



Experiment title: X-ray topography studies of the blue bronze $K_{0.3}MoO_3$ in the Peierls state with pinned or depinned charge density waves

Experiment number:
HE/1603

Beamline: ID19	Date of experiment: from:08/09/03 to: 12/09/03	Date of report: /02/04
Shifts: 15	Local contact(s): J. Baruchel, T. Schenk	<i>Received at ESRF:</i>

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Report:

The so-called blue bronze $A_{0.3}MoO_3$ ($A= K$ or Rb) is a quasi one-dimensional (1D) metal which undergoes at $T_p=180K$ a Peierls transition towards an incommensurate charge density wave (CDW) state. It shows non linear transport properties which are due to the sliding of the CDW in an electric field E larger than a threshold value E_t (2). In fields smaller than E_t the CDW are pinned by impurities and other defects.

The mechanism of depinning and of sliding of the CDW is not well known in spite of many experimental and theoretical studies. The role of CDW structural defects, such as discommensurations and CDW dislocations (phase slip centers) had been emphasized long ago. However, except for one preliminary experiment on the compound $NbSe_3$, no imaging of CDW deformation under sliding has been obtained up to now.

The aim of the present experiment was therefore to perform x-ray topographic imaging in the CDW state of the blue bronze, first in the pinned CDW state, to find out whether preexisting CDW defects or domains could be visualized by using one of the most intense CDW satellites. In the depinned state, one would look for CDW domains elongated along the 1D sliding direction away from the contacts. Close to the contacts, one might hope to find groups of CDW dislocation loops with a size large enough to induce detectable contrast on topographic images.

Since this was the first experiment of this type, the first task was to select blue bronze crystals of quality sufficient for x-ray topography. Several comparatively large crystals of $K_{0.3}MoO_3$ and $Rb_{0.3}MoO_3$, typically $3x2x0.1$ mm³, were selected. The best sample was a $K_{0.3}MoO_3$ crystal, polished on one face in order to diminish the number of surface defects. Electrical contacts were made by sputtering thin layers of silver with an appropriate geometry. The initial mounting of the sample, hanging through the electrical leads in order to minimize the strain effects, was found to allow strong vibration at low temperature. Finally, the crystal was fixed on a kapton film and glued by one corner only. Other small technical problems (leak in the cryostat, problems with a shutter and with the rotation) delayed or hindered the experiment. - The images were then made using the film technique.

The search for Bragg (fundamental) and CDW satellite peaks could be made at 70 K. The rest of the experiment was devoted to obtaining topographic images on the 15 1 2 fundamental and on the 15 1.25 2.5 satellite peaks. There is some uncertainty on these indices since both the “OrientExpress” software applied to Laue diagrams and numerical simulations for the satellite peak were unable to fit well the data. These discrepancies might be due to the high energy needed to obtain these peaks in the experiment geometry and consequently to the dense reciprocal lattice.

Fig.1 shows an X-ray topographic image made using a monochromatic, practically parallel beam of energy 30 keV at temperature 81 K on the *fundamental* reflection. Fig.2, shows the image made under the same conditions using the *satellite* reflection. There is at least one striking difference between these images,

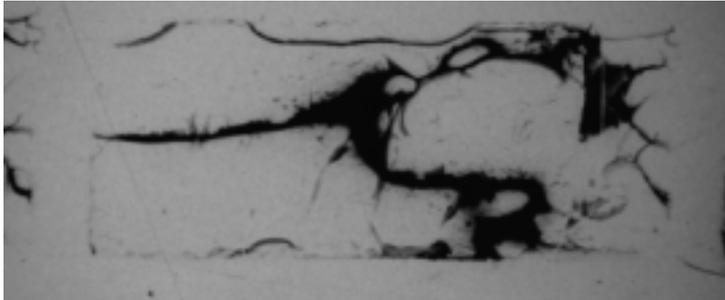


Fig.1 Fundamental reflection, fixed angular position. The length of the crystal is 4.5 mm

both recorded at a fixed angular position (other images were made with the sample rotated over the rocking curve during exposure). Whereas only small regions are imaged at a time on images using the fundamental reflection, as a result of the lattice distortions (mainly subgrain structure) of the specimen, the satellite reflection shows almost the whole sample. This is all the more striking

because the satellite is of comparable intensity, hence structure factor, as the fundamental reflection, and the Bragg angles are very similar

too. Thus the quantities characterizing the diffraction process (Darwin width and Pendellösung period) are of the same order of magnitude in both cases.

This effect, which to our knowledge was never observed before, may be related to the CDW coherence length. The subdivision into small regions as far as the CDW giving rise to the satellite reflection is concerned would lead to a broadening of the reciprocal lattice points, hence to a broadening of the intrinsic rocking curve, similar to a mosaic spread. This in turn would lead to much reduced sensitivity to lattice distortion. From the direct evaluation of the effective mosaic spread on the satellite photo, using this broadening, one obtains through the Debye formula $\Delta\theta = \lambda / d \sin \theta$ a transverse coherence length $d \approx 2000 \text{ \AA}$. This is consistent with the value of roughly 3000 \AA found along the $(2a^*+c^*)$ direction through non-imaging diffraction experiments.

X-ray topographs of the satellite peaks were made as a function of applied current, below and above the threshold for CDW sliding. The threshold field was found to be 500 mV/cm at 70K at the time of the experiment. Possible increase of this field due to x-ray irradiation cannot be excluded. This value corresponded to a threshold current of 8 mA. The pictures obtained for currents between 0 and 20 mA do not show obvious differences. However more detailed evaluations are in progress.

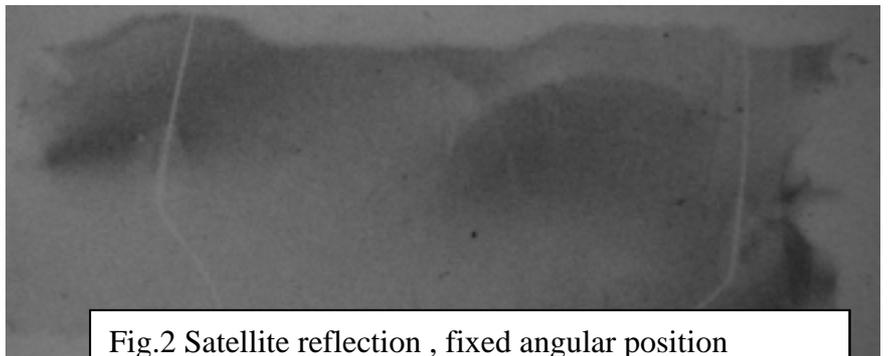


Fig.2 Satellite reflection , fixed angular position

As a conclusion, further experiments should be performed on better quality crystals. Smaller crystals will be chosen. They will be first characterized and preoriented in a crystallography lab (LPS Orsay). One may then hope to obtain pictures of an intense satellite peak and possibly to see the effect of an applied current at least in the vicinity of the electrical contacts.