ESRF	**Experiment title:** Morphology and strain field of the MFP phase of $SrTiO_3$	**Experiment number**: MA-4166
Beamline: ID01	**Date of experiment**: from: 29. August 2018 to: 04. September 2018	**Date of report**: *Received at ESRF:*
Shifts: 18	**Local contact(s)**: Dr. Carsten Richter	

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Report:

The subject of this experiment was a spatially resolved study of the strain fields and polar atomic displacements that occur in strontium titanate SrTiO3 (STO) due to an electric field resulting in strained pseudomorphic layers directly under the electrode. The structure of this migration-induced field-stabilized polar (MFP) phase of STO has already been identified as tetragonally distorted structure with elongation of the cell along the field direction [1]. To refine the structure, the new resonant X-ray diffraction technique Resonantly Suppressed Diffraction (RSD) had been used [2]. The lattice mismatch of cubic STO and the MFP phase results in large stress leading to pronounced inhomogeneities and lattice defects.

We investigated the strain field caused by forming of the MFP phase with fast Scanning X-ray Diffraction Microscopy (SXDM). Different sample geometries have been used to map the morphology of the strain field on the surface and in the depth profile.

We prepared three different kinds of samples to allow for the analysis of different sample geometries:

1. throughout the surface of the transformed, polar layer of a STO single crystal (x, y plane perpendicular to the field),
2. along a crystal face parallel to the field (e.g. x, z plane),
3. throughout the transformed layer between top-top electrodes of varying distances.

None of the crystals have been electro-formed before the experiment.

For geometry 1 we used our standard setup of a 0.5 × 0.5 × 0.1 mm³ sample with the field applied through the top and bottom electrodes. We analyzed the formation process (before, after, and while MFP is present). We measured the reflections 004 and 224 for at a constant energy, see Fig. 1.

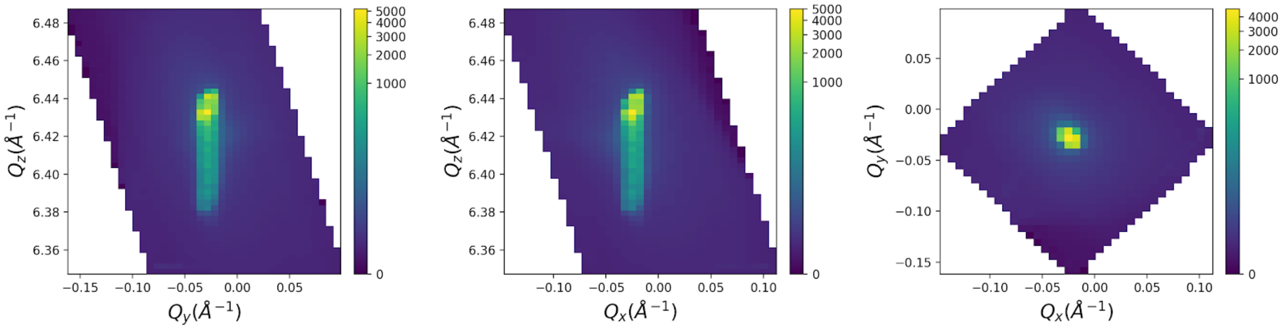
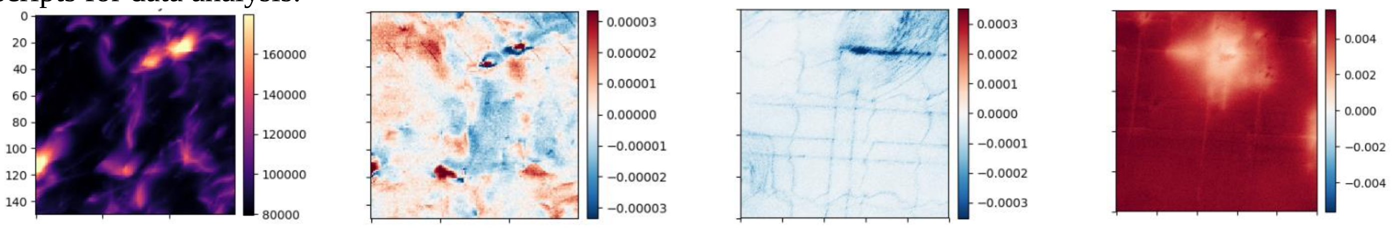


Fig. 1: Sample-averaged reciprocal space maps for the 004 reflection from electroformed STO crystals exhibiting the strained MFP phase in geometry 1.

To microscopically map polar displacements, we scanned reflections 007 and 337 at different energies near the Sr-K edge using RSD in geometry 1. It proved to be challenging to find the same spot on the sample during the different measurements and X-ray visible markers could significantly simplify experiments in the future. Since we need energy-dependent data integrated over 3D reciprocal space, the amount of data to be analyzed is very large. So far it is already sorted, corrected, and converted to h5 format for further processing.
The analysis of non-resonant SXDM data is further progressed. Real and reciprocal space projections are calculated for the reflections 224 and 004 (see Fig. 1 and 2). We are currently improving existing python scripts for data analysis.



substrate intensity | substrate out of plane strain $\varepsilon_\perp$ | substrate strain $\varepsilon_\perp$ after forming | strain $\varepsilon_\perp$ in the MFP phase

Fig. 2: Comparison of real space strain maps before and after forming the MFP phase on STO.

For geometry 2, we needed to ensure that the MFP phase is formed directly below the probed surface. A first idea was to coat the two large surfaces and one side of a STO platelet and subsequently polished the side to break the electrical contact. However, damages due to polishing are strongly visible and would superimpose with the strained areas, a former in-house beamtime revealed.
Instead we prepared a trench with Focused Ion Beam (FIB), on the one hand to create a very smooth surface and on the other hand to allow for measurements deep within the transformed layer, see Fig. 3. In total, we prepared two samples with 2 trenches each. Unfortunately, both samples were short-circuited, probably due to gallium contamination during the FIB treatment. We intend to do EDX analysis of the sides and the trench to find the reason for the short circuit. Furthermore, we need to find a possibility to remove the gallium contamination, as well as to improve the sample preparation.

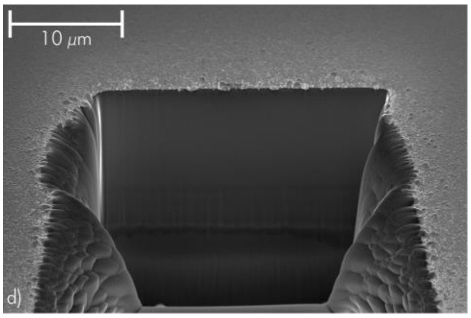


Fig. 3: FIB prepared trench in STO sample.

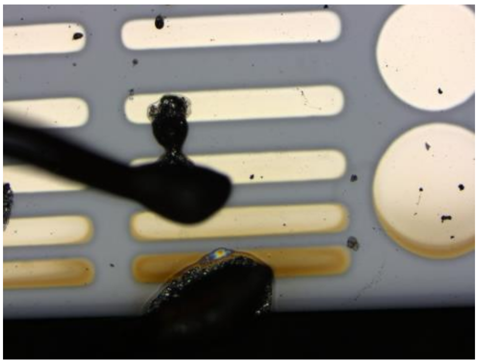


Fig. 4: Contacted top-top contacts.

Geometry 3 demands electrodes with very clear edges to allow for a homogeneous electric field. Additionally, the distance between the contacts needs to be in the order of 0.1 mm to apply the needed DC bias of approx. 1 MV/m with the available current of max. 1 kV. The application of the contacts proved to be challenging, as it has to be ensured that, no short-circuits are created and the conductive silver may not destroy the sharp edges of the electrodes, see Fig. 4. We intended to analyze the MFP phase in the vicinity of the anode. However, the MFP phase is not visible here. An explanation might be that in this top-top contact case, the electric field enters the sample perpendicular to the electrode face and then changes direction parallel to the sample surface. The MFP phase follows this trend and is thus distorted. With this contact geometry the depth profile of the phase may be analyzed.
We were able to complete the challenging measurements of the top-bottom contacted samples, which certainly allows the publication of the data. Thereby, we had to use time that was originally scheduled for the top-top contacted samples. These data could be complemented by an in-house beamtime, however the results are not yet investigated in detail.

References
[1] J. Hanzig et al., Phys. Rev. B 88 2 024104 (2013) 10.1103/PhysRevB.88.024104
[2] C. Richter et al., Nature Commun. 9 1 178 (2018) 10.1038/s41467-017-02599-6