ESRF

Experiment title: Study of Temperature Dependence of the Piezo-electric Effect of KDP by means of single-crystal X-ray Diffraction in an Electric Field.	**Experiment number:** HS-481

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Report:

The experiment served three objectives. The first was to determine the quality of the crystal. The second was to determine the d_{36} piezoelectric constant of KDP when approaching the phase transition temperature. The third was to turn the d_{36} piezoelectric constant into a temperature indicator.

At room temperature, the crystal proofed to produce nice diffraction profiles: the FWHM of the (12 12 0) reflection is 0.01°. Applying voltages in the [001] direction up to 1.00 kV, corresponding to an electric field of $4.3 \cdot 10^6$ V/m, was possible without inducing a significant current in the crystal.
By applying a quasi-static electric field in the [00l] direction, the Bragg peak of a (hh0) reflection will seperate. 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 the shift was measured applying 5 different electric fields, corresponding to: 0, 250, 500, 750 and 1000 V. The expected linear relation between the shift and the electric field was found (figure l), indicating the good quality of the crystal. Furthermore, we measured the (hh0) and (h-h0) reflections, where h = -12, -10, .., 10, 12, in the presence of an electric field. The expected linear relationship between the shift of the peak and position of the peak, i.e. $tan\Theta$, was found (figure 2), again indicating the good quality of the crystal. The slope of figure 2 is related to the d_{36} piezoelectric constant. The offset of the two datasets 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 $20.9(3) \cdot 10^{-12}$ C/N, which is close to values reported in literature [1,2,3].

Subsequently, the crystal was cooled. Due to the large surface size of the sample (10 * 10 mm^2) only a few settings of the crystal were practical for cooling down the sample. Other settings than those corresponding to the {12 12 0} and {10 10 0} reflections positioned the crystal out of the cold stream. 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 exact temperature of the crystal might deviate from the measured temperature.
The {12 12 0} and {10 10 0} reflections were measured at temperatures approaching the phase transition point. As is obvious from figure 3, the peak of the (12 12 0) reflection at 129 K is seperated completely. The d_{36} piezoelectric constant at the various temperatures can be determined. The result, given in figure 4, shows that the d_{36} piezoelectric constant increases by one order of magnitude.
Cooling down the crystal to 127 K (according to the cryostat) was straightforward. However, the way the crystal was mount limited the stress in the crystal (and thus the piezoelectric effect) and caused the crystal to crack at 125 K.

In combination with the data from Mason [l], the d_{36} piezoelectric constant was used to determine more accurate the actual temperature of the crystal. It turned out that 127 K according to the cryostat corresponds to 135.5 K according to Mason. This implies that we are still 12 K above the phase transition temperature.

References:

1) W.P. Mason (1946), Phys. Rev. 69, 173-194.
2) M.P. Zaitseva et al. (1982), Sov. Phys. Crystallogr. 27(l), 86-89.
3) Landolt-Bornstein (1993), New Series 111/29B, p. 151.

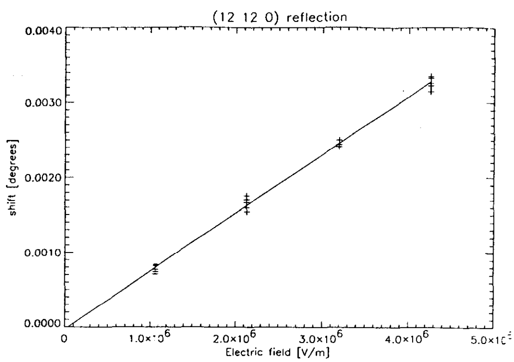

Figure 1: The shift as function of the applied electric field as measured for the (12 12 0) reflection.

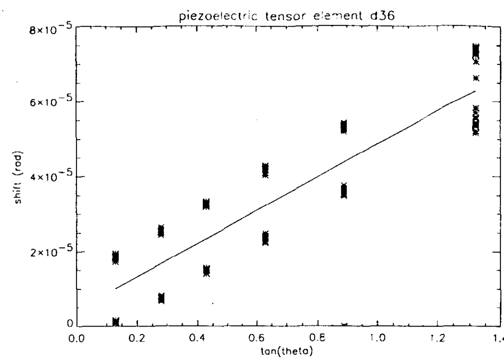

Figure 2: The shift as function of the position of the peak.

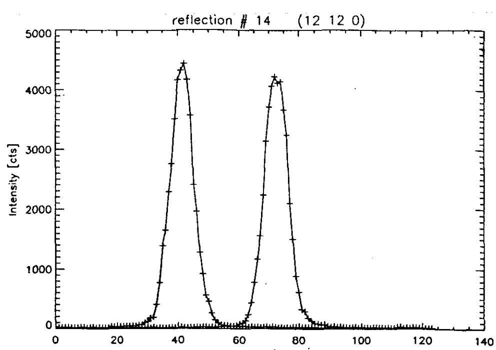

Figure 3: Seperation of the (12 12 0) peak at 129K in the presence of an electric field.

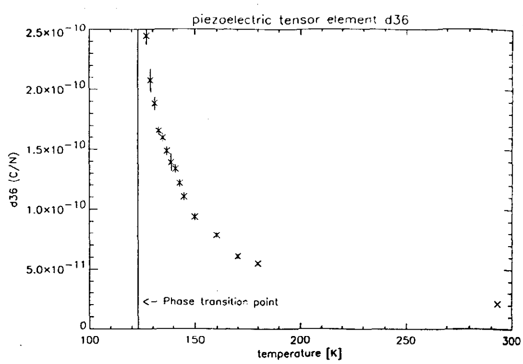

Figure 4: The d_{36} piezoelectric constant as function of temperature.