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PERIODIC FERROELECTRIC INVERSION DOMAINS EXAMINED BY COHERENT-BEAM X-RAY IMAGING AND DIFFRACTION

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Periodic arrays of inversion domains, with lateral periods ranging from 5-60 microns, in the ferroelectric nonlinear optical crystals $KTiOPO_4$, $KTiOAsO_4$ and $LiNbO_3$ have been examined using coherent X-ray beams at stations ID19 of the ESRF and 16.3 of the SRS, Daresbury Laboratory.
At ID19, highly coherent x-ray beams have been used to examine the domains via 'Bragg-Fresnel' imaging, in which the phase shift between structure factor amplitudes in adjacent domains is propagated to produce Fresnel diffraction patterns periodic both laterally and with distance from the crystal (the Talbot effect). Because the images preserve information about the phase 'jump' at the domain walls, unique atomic-level information about the domain-wall structure can be extracted. This is demonstrated for the example of (100) domain walls in KTiOPO4 and KTiOAsO4. Complementary studies using high-resolution diffraction at Daresbury's station 16.3 have also been undertaken. The appearance of beautiful satellite reflections in Bragg reflection is seen for array periods of up to 39 microns. In modeling the satellites, both the lateral coherence length of the x-rays and the inherent perfection of the crystal are of importance. In particular, 5 micron-period $LiNbO_3$, which is rather imperfect, is seen to give rise to an X-ray 'speckle' pattern whereas the diffraction from 9 micron-period $KTiOPO_4$ and 39-micron period $KTiOAsO_4$ can be modeled as more ideal examples of coherent gratings using an optical diffraction model.
The results of the imaging and diffraction experiments are compared and discussed in the context of studies of domain walls.

Keywords: FERROELECTRIC DOMAINS HIGH RESOLUTION DIFFRACTION COHERENT BEAM X-RAY IMAGING

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***IN SITU* STUDY OF THE DISLOCATIONS MOVEMENT THROUGH A GRAIN BOUNDARY IN A Fe-Si BICRYSTAL**

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However frequent during plastic strain of polycrystalline materials, dislocation transmission through a grain boundary (GB) remains poorly understood, even in the simplest case of a slip system common to both grains.
Specimen of $\Sigma = 3$ bicrystals (70.54° rotation around the [110] axis, (1-12) ~/(-112) ~ GB plane) of bcc Fe 4%at Si were prepared with a [152] ~/[15-2] ~ stress axis belonging to the GB plane, to promote slip of [-111] ~/[-11-1] ~ dislocations in the (110) ~/(110) ~ plane. They were observed in situ by reflection X-Ray Topography during straining at the ID 19 beamline of the ESRF. A monochromatic beam was used, and the specimen had to be rocked during exposure to ensure imaging of the whole gauge length, as subgrain orientation may vary up to a degree. Successive images were recorded both using a fixed CCD camera and on X-Ray films rocking with the specimen during exposure. The images are sensitive to local surface elastic strains, and the stress state of the specimen can be deduced from image distortions.
Slip bands from the common primary slip system were created under tension or compression in one grain, and were stopped by the GB, where they accumulated. Transmission was observed ahead of accumulations of several tens to a few hundred dislocations. The apparent difficulty of the transmission was linked to cross slip of dislocations from the slip band, and to the dissociation of each dislocation reaching the GB into three GB dislocations having a 1/6[-111] ~ / 1/6[-11-1] ~ Burgers vector, which may glide within the GB plane.

Keywords: GRAIN BOUNDARY DISLOCATIONS TOPOGRAPHY

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BIOMOLECULAR CRYSTALS: FROM X-RAY DIFFRACTION TOPOGRAPHY TO RECIPROCAL SPACE MAPPING

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The production of high quality bio-molecular crystals remains an open issue with several interesting implications ranging from structural to physical characterization. The basic problem is related to the fact that bio-molecular crystals consist of large unit cells that form a rather flexible medium able to accommodate a certain degree of lattice distortion.
Several techniques from X-ray diffraction to microscopy have been adapted to study systematically the reliable and reproducible growth of bio-molecular crystals. The use of synchrotron-based monochromatic X-ray diffraction topography with triple axis diffractometry and rocking curve measurements to better understand the quality of bio-molecular crystals will be reviewed. These techniques are quite complementary. X-ray diffraction topography images reveal quite frequently several separate volume elements contributing to the intensity, showing reminescent growth sectors and other structural defects. These images, rocking curve measurements (FWHM typically in the range of 50 to 4 mdeg) and reciprocal space mapping seem to indicate that the main deformation mode in bio-molecular crystals is plastic in origin.

Keywords: X-RAY IMAGING, MACROMOLECULAR, 'CRYSTAL QUALITY'

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X-RAY DIFFRACTION IMAGING OF ACOUSTIC WAVE FRONTS IN SINGLE CRYSTALS

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Acoustic wave fronts bring comprehensive information on phonon propagation and scattering in crystals since they reveal the local directions of the energy flow. Coherent X-ray beams provide a unique opportunity to see in one image the acoustic wave fronts and lattice defects, thus visualizing defect-induced scattering processes. This is done by stroboscopic X-ray topography at synchrotron beam lines. In the stroboscopic measurements, the acoustic wave propagating within a crystal is synchronized in time with X-ray bursts arriving from the storage ring to sample position. In such a way, the X-rays provide a “snapshot” of the dynamic deformation field, which actually changes on a ns-range.
In this lecture we will show the stroboscopic X-ray diffraction topographs taken from $LiNbO_3$, Si, and GaAs crystals under high-frequency (up to 0.58 GHz) surface acoustic wave excitation. Measurements were carried out at ID19 beam line of the European Synchrotron Radiation Facility (ESRF). The dynamic deformation contrast in the X-ray images is high enough in order to study phonon scattering by dislocations. The developed technique allows us to visualize phonon propagation and interaction with defects in piezoelectric and non-piezoelectric crystals, which are important parts of modern microelectronics and optoelectronics devices.

Keywords: X-RAY IMAGING, PHONONS, DEFECTS