF	**Experiment title:** Bragg Ptychographic imaging of ferroelectric domains in perovskite thin films	**Experiment number**: HC-2004
Beamline: ID01	**Date of experiment**: from: 22 September 2015 to: 28 September 2015	**Date of report**: 22 February 2016
Shifts: 18	**Local contact(s)**: Dr. Steven Leake	*Received at ESRF:*

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Report:

Ferroelectric thin films demonstrate various domain structures, in the range of a few to a few hundred nanometres in size, depending on their composition, thickness and the substrate upon which they reside [1]. Extremely dense and highly inhomogeneous domain structures can be engineered in ultrathin films [2], which is especially interesting in the light of the recent discoveries of unusual functionalities at ferroelectric domain walls [3]. Superlattices consisting of alternating ultrathin ferroelectric and dielectric layers allow further tailoring of the domain structures by controlling the electrostatic coupling between the individual layers [4] bringing promise of materials with enhanced functional properties through domain and domain-wall engineering. To date, however, little is known about the 3D structure of ferroelectric nanodomains and it has thus become vital to deduce the structure of such domains in the out of plane direction. The simplest component, a thin film, is the first step towards the imaging of more complex heterostructures. We applied to the ID01beamline to image the ferroelectric and ferroelastic domain structure of perovskite thin films using Bragg Projection Ptychography (BPP) [5].

We chose to use the double crystal monochromator (DCM) detuned to provide the coherent wavefront without higher harmonics. Other optical elements at the beamline were tested. The white beam mirror was found to vibrate too much, beam movements at the end station were too large. The wavefront structure and drift of the multilayer mirror prevented its use in this case. A fluctuation in intensity from the DCM was observed (~7%) and attributed to the cryo cooling circuit. At 8keV 3x10^8 coherent photons/s were in the nanofocused beam (200 micron diameter Fresnel Zone Plate). It was evident the beam was too broad for a ptychographic measurement and a suitable method for efficiently locating the focal spot of the optics was required (now developed and implemented).

Thin films of $PbTiO_3$(PTO) deposited via sputtering on both $DyScO_3$(DSO) and $KTaO_3$(KTO) substrates were investigated. An example of the speckle pattern observed from a 30nm PTO/KTO sample is shown in figure 1. The visibility is >90% suggesting a high degree of coherence in the incident x-ray beam. The number of speckle determines the number of domains illuminated, in this case >200. The coherent beam size was estimated to be of the order 180x350nm (vertical x horizontal) producing a footprint of the order 320x350nm (FWHM).

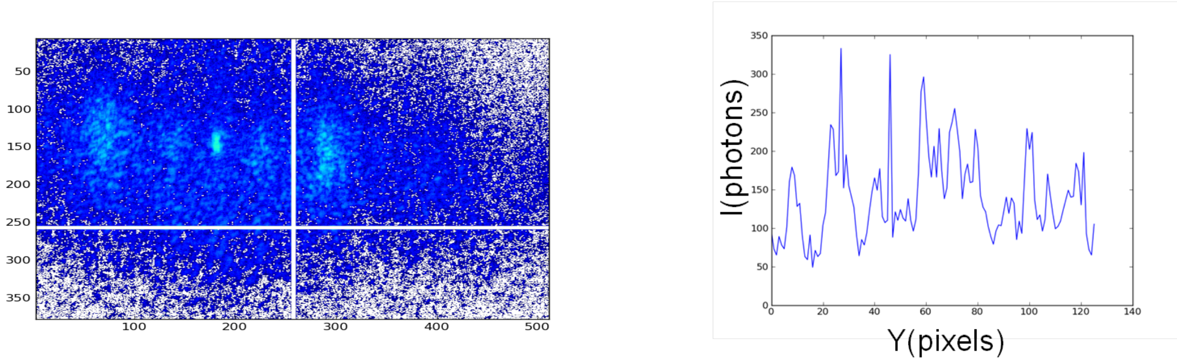

Fig. 1 *(Left) Measured intensity distribution around the* ***003*** *PTO Bragg reflection. 742 photons in the maximum for a 30 second exposure. (Right) Line profile through a single speckled Bragg reflection.*

An example of the a-c domain structure present in PTO/DSO is shown in figure 2. We observe the anticipated a-c domain morphology and through the use of the K-map technique [6] are able to distinguish between regions of differing a-c domain geometry. The K-map method allows us to optimise the height of the sample in the x-ray beam, thus translating the domains of interest to the rotation axis of the diffractometer eliminating associated artefacts.

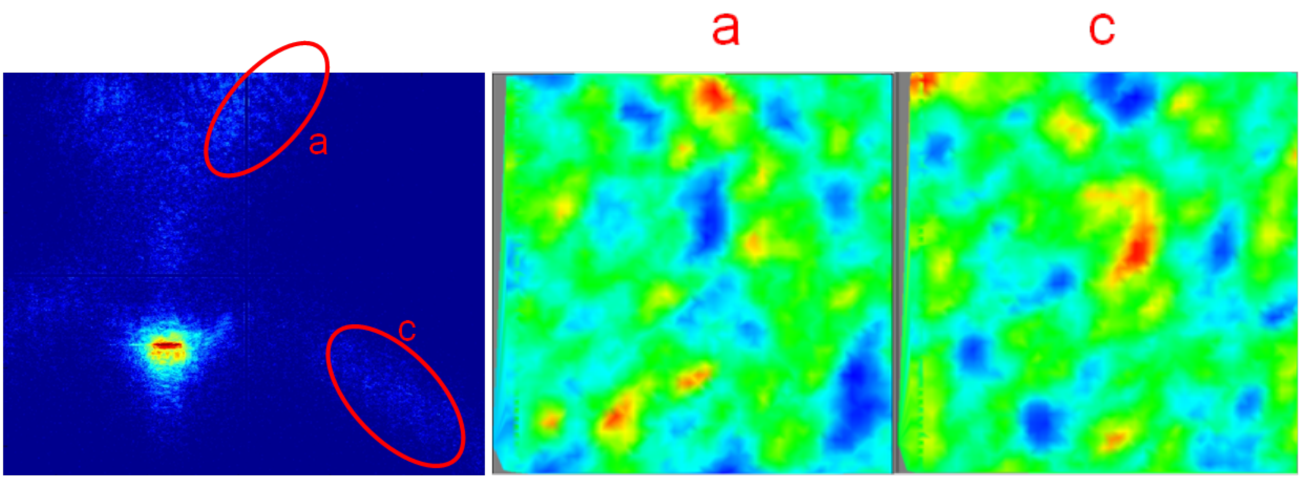

Fig. 2 *(Left) Measured intensity distribution around the* ***002*** *PTO Bragg reflection. (Right) K-maps of the signal attributed to the* ***a*** *and* ***c*** *domains respectively*

Several ptychographic datasets were measured for PTO/DSO thin films with differing thicknesses. As yet reconstructions have proved unsuccessful. We expect the quality of the incident beam is the main cause, however we cannot rule out the impact of stability of the sample relative to the beam.

Finally, several samples were cycled with electric fields, K-maps were used to determine the feasiblity of switching the domains present in the film under x-ray illumination. Both the growth and shrinking of domains was observed as a function of applied electric field. In addition we found the devices to be robust to breakdown when under applied fields for long periods (>12 hours).

References:

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