	Experiment title:	Experiment number:
	$KTiOPO_4$ (KTP) X-ray Diffraction under Electric Fields	HC:429
Beamline:	**Date of Experiment:**	**Date of Report:**
ID11/BL2	from: 20^{th} March to: 25^{th} March, 1996	28^{th} July, 1996
Shifts:	**Local contact(s):**	Received at ***ESRF:***
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Report:

KTP single crystals have strong second order non-linear optical properties, but KTP is also a good quasi-one-dimensional ionic conductor due to the high mobility of K^+ ions; the onset of the ionic conductivity is at about 150 K .

The aim of the experiment is to study the piezoelectric effect and the polarisation of the structure when a quasi-static electric field is applied to single crystals of KTP, below 150 K. The piezoelectric effect can be studied by measuring the modifications of the Bragg angles. The polarization on the other hand will result in a change in the integrated intensities.

This experiment is a continuation of an experiment which was allocated 9 shifts of beamtime in March 1995. The first campaign of measurements in 1995 should be considered a feasibility test for this type of compound. It nevertheless allowed us to make an estimate of the value of the piezoelectric constant of KTP: d_{333} = 6.8 pC/N at 100 K . With 5 days alloted this year a much more careful study of the piezo electric effect was made possible.

The first shift of beam time was used for alignment of the set-up, initial tests of the electronics and counting chains. The second shift was lost due to problems with the Synchrotrons Radiofrequency. During the following four shifts a first sample was studied. After 4 shifts a problem developed with the contacts on the crystal and it was therefore decided to change to a second sample which were used for the remaining nine shifts. No major problems were met during this period.

Both crystals used were shaped into 5 by 5 mm plates: Sample-1 0.32 mm thick, Sample-2 0.44 mm thick. The extended face is (O O 1) (spacegroup $Pna2_1$). Monochromatic X-rays of wavelength 0.56 Å was used throughout and the crystals were cooled to 100 K by a Nitrogen gas-flow cryostat. Electric fields of varying strength (O, 500, 1000 and 1500 V were applied to the sample, alternating between one direction, zero field and the opposite direction). The detection of the diffracted X-ray intensity is synchronized on three counting chains. The experiment was carried out at a frequency of 33 Hz. [1]

in order to minimize the effect of a possible build Up of space charges. We verified that changing the frequency did not affect the measurements.

With the first crystal only the evolution of the peak shape of the reflection O 0 36 was studied as a function of the electric field. The analysis of the induced changes of the peak shape is still under way.

The same type of measurements were also carried out with the second sample, which seemed to be of better quality (narrower rocking curves). Subsequently we measured the Bragg reflections O 01 with l = 14, 18, 20, 22, 28, 30, 32, 34 and 36 for both signs of 1. Each reflection was scanned at least 20 times allowing for improved statistics and testing the reproducibility of the experiments. There was no indication of instrumental instabilities as during the measurements in 1995.

RESULTS

It is observed that the rocking curves of the samples widen somewhat (5 to 10 per cent) during the first hours of exposure to the alternating field, and that the peak shape changes due to the applied electric field. This change is most likely related to space charge build-up at defects and domain boundaries, but a more careful analysis is currently under way.

We clearly observe a shift in the Bragg angles as a function of the field. It is a perfectly linear function of the field strength, but it correlates with the width of the rocking curves. The result therefore depends somewhat on the sample preparation. Analysing all the data collected for the second crystal we find a constant of

$$d_{333} = 8.5(1)\ \text{pC/N at 100 K}$$

This value we may compare with values measured using the direct piezo electric effect :

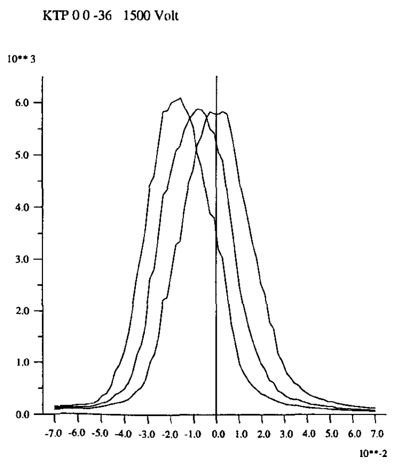
25.8 pC/N [2] , and 10.4 pC/N [3]

A paper about the observed peak shifts and corresponding piezo electric constant is in preparation and will be submitted shortly.

CONCLUSIONS

It would be interesting to analyze carefully the peak shapes on a diffractometer with very high angular resolution.

A second problem would be to measure possible variations in the Bragg intensities as a function of an applied electric field on well-caracterized samples.



Profiles corresponding to three fields: -,0,+

References

[1] Paturle, A., Graafsma, H., Sheu, H.-S., Coppens, P. & Becker, P., Phys.Rev.B43, 14683-14691 (1991)

[2] Sil'vestrova, I. M., Maslov, V.A. & Pisarevskii, Yu.V., Sov.Phys.Cryst. 37(5), 660-663 (1992)

[3] Chu, D. K. T., Bierlein, J.D. & Hunsperger, R.G. IEEE Transactions on Ultrasonics, Ferroelectrics, and Frequency Control, 39, 683-687 (1992)