Report Experiment MA-22

The experiment MA-22 was performer at the ID 10 B "Troika II" beamline in the period from 19 Apr 06 to 25 Apr 06. The aim 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 known as PPLN (Periodically Poled Lithium Niobate). Depending on the experimental technique used to prepare such samples, it is believed that important structural modifications are present and are correlated to the domain structures . If some experimental conditions are met, the Bragg reflections of a PPLN sample split in a series of satellite peaks with a spacing determined by the period of the ferroelectric grating. By the analysis of the experimental reciprocal lattice maps 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 single domains. 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 give a sufficiently spaced satellite structure. On the other hand, the optics and the mechanics of the experimental setup must provide the necessary resolution. In this experiment we explore the possibility of using the high resolution X-ray diffraction technique in reciprocal space mapping mode in order to investigate the structural properties of two PPLN samples prepared with two different techniques.

The first sample was prepared by the off – center Czochralski technique by doping the melt 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 cut from the as grown boules with the main surface oriented along the crystallographic Z-direction. Due to the limitations of the growth technique, the smaller period which could be obtained with a reasonable perfection was about two µm. As a consequence, for a Z-cut sample and the (0 0 12) reflection, a separation of 3-4×10^{-3} nm^{-1} parallel to the sample surface is expected between reciprocal lattice satellite points. This required an adequate reciprocal space resolution of the experimental setup, which was obtained by inserting in the beamline two additional Si (3 3 3) channel-cut crystals as a monochromator and analyser respectively. This resulted in an improved reciprocal space resolution which was estimated in 3.14x10^{-4} nm^{-1} in the lateral Q_x direction and 6.43x10^{-4} nm^{-1} in the vertical Q_z direction. In order to not introduce artifacts due to mechanical vibrations of the heavy detector arm of the goniometer, the reciprocal space maps were performed rocking the sample and the analyzer crystal in a synchronized way. To this purpose several macros were realized using the SPEC program. All the measurements were performed with the incoming beam σ–polarized i.e. with the polarization plane perpendicular to the scattering plane. The sample was aligned so that the periodic domain structure wavevector lies inside

the scattering plane; in other words, the domain walls were perpendicular to the scattering plane. Two reflections were probed: the symmetric (0 0 12) reflection and the asymmetric (2 2 12).

The second sample we measured was a PPLN prepared by the electric field poling technique. In this case the electrodes for the poling were realised using a new interferometric photolithographic process and the poling was performed in the overpoling regime. This allowed for the realization of a PPLN with very short period, which nominally was below one micron. Owing to its small periodicity, this sample did not require a very high instrumental resolution and was measured with a standard High Resolution Diffractometer.

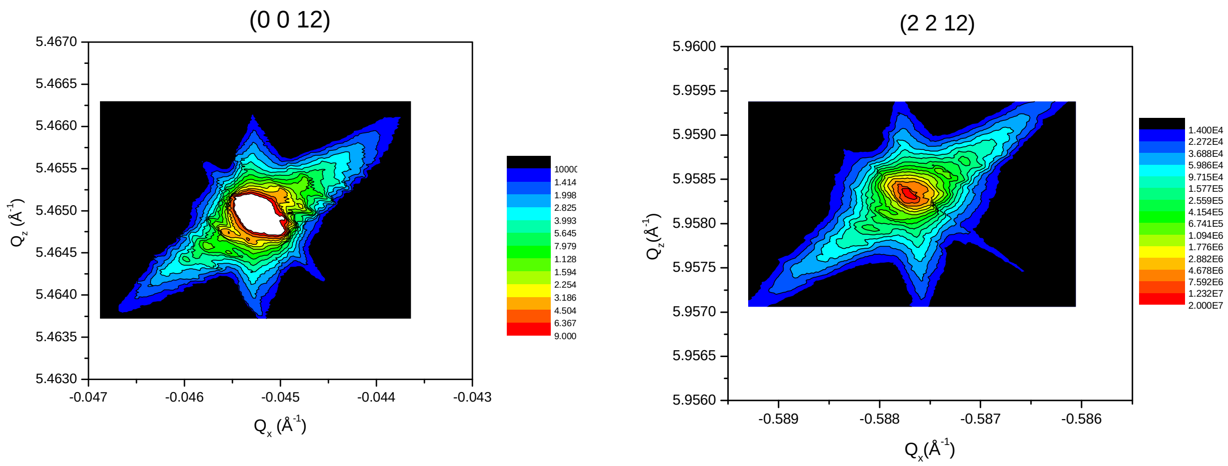

Figure 1: Reciprocal space map of a PPLN sample prepared by Czochralski Off-Center Technique. Reflection (0 0 12) (top) and (2 2 12) (bottom).

In figure 1, two reciprocal space maps for the first sample is reported. The satellite peaks due to the periodic domain structure are visible, even if their contrast is low. Superimposed to the satellite grating, a pronounced diffuse scattering streak is visible. Both the satellite structure and the diffuse scattering streak are inclined with respect to the Q_z direction of an angle given by the angle between the domain walls and the crystallographic Z direction. This feature is due to the fact that in this samples, due to the particular growth technique, the domain walls are not perpendicular to the sample surface, but are tilted of an angle given by the inclination of the satellite structure with the Q_z direction.

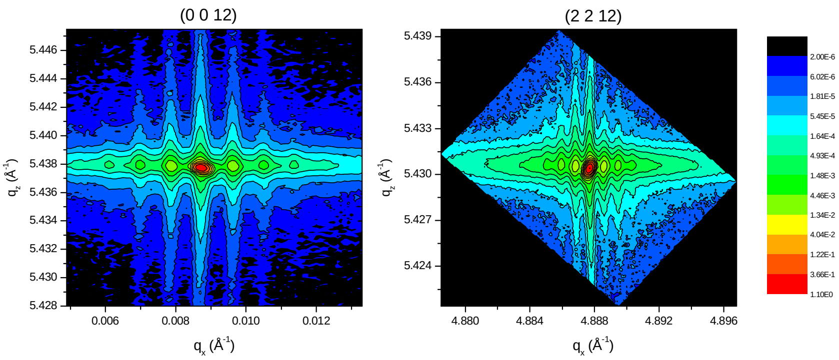

Figure 2: Reciprocal space maps of a sub-micrometric sample prepared by the electric field poling technique.

In figure 2 are reported two experimental maps of the second sample prepared by the electric field poling technique, measured with a standard Philips MRD Diffractometer. Also in this case the periodic structure is visible, as well as the diffuse scattering streak. In this case the domain walls are perpendicular to the surface and as a consequence the domain structure and the diffuse scattering streak are perpendicular to the Q_z direction. Moreover in this case a series of vertical streaks associated to the satellite structure are present, which can be ascribed to the presence of strains inside the domain structure. It should be noted that this feature is not present in the first sample which was prepared at high temperature, directly during growth. It is also remarkable the presence in both sample of the diffused scattering streak which is clearly associated to the domain structure. We believe that this feature is originated by the presence of random deformations present at the domain walls. It is interesting to note that this feature does not depend on the particular technique used to prepare the PPLN sample (in contrast e.g. with the vertical streaks of the electric field poled sample) and therefore seems to be characteristic of the domain wall structure.

To conclude the experiment successfully showed the possibility of probing structural modifications in PPLN samples with periods as large as some microns. Presently we are working on a mathematical description of the experimental maps. We believe that from a comparison of this model with the data, a wealth of quantitative information can be gathered regarding the presence of strain fields inside the PPLNs, as well as on the inner domain wall structure, domain period, domain wall inclination and so on.