Speckle Experiments on Charge Density Waves: Blue Bronze (Rb_{0.3}MoO₃) under an Electric Field

D. Le Bolloc'h,¹ S. Ravy,¹ P. Senzier,¹ C. Pasquier,¹ M.V. Holt,² F. Picca,³ M. Sutton³

¹Laboratoire de Physique des Solides, Université Paris-Sud, Cedex, France

²Advanced Photon Source (APS), Argonne National Laboratory, Argonne, IL, U.S.A.

³Physics Department, McGill University, Montréal, Québec, Canada

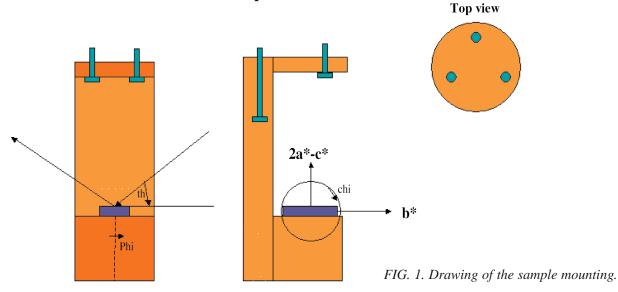
Introduction

The quasi-one-dimensional (quasi-1-D) conductor Rb_{0.3}MoO₃ (blue bronze) undergoes a Peierls transition that stabilizes an incommensurate charge-density wave (CDW) at twice the Fermi wave vector 2k_F. This CDW is a fascinating example of an electronic crystal that can transport a nonohmic current by sliding under a large enough electric field. Below $T_c = 183K$, the lattice distortion associated with the CDW gives rise to satellite reflections at the (H+1, K+0.748, L+0.5) reciprocal positions. Because of the impurities inevitably present in the crystal, the CDW is never long-range-ordered but is 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.

Methods and Materials

Four gold contacts were deposited onto blue bronze of standard size $(1 \times 5 \times 0.2 \text{ mm}^3)$. The crystal was then attached to a sapphire disk by four gold wires glued with General Electric (GE) varnish. The disk was pasted with silver paint onto a copper sample holder. The sample was cooled down to 100K by using a He flux cryostat available on the 8-ID beamline, which does not generate any vibration. The b axis (chain axis) was horizontal so that the (H0L) reciprocal plane was in the vertical diffraction plane (Fig. 1). 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 using 5, 10, or 20-µm pinholes, 10 cm before the sample at E = 6 keV. The temperature was kept at 100K during the whole study.

The speckle patterns were measured by a direct illumination charge-coupled device (CCD) camera with a 22×22 -µm² pixel size, placed 1.8 m away from the sample. For each measurement, an average of 500 to 1000 frames were taken with a 1-s exposure time, and the resulting images were obtained by averaging 100 frames. A typical speckle pattern is shown in Fig. 2. The satellite peak is clearly anisotropic and much thinner along the **b*** direction, which is expected from the quasi-1-D character of the compound. The orthogonal direction is close to the **2a*-c*** direction ($\theta = 21.8^{\circ}$).



Sample holder

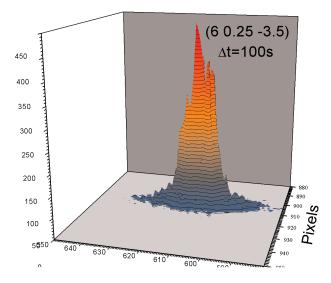


FIG. 2. Speckle patterns of the $(6\ 0.25\ -3.5)$ satellite reflection

Results and Discussion

CDW domain sizes could be measured for the first time in the pure blue bronze, thanks to the high resolution provided by the CCD camera ($\Delta Q = 4.8 \times 10^{-5} \text{ Å}^{-1}$). The half width at half-maximum (HWHM) Δ of the satellite peaks were $\Delta_{b^*} = 0.7 \times 10^{-4} \text{ Å}^{-1}$, $\Delta_{2a^*+c^*} = 1.9 \times 10^{-4} \text{ Å}^{-1}$, and $\Delta_{2a^*-c^*} = 1.03 \times 10^{-3} \text{ Å}^{-1}$, giving a L_b of ~1 µm in

the chain direction and a $L_{2a^*+c^*}$ of ~0.3 μ m and a $L_{2a^*+c^*}$ of ~600 Å in the transverse directions $2a^*+c^*$, and $2a^*-c^*$, respectively. The anisotropy ratio is consistent with the anisotropy of the correlation lengths measured by x-ray diffuse scattering. It is noteworthy that the shape and the width of the satellite reflection are very sensitive to the exact beam position on the sample. Those values have been obtained from the thinner profiles that we obtained during the experiment.

The speckle patterns present only one speckle in the chain direction, while several speckles are clearly visible through 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 micrometers) as the grain size of the blue bronze in the chain directions. This is not the case in the transverse direction, where the few speckles are induced by phase shifts along the CDW, as already observed in NbSe₃ [1].

A preliminary experiment performed at the European Synchrotron Radiation Facility (ESRF) had shown that by applying a current of about 2 mA on a similar crystal, the satellite reflection was slightly displaced in the three reciprocal space directions [2]. This was considered as the footprint of the sliding state, as previously noted [3].

A similar effect has been observed at the APS. A slight shift along the $2a^*-c^*$ has been observed for currents larger than 0.9 mA (see Fig. 3). However, we could not reproduce this result, probably because of the

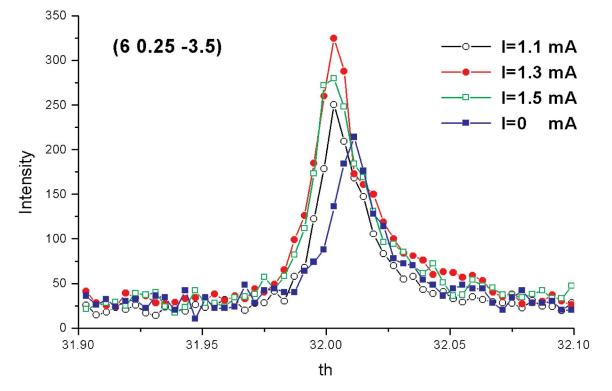


FIG 3. Slight shift of the satellite reflection along $2a^*-c^*$ for currents larger than 0.9 mA..

damage of the contacts. Measurements of I(V) characteristics after the experiment showed that the nonlinear I(V) curve was mainly due to a Joule heating of the crystal. Such damaging of the contacts is known to be difficult to avoid in this compound.

It is noteworthy that the speckle patterns and the intensity evolved slightly during measurements. The cause of this instability was either the beam instability or irradiation effects already observed in the blue bronze. (The irradiation damage induces a decrease in intensity, but beam fluctuations were irratic.)

In conclusion, the observed speckle pattern of the CDW satellite reflections on the Rb blue bronze definitely shows the feasibility of using time fluctuations on this system.

Acknowledgments

Use of the APS was supported by the DOE Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-ENG-38.

References

[1] M. Sutton, Y. Li, J.D. Brock, and R.E. Thorne, J. Phys. IV France **12**, Pr9-3 (2002).

[2] D. Le Bolloc'h, S. Ravy, P. Senzier, C. Pasquier, and C. Detlefs, J. Phys. IV France **12**, Pr9-323 (2002).

[3] T. Tamagai et al., Solid State Commun. **51**, 585 (1984).