ESRF	**Experiment title:** Relaxation time at the lock-in phase transition in Thiourea	**Experiment number**: HS-1287
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Report:

Structural changes at phase transitions examined by X-ray diffraction are well known, little is known about the time scale over which these rearrangements take place. In this case we studied the kinetics of the electric field induced, first-order transition from the commensurate 1/9 lock-in to the ferroelectric phase in thiourea ($SC(NH_2)_2$).

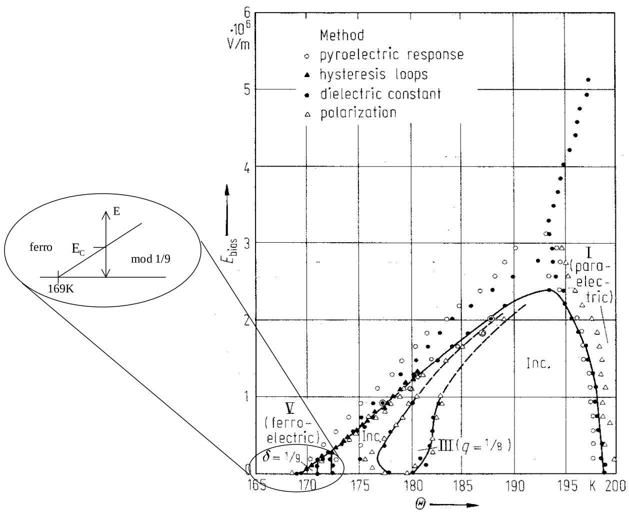

Fig.1

Figure (1) shows the (electric field E-temperature T) phase diagram of thiourea. On cooling down at zero field the system passes from the paraelectric phase (I) to an incommensurate modulated phase (II) with a

modulation vector **q** = δ(T) **b*** and $\delta > 1/8$ which then diminishes and locks into the commensurate phase (III) with $\delta = 1/8$. Further cooling leads to a subsequent incommenurate phase (IV) with shrinking δ until it locks in again at $\delta = 1/9$ before it disappears in the ferroelectric phase (V). A sufficiently high electric field E_c applied along **a** leads to a transition from the modulated phases to the ferroelectric phase (V) (fig.1) and the accompanying satellite reflection disappears.
A sample of 3 x 3 x 2.4 mm^3 was prepared with gold electrodes and mounted into an orange helium cryostat. The data was taken on the high resolution triple axis diffractometer of ID15A. Oxygen precipitated silicon 113 monochromator and analyzer crystals were used at 100 keV. Because of some mosaic spread of the thiourea sample we used the satellite reflection at Q = (2,2-δ,0) which has a sufficient longitudinal component to be observed by an analyzer rocking scan. On cooling, the reflection moved and remained stable in the 1/9 lock-in phase between 169K and 172K while it diminished drastically its intensity by applying an electic field above E_c.
In order to study the kinetics of the phase transition an oscillating electric voltage of rectangular shape (on - off) was applied. The detector was gated such that photon events were counted seperately for the high- and the zero-field half periods. A series of scans for both channels is presented in figure (2) and (3), respectively. The amplitude of the electric field was varied from 500 V to 0 V and then from 50 V to 450 V in 100 V steps. At each amplitude a set of 13 scans was taken changing the frequency from 512 Hz to 0.125 Hz.

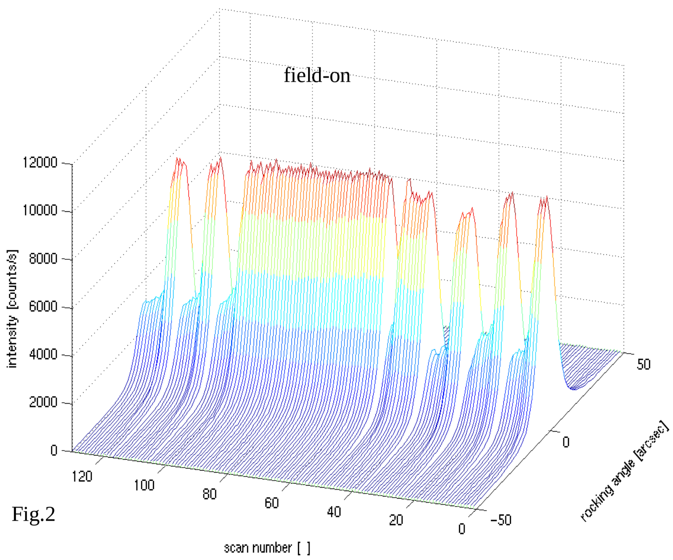

Fig.2

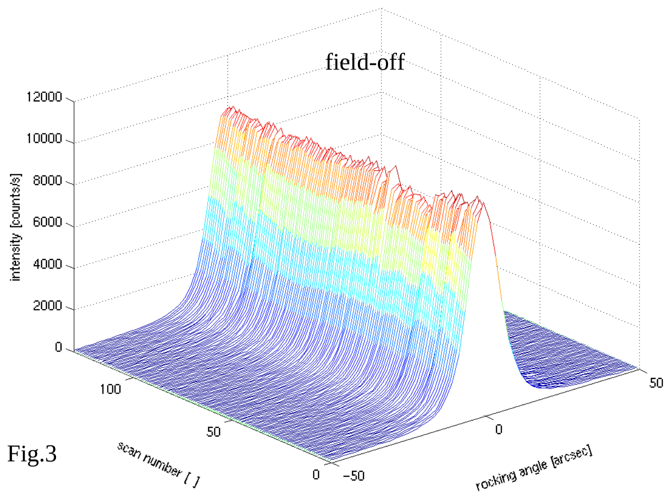

Fig.3

The first important result is the qualitatively different behaviour of both half periods: While the field-on intensities depend strongly on the amplitude and frequency no response is observed for all field-off data! Figure (4) presents the contrast ($(I_{off} - I_{on}) / I_{off}$) calculated from these intensities. It becomes large when the phase transition takes place and vanishes when the system remains in the modulated phase. It never reaches 100 % due to inhomogenities of the electric field in the sample and pinning effects at lattice faults. The characteristic "jump"–frequency in the contrast indicates the inverse of the time period the system needs to undergo the phase transition and depends strongly on the amplitude of the applied field.

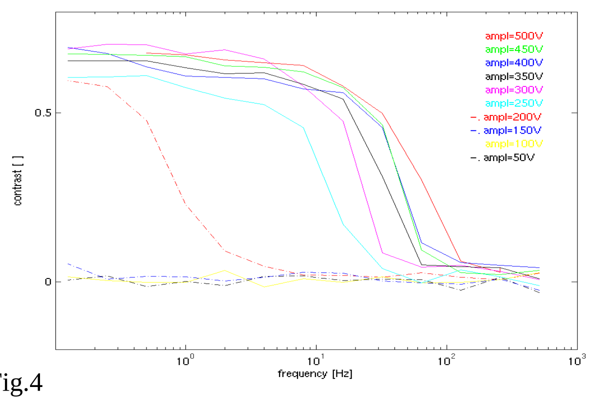

Fig.4

The experimental results could be explained by separate relaxation times for raising and falling field amplitudes. For very high frequencies one would at first expect a state which does not follow the oscillation and that is similar to a static field state with the time averaged field amplitude. The observed intensities, however, do not vary at all for the off-field half period. For the on-field cycle it strongly depends on the amplitude and frequency. It seems, that the relaxation time after switching the field off is immediate on the time scales investigated during the experiment (512Hz was the highest frequency applied), while it takes considerably longer at the raising field edge. The system stays close to the zero field state at high frequencies while it has enough time to relax at low frequencies. Raising field relaxation goes faster with higher amplitudes. This suggests that the relaxation time is inversely related to $| E - E_c |$, the potential difference between the applied and the critical field. Further experiments with a high time resolution in each half period should help to solve these questions.