HS 2384 experiment report (May 2004)

The experiment HS 2384 was performed at the ID10B (Troika II) beamline. The goal of the experiment was to study X-ray diffraction from a superstructure of regular ferroelectric domains in a single crystal of lithium niobate, which is a key system for the photonic industry. Due to the experimental technique used to obtain the samples – namely the Czochralski off-center growth – it is believed that important structural modifications are present in the neighbourhood of the domain walls. If some experimental conditions are met, it is expected that a number of appropriate Bragg reflections would split in a series of satellite peaks with a spacing determined by the period of the ferroelectric grating. By the analysis of such diffraction spectra it is possible to investigate the structural modifications occurring inside the single repeated unit of the superstructure, which in the present case is constituted by the domain wall.
Among the experimental conditions required to observe the phenomenon, the most stringent are the perfection and the period of the superstructures, which have to be as small as necessary in order to resolve the satellite peaks. On the other hand, the optics and the mechanics of the experimental setup must provide the necessary resolution.
The samples prepared were doped with erbium, which is necessary in order to obtain the desired ferroelectric structures. Several samples with various concentrations ranging from 0.3 to 0.7 %mol. were examined. Due to the limitations of the growth technique, the smaller period which could be obtained with a reasonable perfection was 5 μm. As a consequence, in the best case a separation of 1.26×10^{-3} nm^{-1} is expected between reciprocal lattice satellite points.
A Diamond (111) monochromator was employed with a relative energy resolution of 1.5×10^{-4} at a wavelength of 1.59722 Å. The input divergence of the beam was 2.1×10^{-3} degrees. Moreover we employed a (1 1 1) Si analyser with an angular resolution of 2.3×10^{-3} degrees. The final instrumental angular resolution, as measured with a detector scan, was about 4×10^{-3} degrees. The mechanical movements of the goniometers were found to be repeatable with an accuracy as good as 5×10^{-4} degrees on all the axis used in the experiment. Using those data, the resolution in reciprocal space can be estimated, depending on the particular type of scan to be employed.
We performed two types of measurement. The first was with the scattering plane perpendicular to the sample surface and to the domain walls; the momentum transfer vector lies therefore parallel to the domain walls. The second was in near-grazing incidence geometry, with the scattering plane nearly parallel to the sample surface but with the momentum transfer vector perpendicular to the domain walls. In the first configuration the satellite peaks should be revealed by a transverse scan, while in the second case, the satellite peaks should be detected by a radial scan. For the first type of measurement, in the better case (reflection (0 0 6)) the estimated instrumental resolution was about 2×10^{-3} nm^{-1} so that the effect was probably below the resolution limit of the experimental setup. In fact no superstructure was detected in the diffraction peak.
In the grazing incidence configuration, the instrumental resolution depends very markedly on the grazing angle between the impinging beam and the sample surface. We selected several samples with a low surface miscut, obtaining experimental rocking curves that have a width almost equal to that expected for a perfect sample (plus the resolution width of the instrument , namely 3.4×10^{-3} degrees). Being the period for these samples about 8 μm, the periodicity in reciprocal space is therefore very short (less than 10^{-3} nm^{-1}). Nevertheless, if the satellite peaks were very pronounced they could be observed in spite of the instrumental convolution. In this configuration, the scan could be performed, in addiction to the rocking of the sample, both by moving the detector plus analyser crystal block, or by performing an analyser scan. In the first case we observed a series of modulations with a spacing very near to that expected from the ferroelectric grating (8×10^{-4} nm^{-1}) (see figure 1) which, however, were totally absent in the other scan mode. This fact, together with a series of other tests, lead us to conclude that the modulations were ascribed to low frequency mechanical periodic errors which could be avoided without using the goniometer axis

corresponding to the heavier arm for very resolved scans. As the periodicity of our PPLN samples was somewhat large, the features in reciprocal space were therefore hardly detectable. Considering the importance of the systems studied for the modern photonic industry and the original approach undertaken both on the material and on the particular experimental geometry, we believe that the prosecution of this experiment in better experimental conditions (namely, the periodicities of the samples lowered to at least 2 μm and some upgradings on the experimental set up) is a worth-while effort.

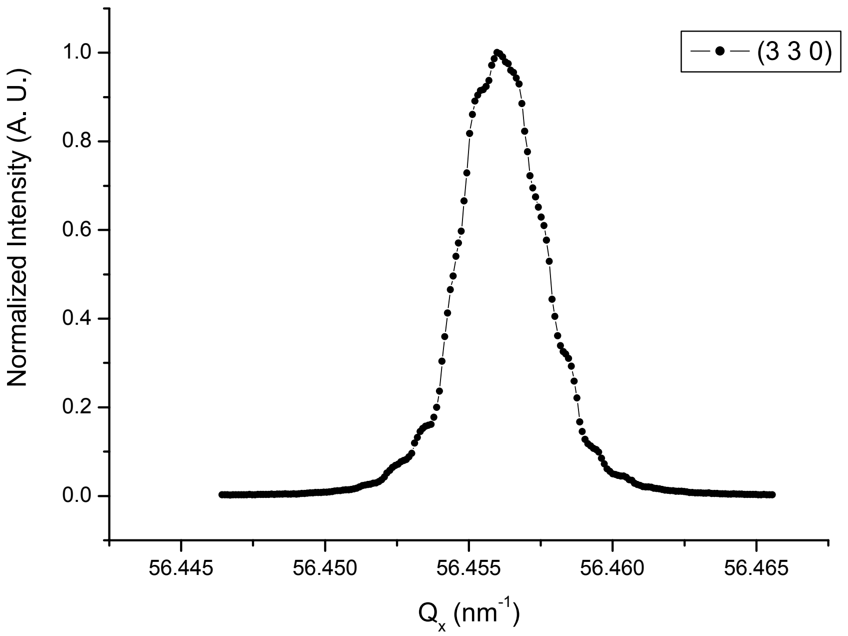

Figure 1: Experimental diffraction peak for reflection (3 3 0) in the Grazing Incidence condition. Intensity modulations are clearly visible.