Session VII

Wednesday, September 7 - morning

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FOLLOWING MACROSCOPIC, MESOSCOPIC AND ATOMIC MOTIONS IN MULTI-DOMAIN CRYSTALS UNDER ALTERNATING ELECTRIC FIELD

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Physical property is a response of a material to an external perturbation, as e.g. converse piezoelectricity accounts for mechanical deformation in response to an electric field. Being observable and applicable in devices on a macroscopic level, physical properties are underpinned by some deeper structural or / and microstructural motions. Importantly, many functional properties (e.g. giant piezoelectricity, super-elasticity and shape-memory effect) are attributed to the mesoscopic length scale, where external electric field or stress move domain walls and change the volume ratios between domains. Mesoscopic length scale and multi-domain structure plays pivotal role in ferroic (e.g. ferroelastic and ferroelectric) crystals, where domains naturally appear after a phase transition from higher- and lower-symmetry phases. Surprisingly, the tremendous potential of mesoscopically dominated properties in devices remains unexplored as long as simultaneous probing of macroscopic, mesoscopic and atomic dynamics in multi-domain crystals remains to be a serious challenge.

In this talk we explore the capabilities of the state-of-the-art high-resolution X-ray diffraction for combined in-situ probes of multi-domain processes in ferroics under alternating and quasi-static electric fields. We will focus on the recent insights to the functional ferroelectric and ferroelastic materials and show how high-resolution reciprocal space scans / 2D maps / 3D volumes can be measured simultaneously with e.g. macroscopic *P-E* hysteresis loops. We will discuss the separation of intrinsic response (displacement of atoms in a unit cell [1], changes of lattice parameters [2]) and extrinsic responses (field-induced domain-wall motion, volumetric exchange between domains) as well as their possible interconnection.

We will demonstrate and discuss *in-situ* X-ray diffraction data (the example is in the Figure 1) from uniaxial $Sr_{0.5}Ba_{0.5}Nb_2O_6$ ferroelectric, where only 180° (inversion) domains are present [2] and from the perovskite-based $PbZr_{1-x}Ti_xO_3$ and $Na_{0.5}Bi_{0.5}TiO_3$, where tetragonal, rhombohedral and monoclinic strain domains are present.

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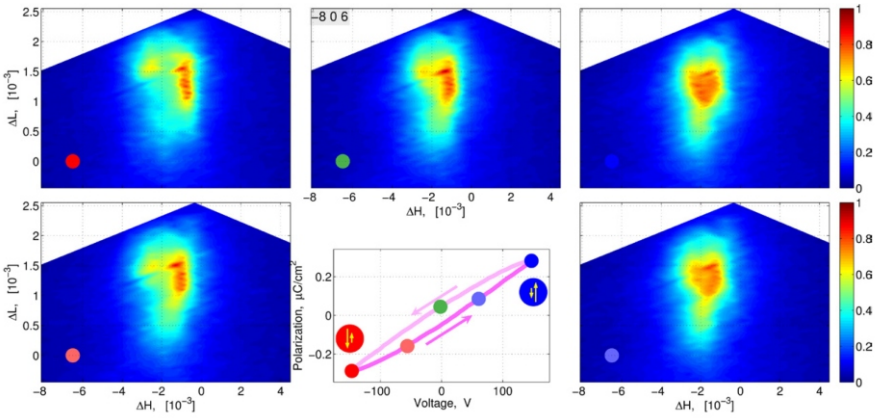

Figure 1. Reciprocal space maps around -8 0 6 Bragg reflections of uniaxial $Sr_{0.5}Ba_{0.5}Nb_2O_6$ ferroelectric, stroboscopically collected under alternating external electric field (P08 beamline at PETRA III). X-ray diffraction was measured simultaneously with the polarization-electric field hysteresis loop (bottom middle). The maps are composed of two Bragg peak components which are displaced against one another due to the opposite (positive / negative) piezoelectric responses in 180° / inversion ferroelectric domains.

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TWIN DOMAIN MAPPING IN TOPOLOGICAL INSULATOR Bi_2X_3 (X = Se,Te) BY SCANNING XRD AND ELECTRON BACKSCATTERING DIFFRACTIONG

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3D topological insulators are a new kind of matter with inverted bulk band gap and Dirac cone-like surface states [1]. Bi_2X_3 with X = Se and Te are prime members of this material class and were shown to exhibit the predicted topological properties [2]. For electrical devices made from these materials large area high quality thin films are required which, however, commonly show the formation of twin defects as can be seen in Fig.1a. Horizontal (c-plane) twin defects were shown to influence the electronic properties [3] whereas little is known about vertical twin defects. We have investigated the horizontal and vertical twin defect formation in molecular beam epitaxy grown Bi2Se3 and Bi_2Te_3 thin films by scanning X-ray diffraction (SXRD) [4] and electron backscatter diffraction (EBSD). With EBSD we directly obtain the crystal orientation in the vicinity of the surface as shown in Fig. 1b. Scanning X-ray diffraction probes the bulk of the thin films and thus complements the surface sensitive electron imaging techniques. For SXRD a focused X-ray beam (~150nm diameter) is used and with the samples mounted on piezo-scanners the XRD intensity is mapped in real space. Performing measurements at the asymmetric (10-1.20) Bragg peak the XRD intensity (Fig. 1c) therefore reveals that defects separating the two twin domains are not strictly vertical but that one twin domain might also overgrow another second one. Based on these results we are able to present a strategy to reduce the surface density of such defects which has important implications for the study of topological surface states.

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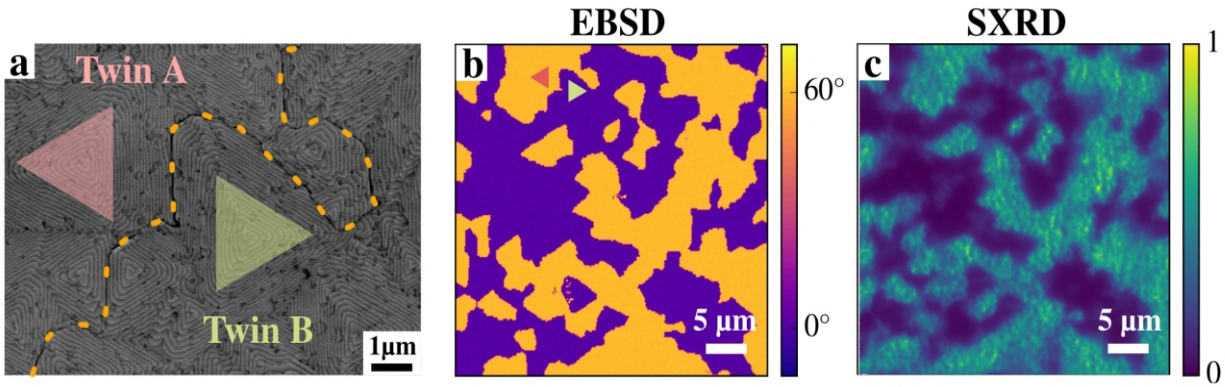

Figure 1. Twin formation in Bi_2Te_3 thin films. a) Scanning electron micrograph of a twin boundary (dashed line) separating two crystallographically twinned areas. b) Electron backscatter diffraction (EBSD) inplane orientation map showing the twin domains in a larger area. c) bulk sensitive scanning XRD measurement of the very same area shown in b), however, with bulk sensitivity.

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IN SITU NANO-MECHANICAL TESTS IN THE LIGHT OF SYNCHROTRON X-RAY DIFFRACTION

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In the recent past, low-dimensional materials attracted enormous attention due to the extraordinary properties compared to their bulk counterparts. For instance, micro-and nano-mechanical tests revealed an increasing yield strength with decreasing structure size reaching the ultimate limit of the material for nanowires [1, 2]. To shed additional light on the mechanical behavior of low-dimensional materials, *in situ* experimental setups are being designed for monitoring the evolution of the structures during the mechanical deformation. So far, in situ mechanical tests coupled with X-ray diffraction techniques concentrated on micrometric samples [3, 4]. For in situ nano-mechanical tests, a scanning force microscope was developed which can be installed at different 3rd generation synchrotron beamlines [5, 6, 7]. Here, we will present the coupling of this new tool with Bragg coherent X-ray diffraction imaging (BCDI) and µLaue diffraction for *in situ* nano-indentation on Au nanocrystals and *in situ* three-points bending tests on self-suspended Au nanowires, respectively [7, 8]. These *in situ* experiments enabled us for the first time to image by BCDI a prismatic loop in a Au crystal which had been induced by nano-indentation. A scanning electron micrograph and the reconstructed electron density of the indented Au nanocrystal are presented in Fig. 1(a). Moreover, the in situ coupling with µLaue diffraction allowed for measuring the complete profile of a mechanically loaded nanowire giving access to the elastic as well as the plastic deformation of the nanostructure. Figure 1(b) displays integrated diffraction patterns of the Au 222 Laue spot recorded along the nanowire at different deformation stages employing a newly developed KB scanning method [8]. From these integrated diffraction patterns, the bending angle and the complete nanowire profile also shown in this figure were determined.

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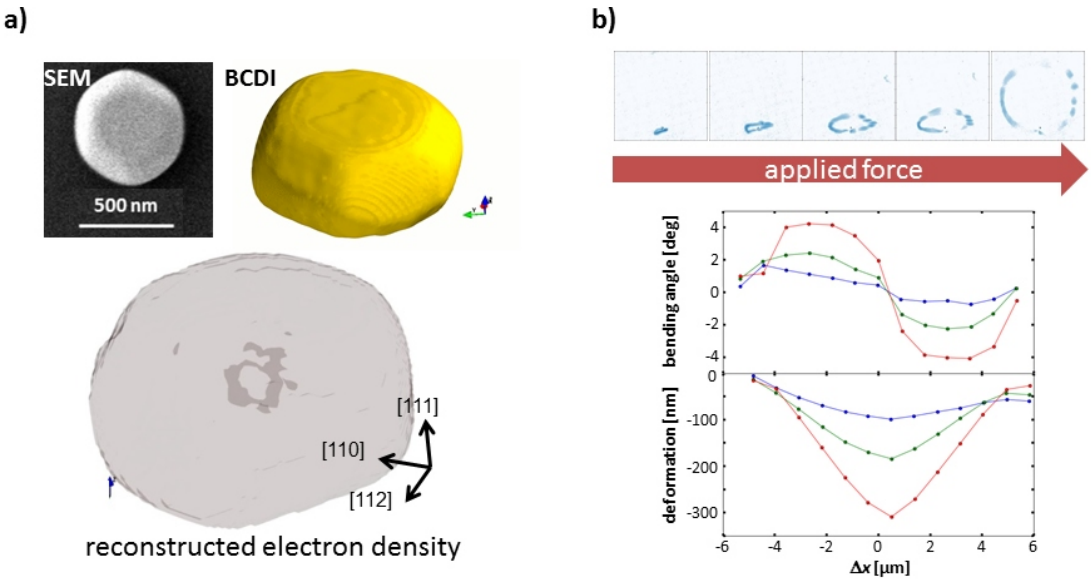

Figure 1. a) SEM image, reconstructed shape and electron density of an indented Au nanocrystal. b) Integrated diffraction patterns of the Au 222 Laue spot recorded along the deformed nanowire, bending angle and nanowire profile inferred from the diffraction patterns.

C22

OBSERVATION OF SAGITTAL X-RAY DIFFRACTION OF SURFACE ACOUSTIC WAVES IN BRAGG GEOMETRY

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X-ray diffraction on surface acoustic waves (SAW) was previously demonstrated in meridional diffraction geometry in several works [1, 2]. SAW travel on the crystal surface, temporarily creating grating-like structures with amplitude up to one nanometer and near-sinusoidal deformation profile. Using this effect *Tucoulou et al.* demonstrated the feasibility of a high frequency chopper for synchrotron radiation at ESRF [2]. But due to the fact that the velocities of the SAW are typically on the order of 3000 m/s, the time resolution of the device was limited by the value of ~1 μs due to the large travel distance of the SAW pulse through the footprint of the X-ray beam at a small grazing incidence angle. In this work we observed an X-ray beam splitting by SAW in sagittal diffraction geometry. A similar experiment was already done by *Roshchupkin et al.* [3]. The (200) Bragg reflection on a langasite ($La_3Ga_5SiO_{14}$) single crystal was used. SAW with frequencies up to 500 MHz were excited in piezoelectric materials using interdigital transducers deposited on the crystal surface. The experiment was performed in sagittal diffraction geometry (Fig. 1 b) by electronic pulsing of the SAW emission and synchronization with the arrival of the synchrotron x-ray pulses. The achieved time resolution was in the order of 50 ns, and it was smeared by the time that the SAW need to cross the beam footprint. In Fig. 2 it is shown the Bragg reflection without and with SAW. Once the SAW are excited the 0-th order is suppressed and only the m=+/-1 satellites are visible. The observed effect can be used for fast modulation of X-ray beams with the time resolution better of 50 ns, which is faster than most mechanical choppers. The results of the measurement were compared with theoretical calculations using GSolver, a rigorous diffraction grating analysis program.

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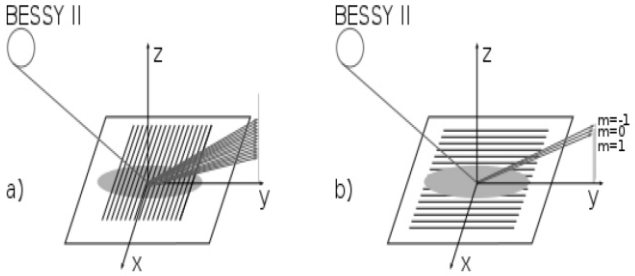

Figure 1. SAW experiment in meridional a) and sagittal b) geometry.

Figure 2. Bragg peak recorded with a CCD b) geometry camera.When SAW are switched on the 0-th order is suppressed and the satellite peaks appear

C23

SUSTAINED kHZ FRAME RATES FOR ULTRAFAST TOMOGRAPHY - INTRODUCING GigaFRoST

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Developing the means to observe a sample's full volumetric structural evolution during dynamic processes with high temporal and spatial resolution has been a key project of the tomographic microscopy beamline TOMCAT [1] at the Swiss Light Source. We have achieved the acquisition of tomographical scans with 3 microns voxel size at 20 Hz [2], and were able to push the time-resolution into the kHz regime for quasi-periodic motions [3].

A major technical innovation enhances the capabilities of our high-speed camera system. Commercially available CMOS detectors able to collect images at multi-kHz rates are designed for burst operation. Images are first buffered in on-board memory, and the subsequent readout process to external storage is slow, thus precluding a sustained data acquisition. The available amount of internal memory limits the total number of frames that can be recorded, and is in many cases much too small to capture the entire duration of dynamic processes at a sufficient temporal resolution and field of view. To overcome this limitation, we have developed the "Gigabit Fast Read-out System for Tomography", coined *GigaFRoST*. The data collected by a pco.Dimax fast imaging sensor are read out by custom-designed readout electronics and directly streamed to the processing servers via eight parallel 10 Gbit fiber-optic links, reaching a transfer rate of 7.7 GB/s, which is sufficient to handle the maximum data rate produced by the chip. On the server side, independent processes can access the data simultaneously to produce real-time previews and write data to permanent disk storage. In parallel, we plan to perform on-the-fly data reconstruction and analysis to select only the useful data for storage or to provide on-line feedback to the experiment. GigaFRoST also offers very flexible trigger schemes, providing an adaptable and versatile interface for complex *in situ, in operando,* and *in vivo* experiments.

We will present an overview of the system architecture and its implementation at TOMCAT, as well as examples of experiments that will greatly profit from the GigaFRoST capabilities, ranging from the observation of crack propagation in metals [2], and self-healing in ceramics [4], to *in vivo* measurements of lung tissue during breathing in mice [5] and the musculoskeletal motion of a fly thorax in flight [3]. The sustained high-speed acquisition has already enabled the detailed observation of sintering dynamics in volcanic materials in real-time for up to 30 minutes.

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