![ESRF logo] **ESRF**	**Experiment title:** Coherent X-ray Imaging of Atomic-level Structure and Dynamics of Ferroelectric Domain Walls	**Experiment number**: ME302
Beamline: ID19	**Date of experiment**: from: 9th December 2001 to: 13th December 2001	**Date of report**: 26th February 03
Shifts: 12	**Local contact(s)**: Jose Baruchel, Petra Pernot	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. P.A.Thomas*, *Dept of Physics, University of Warwick, UK*: **Prof. J.Baruchel***, **Dr. P.Pernot***, **Dr. P.Cloetens**, *ESRF, France*: **Dr. R.C.C.Ward, Dr. K.B.Hutton**, *Clarendon Laboratory, University of Oxford, UK*: **Dr. F. Laurell**, *Royal Institute of Technology, Stockholm, Sweden*: **Dr. P.Smith**, *Optoelectronics Research Centre, University of Southampton, UK*

Report:

In-situ poling experiments were continued with new low-conductivity "stoichiometric" $RbTiOAsO_4$ (hereafter referred to as LCRTA) and normal conductivity flux-grown $RbTiOAsO_4$ (RTA), both from Coherent Crystal Associates, and stoichiometric $LiNbO_3$ from Oxide, Japan (hereafter SLN). The poling rig had undergone modification to the electrodes to permit a better entry and exit path for x-rays. Experiments were confined to those at fields not requiring operation *in vacuo* (< 4 kVmm^{-1}). This was insufficient to obtain any poling in the SLN, which will not be referred to further.

Results

(These are very preliminary from beam-time in December 2002).

Very novel and unexpected results were obtained with LCRTA patterned with a periodic electrode of 38 microns. Fig. 1 shows a series of sections for the 07-3 reflection taken as a function of time before and after application of a sequence of 16.5 ms negative-polarity sinusoidal electric field pulses of maximum amplitude −1.91 kV/mm. From hysteresis loop tracing experiments conducted off-line at Warwick, it is known that this field is sufficient to produce partial ferroelectric switching (a hysteresis loop and a finite remanent polarisation are obtained). The exposure times for the sections were 15s and the time intervals between exposures were varied systematically from 7s to 105s. The first section shows the zero field state of the crystal. The second section was taken while the field was being applied. The regions of the crystal to the left and right of the narrow waist on the section show different time dependences (this may be related to their proximity to the high-voltage contact on the surface). Concentrating on the region to the right of the waist, in the image under field (t=0 to t=15s), periodic striations corresponding to the grating are seen to penetrate about half-way across the 0.8 mm thickness of the crystal. By the third image (starting at 22s and ending at 37s), they have penetrated fully across the crystal and the contrast is strong. On the 4th image starting at t=88s, the contrast is fading and by t=417s, the image appears as for zero field. A much quicker propagation and back-switching appears on the left-hand side of the images with contrast completely across the thickness of the crystal during application of the field and most of the backswitching having occurred by the t=89s exposure.

There is clearly a wealth of detail to be extracted from these and the other field and time-dependent images that were taken in December 2002. This preliminary report serves to show <u>proof of concept</u> that ferroelectric domain dynamics can indeed be followed by coherent-beam x-ray imaging. The experiments produced the unexpected observation that processes are on the timescale of minutes in some samples under particular conditions of field and frequency. To our knowledge, our observations are the first direct visualisations of such dynamic domain processes and we are looking forward to extending these unique experiments further.
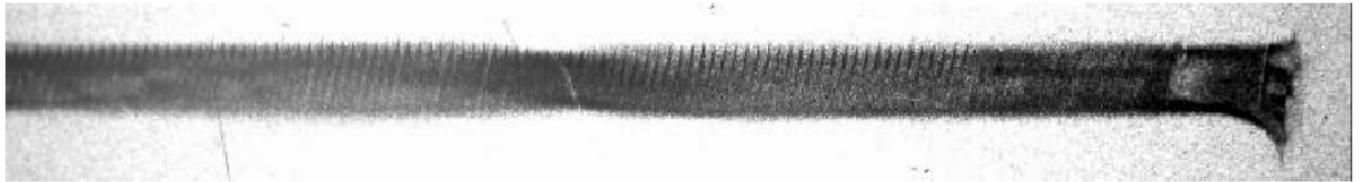
Figure 1

Time Dependent study of LCRTA, Reflection 0 -7 3, Lamda = 0.80882A, ThetaB = 9.66
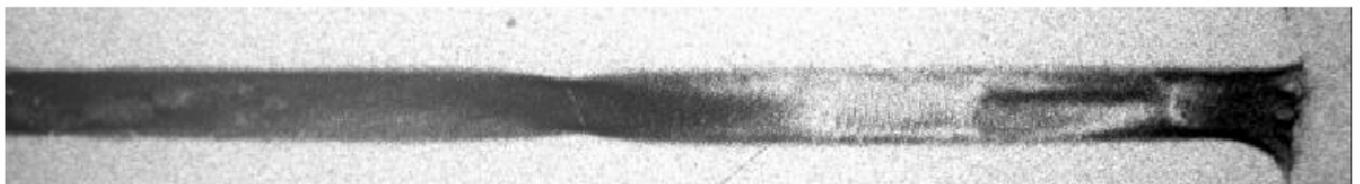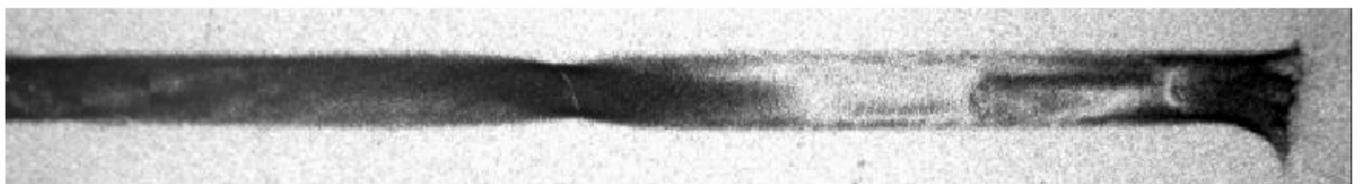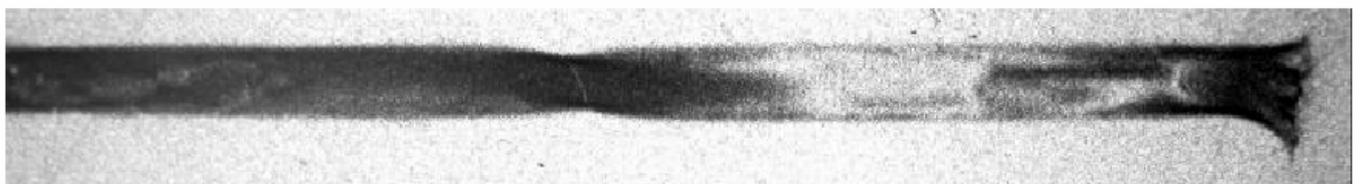Film 02 12 15 07
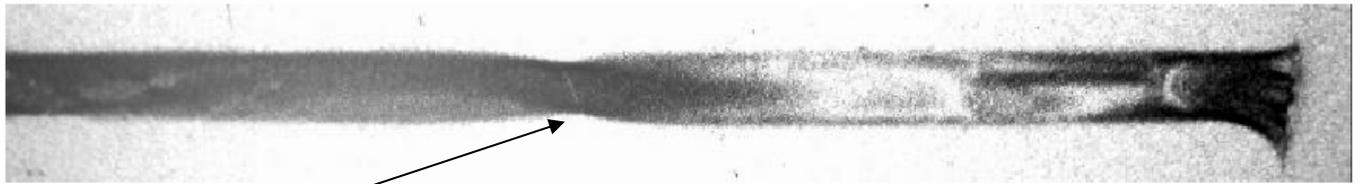
waist

Zero Field

-1.91kV/mm

t = 22secs

t = 89secs

t = 156secs

t = 417secs

t = 925secs