ESRF	**Experiment title: In situ study of the piezoelectric, a-quartz isotypes aluminum phosphate and gallium phosphate in an electric field**	**Experiment number**: HS-1711
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Shifts:	**Local contact(s)**: J.F. Berar, N. Boudet	*Received at ESRF:*

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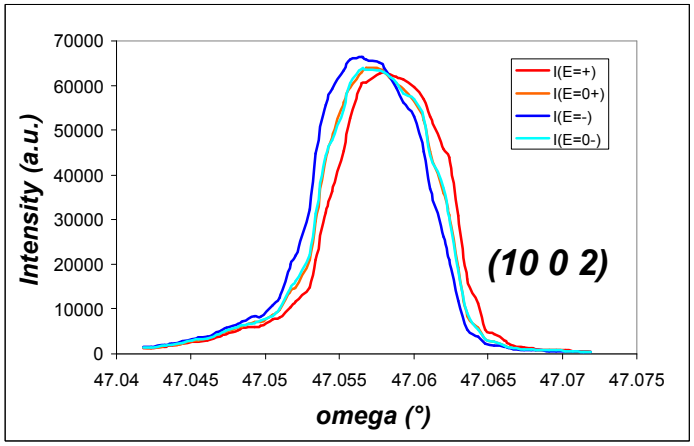
Report:

During the 18 shifts attributed for this experiment, we have performed in-situ electric field diffraction measurements on gallium phosphate ($GaPO_4$) single crystals in order to obtain a better understanding of the reinforced piezoelectric properties of this compound compared to those of low quartz.

We used a home-made device, which was interfaced with the computer driving the D2AM diffractometer: an alternating high voltage is applied to the sample to be studied generating a four levels electric field in the crystal (+E, 0_+, –E, 0_-). The counting of the diffracted photons is done synchronously on four different channels. Rocking curves of Bragg reflections are measured in a step-scan mode. At each step, the alternating high voltage is cycled several times, and the photons are accumulated in the four channels to reach a high signal over noise ratio. The X-ray wavelength was set at 0.62 Å. Preceding the measurement under applied electric fields, the diffraction conditions on a particular family of lattice planes have to be optimized: maximum diffracted intensity, rotation around the scattering vector to probe a uniform field region of the sample. In order to be able to measure small intensity variations when the field is applied ($\Delta I/I \sim 1\%$) a minimum of 10^6 integrated intensity is required. About 15 minutes are therefore required to measure the set of four rocking curves. Furthermore each scan was measured at least four times allowing the elimination of outliers due to possible experimental perturbations.

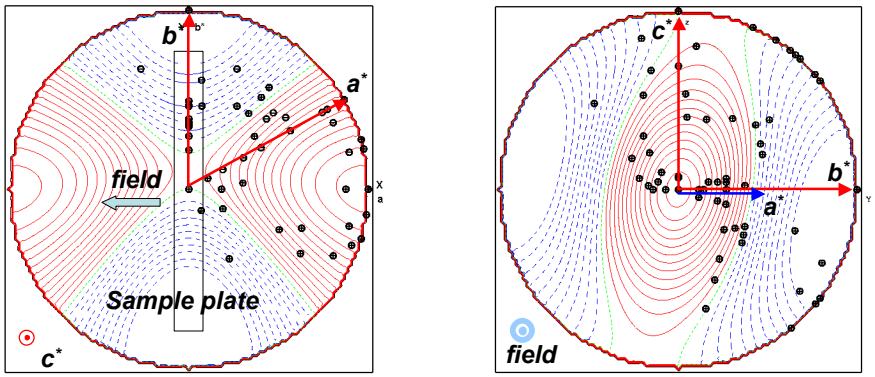
We also took profit of the high spatial resolution of the beam line: shifts in Bragg angles induced by the converse piezo electric effect ($\Delta\theta_B \sim 10^{-3}$ to 10^{-2} °) can easily be measured. We measured the angular shifts of 5 harmonics of the ($2h$ $-h$ 0) reflections (h = 3,4,5,6 and 7), and 3 harmonics of the (0 k 4k) family (k = 3, 4, 5). We were able to determine the two independent piezo electric moduli, d_{11} and d_{14}, of $GaPO_4$. Their magnitudes were in excellent agreement with the known values and therefore prove that the electric field is correctly applied to the sample.

Since we want to study shifts of atomic positions in the unit cell, we have carefully selected the Bragg peaks to be measured. High q scattering vectors ($q = \sin\theta / \lambda > 0.8$ Å^{-1}) were preferred, in order to be sensitive to the core electrons only, and only to have a minimal contribution from the valence electron on the diffraction. Furthermore, some reflections were periodically remeasured to detect any time evolution of the sample, or instabilities of our device. All together a total of 110 reflections were measured. A maximum of 5% intensity change was observed for the 10,0,2 reflection as illustrated in the figure below.



The angular shifts of the profiles as well as the intensity variations induced by the field are clearly visible.

The different orientations of the chosen scattering vectors gave a reasonable sampling of directions in the reciprocal space as shown in the figures below, where the stereographic projection of the measured reciprocal vectors is displayed.



Relative intensity variations of 65 crystallographically independent reflections could be injected in our special refinement program for determining shifts in atomic positions induced by the applied electric field: the main effects of the electric field on the crystallographic structure are a reorientation of almost rigid GaO_4 tetrahedra, and valence angle changes in the PO_4 tetrahedra. The deformations are about four times larger than what we find for α-quartz (a similar experiment, which we have carried out at LURE in 2002)

The results of this study have been presented in R. Guillot's PhD thesis (the 4th of Dec. 2002, Université Henri Poincaré – Nancy I) and an article will be published.