Report on HS-1326

Bragg-Fresnel imaging of new nonlinear optical materials and devices in the $KTiOPO_4$ family.

Beamline: ID19

Local Contact: Jose Baruchel, Petra Pernot

Experimenters: P.A.Thomas, T.S.Lyford, J.Baruchel, P.Cloetens

Time: 12 shifts 14/12/00-09/12/00

During this very recent beamtime, a range of Bragg-Fresnel imaging experiments[1-4] was carried out on crystals of the $KTiOPO_4$ (KTP) family and novel periodically-patterned deep-etched $LiNbO_3$ devices. The range of experiments undertaken is briefly outlined here as an interim report on the work to accompany a proposal (number 1485) in the March 2001 round. We intend to make a full report once all the experiments have been analysed.

The primary aim of this experiment was to gain information to add to the study of $KTiOAsO_4$, which is described in refs. [1], [2] &[4]. In summary, this published work explains how combined Bragg and Fresnel imaging was used to discover how the crystal structures of inversion domains in $KTiOAsO_4$ are matched at the atomic structural level across the walls. The sample investigated was periodically poled with an array of enforced domain walls normal to [100] and a periodicity of 39 microns. We were interested to discover whether the analogue KTP showed the same domain wall matching scheme (preliminary results had previously suggested otherwise) and also, whether domain walls normal to the unconventional wall direction [010], showed the same characteristics or not. A longer term goal was (and is) to investigate the characteristics of domain walls in crystals of the KTP family in general – hence the preliminary experiments on $RbTiOPO_4$ and novel doped Rb:KTP crystals. The deep-etched $LiNbO_3$ was included for interest and to see if these samples would produce a fruitful line of future study.

Samples and Experiments

The experiments commenced with a survey in white beam of the range of samples assembled for study. These were :

(1) etched and un-etched periodically-poled KTP from F.Laurell, Stockholm, which was prepared with a 17 micron period using a surface modification (rubidium in-diffusion) technique, with domain walls normal to [100].
(2) periodically-poled KTP from G.Rosenman, Israel, which was prepared with a 24.7 micron period by the low-temperature poling technique, with domain walls normal to [100].
(3) a KTP sample with a single domain wall written normal to [010], prepared by Rosenman's low-temperature method.
(4) periodically-poled "stoichiometric" KTP, which was prepared with a 24.7 micron period by G.Rosenman at room temperature, with walls normal to [100].
(5) periodically-poled $RbTiOPO_4$, with period 24 microns, poled at room temperature, with walls normal to [100].
(6) naturally twinned Rb-doped KTP's (Rb doping between 0.01 and 0.03 mol %)
(7) planar step-poled KTP from F.Laurell, prepared with domain walls normal to [100] and [010], by the surface modification method.
(8) periodically-poled then deep-etched LiNbO3.

Of the above samples, the initial white-beam survey showed that (1), (3), (4), (5) and (7) could be profitably studied further in the time available. The samples under (2) and (8) were simply too high in defect density for study and the samples under (6), whilst they presented interesting naturally-occurring domain wall configurations, required dedicated study with a monochromatic beam. Accordingly, samples (1), (3), (4), (5) and (7) were investigated in white-beam as previously described[1-4] , with images being taken as a function of crystal to film distance in order to follow the development of combined Bragg and Fresnel images.

A monochromator was then inserted and energy 16.53 keV was selected. The most promising sample, number (4)– periodically-poled stoichiometric KTP, period 24.7 micron, orientation of walls perpendicular [100] – was selected for detailed study. Reflections 040, 142 and 140 were collected as a function of crystal-to-film distance, with this distance varying from 0.1m to 5.4m. At the start of the experiment, we expected the 040 and 140 reflections to show no periodic contrast arising from the walls and only the 142 reflection to show contrast.

Preliminary interpretation

The key piece of information emerging from this study at this early point is that in order to explain all the observed contrasts for the domain wall configurations in the KTP crystals studied, it is necessary to invoke an extra fault or displacement vector to match the structures at the walls. The necessity for this fault vector is revealed by the presence of contrast in reflections like 140 (hk0 with h+k odd) and is consistent with lack of contrast in e.g., the 040 reflection, in which no contrast was indeed observed. The fault vector appears to be 0.5**a**+0.5**b**, and we are presently working on a crystallographic model for why this particular fault arises. This result is consistent with the published work for $KTiOAsO_4$ and represents additional information about the structure of the walls, which was not available from that experiment alone.

References

[1] P.Rejmankova-Pernot, P.A.Thomas, P.Cloetens, F.Lorut, J.Baruchel, Z.W.Hu, P.Urenski & G.Rosenman *Periodically-poled KTA Crystal Investigated using Coherent X-ray Beams* J.Appl.Cryst. **33**(2000), 1149-1153.

[2] **Same** Authors as for [1] *Use of Bragg Diffraction Imaging, with a Coherent Beam, to Determine the Matching of Ferroelectric Domains.* ESRF Highlights (2000), 79-80

[3] P.Rejmankova-Pernot, P.Cloetens, J-P.Guigay, J.Baruchel & P.Moretti Phys.Rev. Lett. (1998) **81**, 3435

[4] **Same** Authors as for [1] (1999) Report on Experiment HS709