ESRF	**Experiment title:** A 'hidden' phase transition in lithium niobate crystals highlighted by the ferroelectric domains presence?	**Experiment number**: ME-1246
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Report:

A periodically poled crystal contains a periodic array of inverted ferroelectric domains. The coherent beam wavefront is distorted, when Bragg-diffracted by such a crystal, through the phase difference between the structure factors of adjacent domains. The phase shift produces contrast on the images recorded at various sample-to-detector distances. This contrast is low (at room temperature) when the sample-to-detector distance is "small" (< 20 cm).

The low temperature experiment we performed on periodically poled lithium niobate crystals using the white beam section topography set-up has confirmed our preliminary, unexpected, results: when cooling below 190 K: a dramatic increase of the periodic contrast, in the form of black lines, occurs even for short sample-to-detector distances (10 cm).

We have performed a systematic study of the phenomenon:

1) repeated cooling and heating around T = 180 K (interval of temperatures going from 160 K to 230 K was used)

The effect appears and disappears in an inchanged manner, however a small hysteresis was observed. The phenomenon, observed on a detector set at a 'small' distance, seems to appear for T~ 190 K when cooling, and disappears at T ~ 180K when heating.

2) observation of periodically poled lithium niobate samples coming from different congruent crystals, with various periods of poling (19, 23 and 30 μm)

The phenomenon of appeareance/disappearance of domain contrast under temperature changes is not a fuction of the period of poling. It can, however, be related with the fact that all investigated samples were congruent. Non-stoichiometry is expresed by the ratio [Li]/[Li+Nb]=48.5% meaning that there is more Nb than Li in a congruent crystal. This leads to the presence of Nb antisites (Nb in the structure occupies the Li location) in the lattice.

3) observation of the phenomena on the sample with Al electrodes deposited on both large sample surfaces perpendicular to the c-axis

The aim of this experiment was to exclude a possible surface charge influence on the observed phenomena. No difference with non-electroded sample was noticed, the effect remains unchanged.

All experimental images were recorded also as a fuction of the sample-to-detector distance

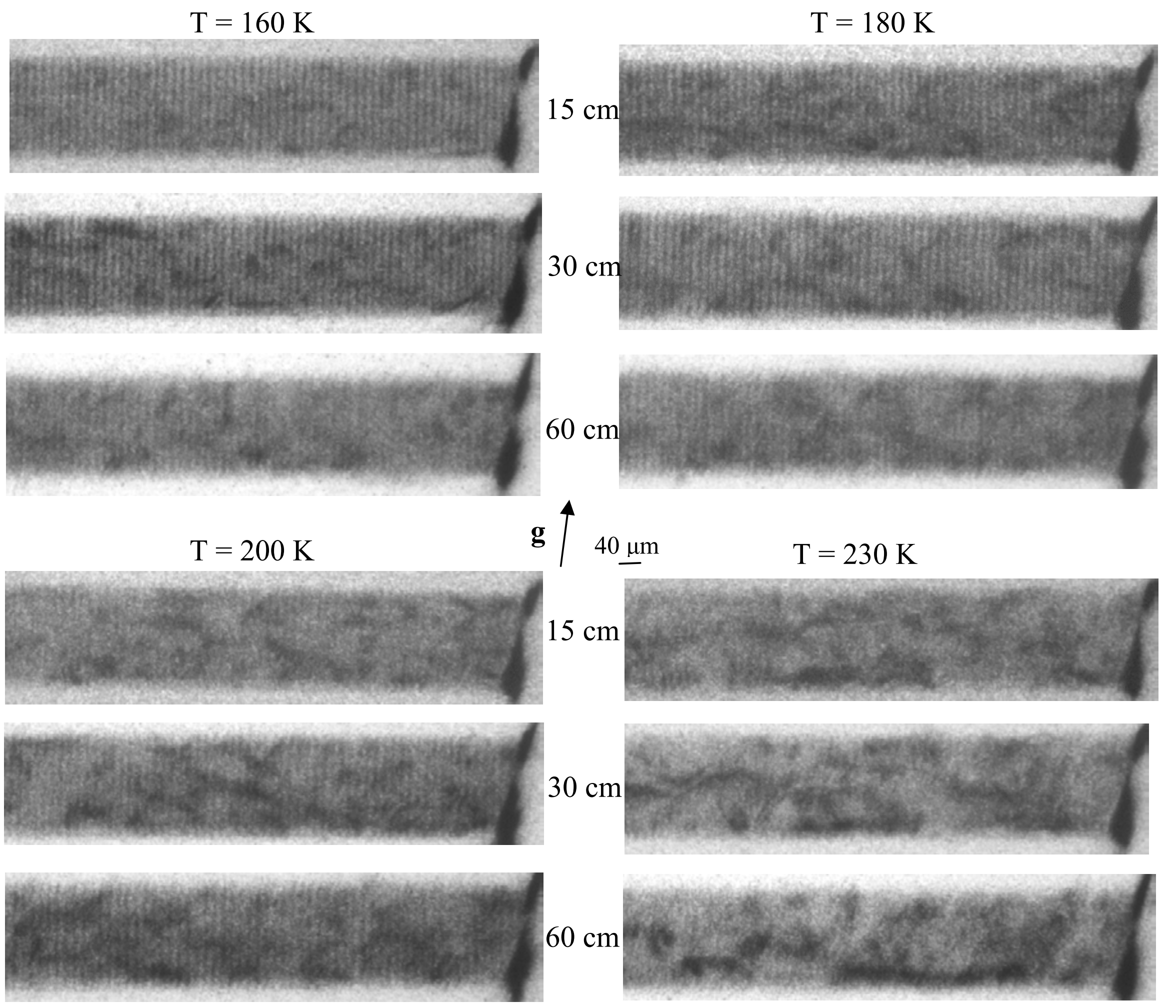

Fig.1: Series of white beam section topographs of $1\bar{3}\bar{2}$ reflection as a fuction of the sample-to-detector distance at various temperatures, λ=0.8Å, **g** is the projection of diffraction vector on the film, period of poling (and contrast) is 23 µm.

Fig. 1 shows a series of sections for the $1\bar{3}\bar{2}$ reflection taken as a function of the sample-to-detector distance at T = 160, 180, 200 and 230 K. Notice, for example, the vanishing of the domain contrast at 200 K if close (15 cm) from the sample, however the contrast seems even more pronounced if recorded further away (60 cm) than at 180 K. The phenomenon disappears completely (for all investigated sample-to-detector distances) at 230K.

There is clearly a wealth of detail to be extracted from these systematic studies; we are presently working to elucidate these phenomenon (and contrast mecanism) in the near future.