ESRF	**Experiment title:** Micro-focus diffraction and phase-contrast imaging of domain-wall structure in periodically domain-inverted lithium niobate	**Experiment number:** HS-424
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Report

(1) Following upon the successful experiments with A. Snigirev's team at the Optics Beamline, (Experiment HS-112, to appear in *Nature)* a further experiment on periodically domain-inverted (PDI) $LiNbO_3$ (LN) was conducted at the μ-FID Station (ID22) during its commissioning period. The aims of this experiment were three-fold: To test the use of the high-resolution CCD camera for recording phase-contrast images of PDI LN as an alternative to film recording;

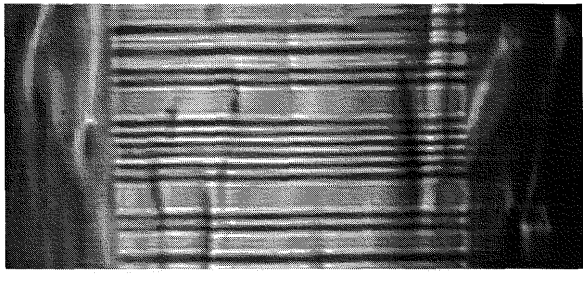
(2) To demonstrate that PDI arrays of periods as short as 4 μm could be successfully imaged with sufficient resolution;

(3) To collect diffraction and imaging data on PDI arrays of period 20 μm and 4 μm with images taken at different points on the 006 and 0012 rocking curves from the diffuse scattering on the low angle side through the maximum to the diffuse scattering on the high-angle side for subsequent interpretation.

All of the three aims were accomplished. It was first of all necessary to demonstrate that the phase-contrast images of PDI 20 μm period $LiNbO_3$ (hereafter, LN20) produced at the Optics Beamline could be reproduced at ID22 using a monochromatic beam of wavelength 1 Å (Si 111 monochromator). Images were recorded as a function of distance from the sample in a range from 5 mm to 50 cm both with high-resolution x-ray film and the high-resolution CCD camera, which was established to be an excellent means of recording. Further to the original experiments on LN20, images were recorded in the diffuse scattering as well as on the maxima of reflections 006 and 0012. The diffuse scattering images of 006 showed interesting outcrops of dislocations and strong contributions from surface defects: for example, the "ghost" image of the masks used during the electroding process for electric-field domain-inversion appeared in these images.

From analysis of the sets of images, which is in progress , it should be possible to reconstruct the effect that different stages of the domain-inversion processing have produced on the microstructure of the resulting sample. Since 006 is a surface-sensitive reflection and 0012 is a bulk-sensitive reflection, the comparison of these two sets of data will be particularly instructive in this respect. It should be noted that both 006 and 0012 reflections gave rise to very similar phase-contrast images in the details and intensity of the interference fringes. Since the images arise from "phase-jumps" at the domain walls, a similar phase jump must be occurring for both the 006 and 0012 reflections, although the effect of anomalous scattering in distinguishing between 006 and 00-6 is much stronger than the difference between 0012 and 00-12. In interpreting the data, a self-consistent explanation for the observations for 006 and 0012 must be found. To illuminate this further, it is proposed that a further set of experiments, which will examine the images as a function of wavelength, should be undertaken.

Data were then collected in a similar manner on an $LiNbO_3$ sample in which a PDI array of period approximately 4 μm (LN4) had been written, thus presenting a more stringent challenge to the x-ray imaging technique. Images were successfully obtained with sufficient resolution using the high-resolution camera and images on film as a function of distance between approx. 5 mm and 25 cm were recorded also. Beyond about 25 cm, the fringe patterns blurred and became indistinct, as expected. Only the 006 reflection was recorded in any detail (see figure). The aim of studying LN4 is to compare the nature of the domain walls and the effect of domain-inversion processing in a large period and a short period array. It is much more difficult to keep the registry of the domains in a short-period array, an observation which we believe to be associated with the strain-fields induced during electric-field processing as well as the increased technological difficulties associated with patterning a short-period array. The images of LN4 were of sufficient resolution to see the greater occurrence of faults in the periodicity of the array and showed the LN4 crystal to be considerably less perfect than LN20, observations which remain to be linked to factors in the domain-inversion processing.



The figure shows a phase-contrast image of LN4 taken at a distance of 12 cm, exposure time 20 s, with the high-resolution CCD camera. The set of strong thick horizontal white lines in the centre of the image correspond to domain walls separated by approximately 2 μm as appropriate to a 4 μm period array. The images of dislocations, and associated faults in the walls, can clearly be seen crossing the array.