



Experiment title: Study of the Electric Field induced Electron Density Changes as function of Temperature in Piezo-electric DKDP by means of single crystal X-ray Diffraction.

Experiment number:
HS-787

Beamline:
BM01A

Date of Experiment:
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Shifts:
17

Local contact(s):
K. Knudsen

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Names and affiliations of applicants (*indicates experimentalists):

A. Puig-Molina*, ESRF-Grenoble
H. Graafsma*, ESRF-Grenoble
S.J. van Reeuwijk*, ESRF-Grenoble

Report:

The experiment served three objectives. The first was to determine the quality of the crystal and specifically the crystal-mount. The second was to determine the d_{36} piezoelectric constant of DKDP when approaching the phase transition temperature, and to compare its temperature dependence with the temperature dependence of the d_{36} piezoelectric constant of the non-deuterated compound: KDP. The third was to follow for 1 or 2 reflections the change in integrated intensity as function of temperature.

At room temperature, the crystal produced sharp diffraction profiles: the FWHM of the (12 12 0) reflection is 0.01° . Applying a voltage of 400 V in the [001] direction, corresponding to an electric field of $1.3 \cdot 10^6$ V/m, was possible without inducing a significant current in the crystal.

By applying a quasi-static 30 Hz electric field in the [001] direction, the Bragg peak of a (hh0) reflection will separate. One peak corresponds to the negative field, while the other corresponds to the positive field. The d_{36} piezoelectric constant can be determined from the shift of the peak.

At room temperature and $E = 1.3 \cdot 10^6$ V/m, the (4 4 0), (10 10 0), (12 12 0) and (14 14 0) reflections were measured and the expected linear relationship between the shift of the peak and position of the peak, i.e. $\tan\Theta$, was found (figure 1). The slope of figure 1 is related to the d_{36} piezoelectric constant. The abscissa of the curve is, as expected, not zero and is related to the rigid rotation of the crystal. The d_{36} piezoelectric constant at room temperature was found to be 44 (2) pC/N, which is the same order of magnitude as values reported in literature [1].

Subsequently, the crystal was cooled. The temperature was measured at the end of the gas tube. Since the crystal was a few millimeters from that measuring point, and since the sample was large, the actual temperature of the crystal might deviate from the measured temperature by several degrees (knowing the transition temperature of DKDP, it is easy to correct for this deviation).

The (12 12 0) and (10 10 0) reflections were measured at temperatures approaching the phase transition point. The d_{36} piezoelectric constant at the various temperatures can be determined from these two reflections. The result, given in figure 2, shows that the d_{36} piezoelectric constant increases by a factor of 6 when going from room temperature to 230 K. The relation between d_{36} and the temperature is usually given by [2] $d_{36} = d_{36}^0 + B/(T - T_0)$, where T_0 is the transition temperature. In the vicinity of the Curie temperature the temperature independent constant d_{36}^0 can be neglected and B can easily be determined. From this experiment B was found to be $4.0(2) \cdot 10^{-9}$ KC/N for DKDP. From the data obtained in experiment HS-481, B can also be determined for the non-deuterated compound: KDP. A value of $3.79(3) \cdot 10^{-9}$ KC/N was calculated for KDP (literature reports a value of $4.2 \cdot 10^{-9}$ KC/N [2]). Although the transition temperature of KDP is 123K and of 98% deuterated DKDP is 223K, the shape of $d_{36}(T)$, expressed by the coefficient B , is the same.

At each temperature, not only the shift of the peak was to be determined, but also the difference between the integrated intensities when the electric field is positive and when it is negative. The diffractometer and the controlling equipment form an almost closed system. A perfect synchronisation of the detector output with the applied perturbation (i.e. the gating electronics) is not possible. This severely limits the accuracy of the measurements of the difference in integrated intensity (here typically 1-3 times the e.s.d.). As can be seen from figure 3, no hard conclusions can be drawn from these measurements concerning the temperature dependence of the difference in integrated intensity.

Cooling down the crystal to 230 K (according to the cryostat) was straightforward. Despite the fact that the crystal-mount was different than during a former experiment (HS-481; when the crystal cracked) the stress in the crystal (and thus the piezoelectric effect) is still limited and caused the crystal to crack at 229 K. Both in experiment HS-481 and this one, the crystal cracked when the d_{36} reached a value higher than roughly 200 pC/N.

References:

- 1) Landolt-Börnstein (1993), New Series III/29B, p. 151.
- 2) Franco Jona & G. Shirane, Ferroelectric crystals (1993), p. 73-74.

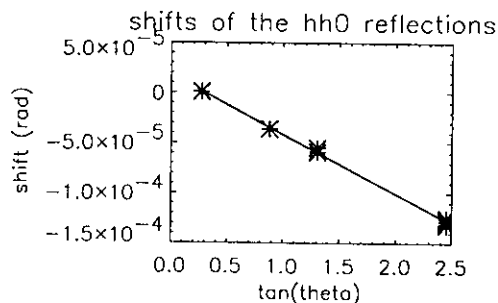


Figure 1: The shift as function of the position of the peak.

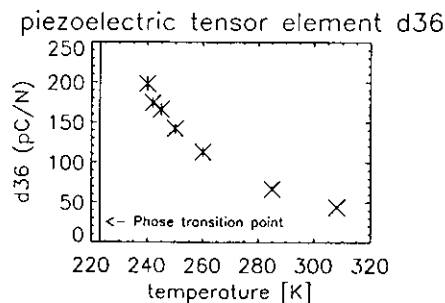


Figure 2: The d_{36} piezoelectric constant as function of temperature.

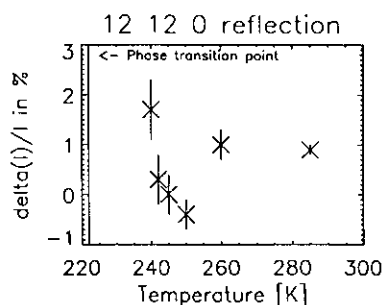


Figure 3: $\Delta(I)/I$ as function of the temperature.