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Report:

Interaction of a crystal with a permanent external electric field is well known in the form of macroscopic phenomena such as dielectric polarization and converse piezoelectric effect. The atomistic origin of corresponding physical properties of solids (dielectricity and piezoelectricity) may be understood on the basis of precise investigation of the atomic movements induced by an applied electric perturbation. In the previous studies we used stroboscopic (modulation-demodulation) technique, involving periodic switching of the amplitude and direction of applied field and synchronized separation of the diffracted signal into corresponding time channels, to observe tiny displacements of atoms (~10⁻⁴ Å) from their field-free positions. The aim of the present work was to initiate the first X-ray diffraction study of dynamical structural responses of piezoelectric Li₂SO₄H₂O and semiconductor GaAs single crystals to a fast change of an applied electric perturbation. To do so a periodic stepwise high voltage up to 3.5 kV was supplied to the faces of plane-shaped crystals with a repetition rate of 18 Hz (Fig.1). To perform the time-resolved experiment we created the special FPGA-based data acquisition system, which was responsible for the redistribution of the detector counts into 4000 single time channels and their further summation over the periods of high voltage cycles. Depending on the experimental conditions the time resolution of the measurements was varied from 1 µs to 14 µs.

The measurements of the diffraction intensities of selected Bragg reflections were performed on a single crystal diffractometer (BM01A@ESRF, Swiss-Norwegian beamline) in the ω step scan mode (ω -step was 0.001 degree). The recorded rocking curves were used to calculate the change of the peak position, integrated intensity and full width at half maxima. As an example of obtained results we demonstrate the time dependence (Fig. 2 and Fig. 3) of the peak position and full width at half maxima (FWHM) of the (-7,-8,6) reflection near the 5-ms-slow switching of the high voltage. The profile of the high voltage is shown by the red line.



Fig. 1 Schematical illustration of four-step modulation-demodulation technique used for (quasi) simultaneous intensity measurements. The switching on the electric field from (red and blue) are ~ 100 times faster than switching off.



Fig. 2 The change of the peak position of the (-7,-8,6) reflection for Li₂SO₄H₂O crystal in the region of the 5-ms-slow switching of electric field.



Fig. 4. Change of the (0,0,10) peak position for a GaAs crystal during a single high voltage cycle. The states of the external high voltage are marked on the plot..

 $\begin{array}{c} \text{Li}_2\text{SO}_4\text{H}_2\text{O} (010) \text{ plate, Reflex (-7,-8,6), -3.5 kV -> 0} \\ \begin{array}{c} \text{Set}_{12} \text{ Time resolution is 40 } \mu\text{s} \\ \text{Time resolution is 40 } \mu\text{s} \\ \text{Set}_{12} \text{ Time resolution is 40 } \mu\text{s}$

Fig. 3 The change of the full width at half maxima of the (-7,-8,6) reflection for Li₂SO₄H₂O crystal in the region of the 5-ms-slow switching.



Fig. 5. Change of the (0,0,10) peak half maxima for a GaAs crystal during a single high voltage cycle. The states of the external high voltage are marked on the plot.

The same type of measurements was performed with the semiconducting GaAs crystal in applying the external electric field perpendicular to the (001) Miller planes. Fig. 4 and Fig. 5

demonstrate the position and the width of the (0,0,10) rocking curve during a full periodically alternating high voltage cycle.

Conclusions

We performed the first time-resolved X-ray diffraction studies of the Li₂SO₄·H₂O and GaAs responses to a permanent external electric field. Although a deeper analysis is necessary and underway, the first results show the presence of a small *delay* of a crystal response with respect to applied voltage. The most pronounced delay is observed for the change of the peak width, whereas a smaller one was measured for the peak position. We did not find any clear (within the presently achieved counting statistics and time resolution) differences between the behavior of the integrated intensity of a reflection and applied electric field. Up to now we did not succeed with the investigations of a crystal response to a faster than 50 µs switching of the electric field. At the same time simple theoretical calculations (simulation of elastic wave in a crystal induced by the time dependent external stress) show that the response of a system to a faster change of an external field may have the form of oscillations whose frequency depends on the internal parameters of the structure: The faster the change of an external force, the bigger is the amplitude of the induced oscillations. Therefore the observations of a crystal response to the ultrafast switching of an external electric field are a challenge for the next experiment. The ratio of the frequency of the free vibrations and the damping constant are also crucial for the description of the response of a system to an external perturbation. A system with dominated damping would show a delay instead of oscillations. This would explain the observed pronounced delay in the change of the peak width (related to the deformation of domain walls) in comparison to the behavior of the external high voltage.