ESRF	**Experiment title:** X-ray Photons Correlation Spectroscopy on pinned charge-density waves in the blue bronze $Rb_{0.3}MoO_3$	**Experiment number**: HE-1284
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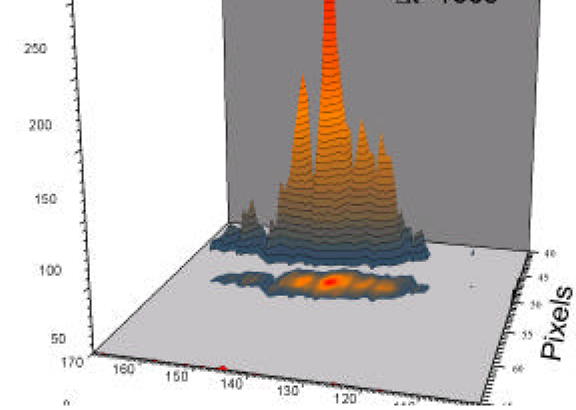
Report:

The quasi-one-dimensional (1D) conductor $Rb_{0.3}MoO_3$ (blue bronze) undergoes a Peierls transition which stabilizes a incommensurate charge-density wave (CDW) at twice the Fermi wave vector $2k_F$. This CDW is a fascinating example of an electronic crystal, having the capability to transport a non-ohmic current by sliding under a large enough electric field. Below T_c= 183 K, the lattice distortion associated to the CDW gives rise to satellite reflections at the (H,K+0.748,L+0.5) reciprocal positions. Due to the impurities inevitably present in the crystal, the CDW is never long range ordered but pinned by the defects. The purpose of this experiment was to use coherent x-rays to study the speckle pattern of the satellite reflections in the pinned state, and to examine its change in the sliding state.

Four gold contacts were deposited onto a blue bronze of standard size ($1{\times}0.5{\times}0.2mm^3$). The crystal was then attached to a sapphire disk by four gold wires glued with GE varnish. The disk was pasted with silver paint onto a copper sample holder and mounted inside an orange He cryostat. The b axis (chain axis) was vertical so that the (H0L) reciprocal plane was in the horizontal diffraction plane. With 8 keV photons, a reflection geometry was needed. In this configuration, the strongest observable satellite reflection was at q_s=(6 0.25 –3.5). The conditions of coherence were obtained by placing $10{\times}60\mu m^2$ (H×V) secondary slits and 5 or 10 µm pinholes 1 m and 10 cm before the sample, respectively. The temperature was kept at 100 K during the whole study.



The speckle patterns were measured by a direct illumination CCD camera with 22x22 μm^2 pixel size, placed 2 m away from the sample. For each measurement, an average of 500 to 1000 frames were taken, with 1 s exposure time, and the resulting images were obtained by averaging 100 frames. A typical speckle pattern is shown in the opposite figure. The horizontal directions are the chain direction **b*** and the transverse 2**a***-**c*** direction (from left to right). The satellite peak is clearly anisotropic, much thinner in the **b** direction, which is expected from the quasi-1D character of the compound. CDW domain sizes could be measured for the first time, given the high resolution provided by the CCD camera (4.8 10^{-5}Å^{-1}). The Half-Width at Half-Maximum Δ of the satellite peaks were Δ_{b*}= 0.7 10^{-4}Å^{-1}, Δ_{2a*+c*}= 1.9 10^{-4} Å^{-1}, Δ_{2a*-c*}= 1.03 10^{-3} Å^{-1}, giving L_b~1 µm in the chain direction, L_{2a*+c*}~0.3 µm and L_{2a*-c*}~600 Å in the transverse directions 2a*+c*, and 2a*-c* respectively. The L anisotropy ratio is consistent with the anisotropy of the correlation lengths measured by x-ray diffuse scattering. Nevertheless, the domain sizes and the speckle pattern both depend on the exact position of the beam on the sample, as can be seen from the figure 2.

The speckle patterns present only one speckle in the chain direction, while 3 or more speckles are clearly visible in the 2a*-c* direction. This can be interpreted by the fact that the CDW domain size is of the same order of magnitude (a few µm) as the grain size of the blue bronze in the chain directions. This is not the case in the transverse direction, where the speckles are due to the interference between many domains.

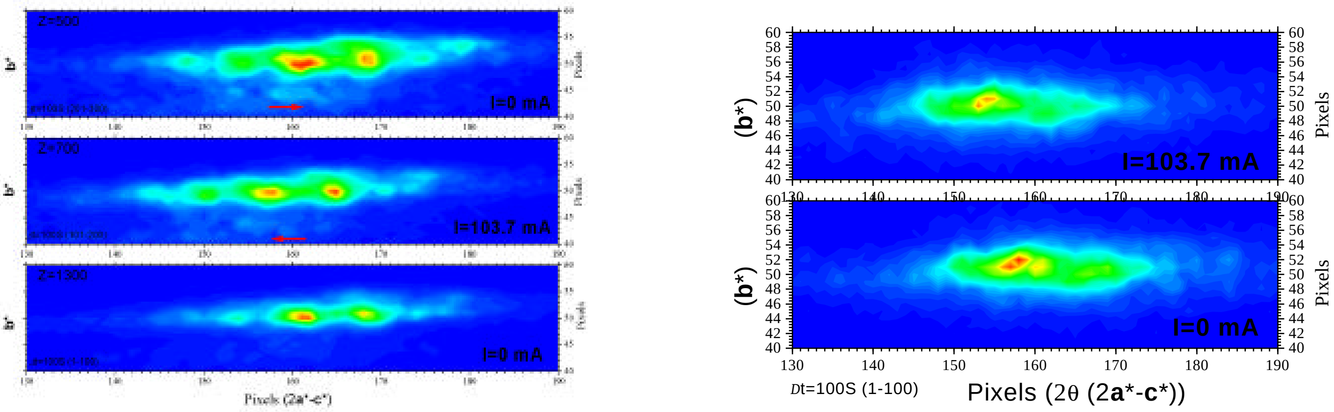


Figure 2 : Speckle patterns of the (6 0.25 –3.5) satellite reflection. The left and right patterns have been obtained for different position of the beam on the sample. Their shape are slightly different but the anisotropy is still clearly visible. The values of the current are indicated.

This experiment should have been performed one year ago, but due to a problem on the beamline, it was postponed. To compensate, the ID20 staff provided us three days to test the feasibility of the experiment. This preliminary experiment had shown that by applying a current of about 2 mA, on a similar crystal, the satellite reflection was slightly displaced in the 3 reciprocal space directions [1]. This was considered as the footprint of the sliding state, as previously noted [2].

On the **same** crystal as used in this study, a diffraction experiment at the Advanced Photon Source performed one month before had shown the sliding state to occur for I>0.9 mA. However, we could not reproduce this result. A slight shift of the satellite position was observed for I~60 mA and for larger current values, the satellite was sizeably shifted, as shown in fig. 2. However, measurements of I(V) characteristics after the experiment showed that this shift was mainly due to a Joule heating of the crystal. From the observed shift of the satellite position and the temperature dependence of the lattice parameters, we estimate the heating to be 5 K at 100 mA. Of course, the possibility that the CDW was also in the sliding state is not excluded, but this would have been hidden by the heating effect. Such damaging of the contacts, which are very fragile in this compound, are well known and difficult to avoid in this compound.

It is noteworthy that the speckle patterns slightly evolved during long measurements. We could not find the reason for this instability. Due to the weak amount of heat provided by x-rays, a temperature effect is to be excluded. Irradiation effects, already observed in blue bronze, could occur and could be minimized by attenuating the beam. Finally, a slight drift of the sample inside the cryostat could also explained such an effect.

In conclusion, we have clearly observed a speckle pattern of the CDW satellite reflections on the Rb blue bronze, which definitely shows the feasibility of our project on ID20. However, the quality of the contacts, which is a key parameter for the success of the experiment, has to be improved. In particular the crystals have to be carefully tested a few days before the experiment, in order to avoid the damaging of the contacts.

[1] D. Le Bolloc'h, S. Ravy, P. Senzier, C. Pasquier and C. Detlefs, *J. Phys. IV France* **12**, Pr9-323 (2002).
[2] T. Tamagai et. al., *Solid State Commun.* **51**, 585 (1984).