

**MS17.00.05 AN APPLICATION OF X-RAY TOPOGRAPHY AND DIFFRACTION: PERIODIC INVERSION DOMAINS IN NON-LINEAR OPTICAL MATERIALS.** Z. W. Hu<sup>1,2\*</sup>, P. A. Thomas<sup>1</sup>, <sup>1</sup>Department of Physics, University of Warwick, Coventry, CV4 7AL, UK, <sup>2</sup>Clarendon Laboratory, University of Oxford, Parks Road, Oxford, OXI 3PU, UK

Periodic inversion domains or twins formed in non-linear optical materials by periodic-poling techniques, which are used for generating quasi-phased second harmonic generation, are investigated by synchrotron transmission topography, multiple-crystal topography and diffraction. Using a high-resolution X-ray diffractometer with a triple-bounce analyser attached, the inversion domains in  $\text{KTiOPO}_4$  (KTP) and the inversion domain walls in  $\text{LiNbO}_3$  (LN) have been, respectively, imaged with a high spatial resolution mode, furthermore, diffraction contrast subtly dependent on choice of reflection and position has been revealed. With reciprocal-space mapping, the different broadening effects of the widths of diffraction profiles along  $\Delta q_{\parallel}$  and  $\Delta q_{\perp}$  for KTP and the extended diffuse scattering tails along  $\Delta q_{\parallel}$  for LN have been extracted and shown to relate to the lattice tilting or strain induced in the domain-inversion processing through high angular resolution topography. Synchrotron Laue topography has been carried out and the contrast of the domain walls observed in KTP is suggested to be the result of interbranch scattering caused by the phase-shift between the X-ray wavefields in neighbouring domains. Differences in topographic images of the domain-inverted structures between KTP and LN are discussed. The results of the above artificial inversion domains are compared with those of the naturally occurring inversion domains and the similarities and differences between them are examined in the context of diffraction topography. Finally, the observation of the evolution of periodic inversion domains of KTP with changing temperature is briefly discussed.

\*On leave from the National Laboratory of Solid State Microstructures, Nanjing University, P. R. China

**MS17.00.06 HIGH RESOLUTION DIFFUSE X-RAY SCATTERING STUDY OF POINT DEFECT CLUSTERS IN NATURAL DIAMOND CRYSTALS.** R. R. Ramanan, Krishan Lal, National Physical Laboratory, New Delhi-11012, India

Point defect clusters in natural diamond crystals of type I and type II variety have been investigated by measuring and analysing distribution of diffuse X-ray scattering around 1 1 1 and 2 2 0 reciprocal lattice points. A multicrystal X-ray diffractometer with three plane-silicon crystal monochromators crystals oriented in (+,-,-) configuration which yield a highly collimated and monochromated  $\text{MoK}\alpha_1$  beam was employed in these measurements. Specimen crystals were natural { 111 } diamond platelets. IR absorption measurements were used to identify type I and type II specimens. High resolution X-ray diffractometry and topography has been employed to monitor the crystalline perfection. Also, absolute values of integrated intensities were determined. We had selected specimen crystals having varying degree of crystalline perfection. The experiments were performed in symmetrical Bragg geometry. Half widths of diffraction curves were in the range:  $\sim 20$  arc sec to  $\sim 300$  arc sec. Diffuse X-ray scattering measurements were made around both the reciprocal lattice points in four different directions. The DXS intensities versus  $K^*$  plots show regions with slope of -2 (Huang region) and -4 (Stokes-Wilson region), as also observed in silicon crystals [e.g. Ramanan, Bhagavannarayana and Lal, JCG, 156, 377 (1995)]. An analysis of these results showed the presence of submicron size platelets of interstitial type due nitrogen. Some vacancy clusters have also been observed. Shapes and sizes of clusters have been analysed. Interesting differences in defects structures have been observed between crystals of type I and type II variety. An attempt has been made to co-relate defect details with the gross perfection of these crystals and with nitrogen concentration

**MS17.00.07 LARGE ANGLE CONVERGENT BEAM ELECTRON DIFFRACTION OF GRAIN BOUNDARIES.** J. P. Morniroli, Laboratoire de Métallurgie Physique de l'URA CNRS 234, Université de Lille I, 59655 Villeneuve d'Ascq Cédex, France

The transmitted disc (Bright Field disc) of Large Angle Convergent Beam Electron Diffraction (LACBED) patterns exhibits three main features which are very well-adapted to the analysis of grain boundaries:

- it is composed of many dark lines called Bragg lines or Bragg contours. The lines with large extinction distances are very thin.
- most of the inelastic scattering is removed meaning that the Bragg lines have a very high contrast with respect to the background.
- it displays, at the same time, an image of the illuminated area.

Therefore, LACBED patterns can be used to analyse with great accuracy the crystal orientation with respect to the incident beam. For a grain boundary, they can be used to measure the misorientation. LACBED experiments performed on many types of grain boundaries (twins,  $\Sigma 9$  and  $\Sigma 25$  coincidence boundaries, subgrain boundaries and high misorientation boundaries) present in various types of specimens (alloys, semi-conductors, minerals) have shown that the misorientation can be measured with an accuracy (about  $0.01$  to  $0.05^\circ$ ) better than the one obtained with other conventional techniques (Kikuchi lines, ECP, EBSD..).

In addition, since the LACBED technique also allows the characterization of the Burgers vector of dislocations [1], we have analysed dislocations present in  $\Sigma 3$  and  $\Sigma 9$  silicon coincidence grain boundaries as well as dislocations present in subgrain boundaries of the  $\sigma$  phase in steels. The results obtained are in good agreement with theoretical models [2].

1 D. Cherns and A. R. Preston, J. Electr. Micr. Tech. 13 (1989) 111  
2 J. P. Morniroli and D. Cherns, to appear in Ultramicroscopy

**MS17.00.08 MICROTTEXTURE EVALUATION WITH LAUE DIFFRACTION.** Heidelberg, F.<sup>1</sup>, Wenk, H.R.<sup>2,3</sup>, Chateigner, D.<sup>2,3</sup> and Zontone, F.<sup>1</sup>, <sup>1</sup>European Synchrotron Radiation Facility, B.P. 220, Grenoble, France, <sup>2</sup>Laboratoire de Cristallographie, CNRS, Grenoble, France, <sup>3</sup>Department of Geology and Geophysics, University of California, Berkeley, U.S.A

An fully automated procedure was developed for orientation determination of single crystals from Laue diffraction patterns generated by synchrotron radiation. The diffraction patterns were formed with a highly focused X-ray beam (10-30 micrometer) from a thin slab (100 micrometer) of material in transmission geometry. The fully computerized recognition of zones was achieved by transformation of the Laue pattern into a gnomonic projection (where zones are straight lines) and the subsequent application of the Hough transform for line identification. The orientation at each data point was then determined by comparison of angular relationships between zones with a standard table for the known structure. The fully automated procedure allows for rapid orientation determination of single crystals which is particularly important for microtexture analysis. Similar measurements with electron diffraction in the electron microscope are restricted to thin, relatively undeformed surface layers whereas the Laue patterns contain information about a much larger volume which may be considerably deformed. The advantages of the method are demonstrated with the analysis of the microtexture of an olivine rich peridotite from the upper mantle.