

Transverse correlations and plasticity in the CDW conductor NbSe₃ studied by X-ray microbeam diffraction

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Abstract. In whisker-like samples of the quasi-1D conductor NbSe₃, the presence of longitudinal steps causes shearing of the CDW, and leads to a loss of transverse correlations. We use a microdiffraction setup with a spatial resolution of 300 nm and an angular sensitivity of 5 mdeg to image the resulting CDW contrast between thick and thin portions of the sample. Microdiffraction in the $b^* - c^*$ plane shows that depinning on the thick, weakly pinned side is accompanied by the loss of diffraction intensity, demonstrating a loss of correlations in qualitative agreement with previous X-ray diffraction topography measurements¹, but with an order-of-magnitude improvement in spatial resolution. Microdiffraction images in the $a^* - b^*$ plane reveal a sharp increase in the full width at half maximum in an approximately 1 micron thick region near the step edge and a rotation of the CDW wavevector that varies with applied field. We use the extremal value of the CDW wavevector rotation to estimate the shear modulus of this electronic crystal.

1. INTRODUCTION AND MOTIVATION

The strong interplay between the elastic and plastic properties of charge density waves (CDWs) and their electronic properties has yielded a wealth of information on the mechanics of these electronic crystals [1]. We demonstrate the first application of the X-ray microbeam diffraction to CDWs to determine the shear strain and the shear modulus of a driven CDW. A natural way to examine CDW shear in quasi-1D compounds like NbSe₃ is to employ their ubiquitous longitudinal steps in crystal thickness that cause non-uniform pinning. Shearing along steps produces a loss of CDW coherence that can be probed by microdiffraction [2, 3]. We expect micro-beam diffraction to provide a powerful probe of the character of the CDW depinning transition.

2. EXPERIMENTAL METHODS

X-ray micro-beam diffraction has recently emerged as a tool for structural studies of strongly correlated compounds and other complex condensed matter systems. Examples of its application to understanding mesoscale structural properties include domain switching in spin wave systems [4] and similar studies in manganates [4].

Figure 1 shows the micro-beam setup at Advanced Photon Source beamline 2-ID-D. 10 keV X-rays are selected by a double bounce Si(111) monochromator and passed onto focusing X-ray optics composed of a Fresnel zone plate and a 30 μm order sorting aperture. The diffracted X-rays are collected using a photon-counting detector (CCD camera or pin diode). The incident flux does not exceed 5×10^9 photons/second. Samples are mounted on X-ray transparent, 60 micron thick silicon substrates with prefabricated four-point probes. Samples are placed in a He-flow cryostat which is then placed in a six-circle diffractometer. Data are collected in the form of images, obtained by raster scanning the sample relative to the zone plate while respecting the diffraction condition of a fixed scattered wave vector.

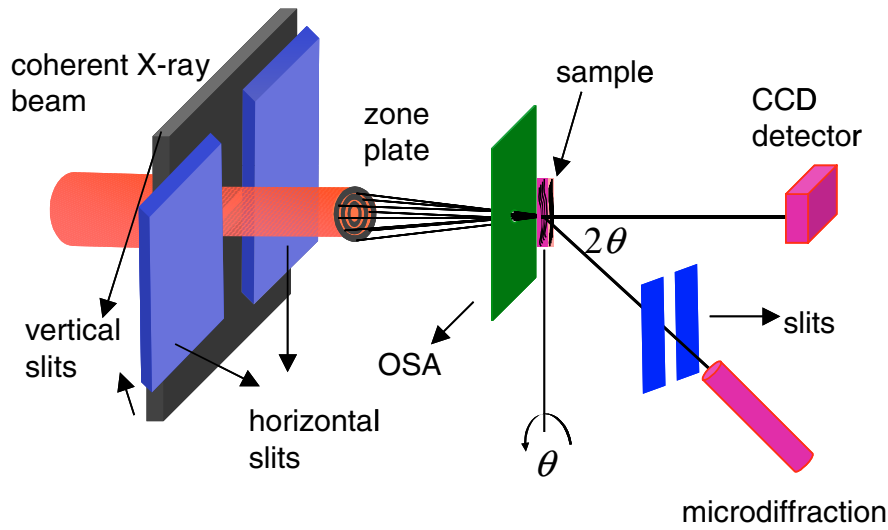


Figure 1. Schematic of the micro-beam diffraction setup at the Advanced Photon Source (2-ID-D). Slits can be controlled by hand or automatically. The sample is placed in a He-flow cryostat. OSA stands for order-sorting aperture.

Samples are carefully selected to have only one longitudinal step. Their profile is confirmed by a combination of optical and atomic force microscopy. The step heights for three samples discussed here are between 400 nm and 700 nm, thicknesses are less than 2 microns, and widths are between 8 and 16 microns.

3. RESULTS

Two kinds of microdiffraction measurements were performed. Figure 2 shows the microdiffraction intensity upon CDW depinning recorded in the $b^* - c^*$ plane for sample B at $T = 120$ K. The region with a higher pinning strength produces a stronger intensity, indicating a smaller rotation (tilt) of the CDW q -vector. This type of behavior was suggested in previous X-ray topography experiments [2], but microdiffraction provides an order of magnitude improvement in spatial resolution. The width of the region with reduced intensity has a nonlinear dependence on the electric field [5]. The most interesting region in which to examine the shearing process is the interface between the thick and thin portions of the crystal, because the lower portion of the thick part is held back by the more strongly pinned thin part.

Maps and rocking curves acquired in the $a - b^*$ plane at several dc biases are presented elsewhere [5]. Figure 3 shows the full-width at-half-maximum in the step region. Both samples show a sharp increase of the FWHM within ~ 1 μm of the longitudinal step, indicating a loss of transverse coherence. The extremal rotation of the CDW wavevector is about 30 mdeg, which leads us to a value for the maximum CDW shear strain of $\epsilon_s \sim 5 \times 10^{-4}$. Using the value for the plastic shear strength of $\sigma_s = 9.5 \times 10^3 \text{ N/m}^2$ determined by O'Neill *et al.* [3], we estimate that the CDW shear modulus is $G = 1.8 \times 10^7 \text{ N/m}^2$.

4. ELASTIC AND PLASTIC PROPERTIES OF NbSe_3

With the present results, a reasonably complete and consistent picture of the elastic and plastic properties of the $T_{P1} = 145$ K CDW in NbSe_3 has emerged. Table 1 briefly summarizes these properties and the methods used to determine them.

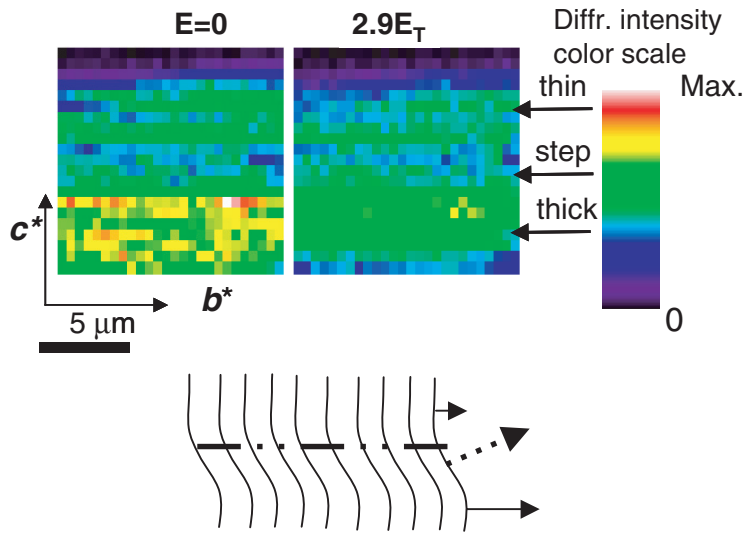


Figure 2. Images show a decrease of the microdiffraction intensity for sample B, after depinning and due to the loss of transverse coherence ($T = 120\text{ K}$). The sketch below images schematically indicates a different wave front dynamics in two parts of the crystal, and the tilt of the CDW q -vector.

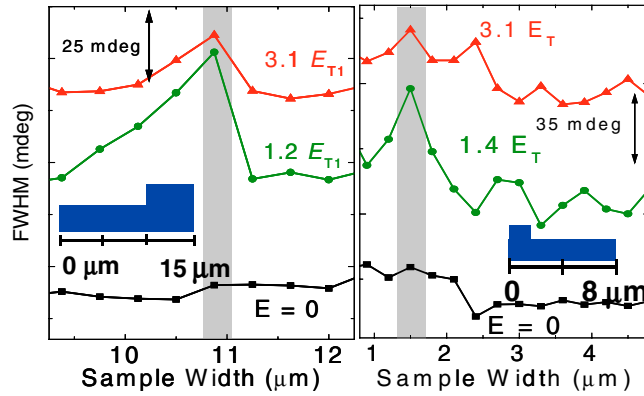


Figure 3. A comparison of the lateral spatial variation of the FWHM of rocking curves in two stepped samples of NbSe_3 . Sketches represent the a^* - b^* plane profiles of each sample. Data for sample A (left) is at $T = 90\text{ K}$, while data for sample C (right) is at $T = 110\text{ K}$.

Table 1. Summary of the elastic and plastic properties of NbSe_3 .

Quantity	Value	Experimental method and Reference
Crystal Young modulus	$1.5 \times 10^{11} \text{ N/m}^2$	Vibrating reed, [7]
CDW Longitudinal strain	1.3×10^{-3}	X-ray diffraction [8]
CDW Longitud. modulus	$9 \times 10^8 \text{ N/m}^2$	
CDW plastic shear stress	$6.6 \times 10^3 \text{ N/m}^2$ $9.5 \times 10^3 \text{ N/m}^2$	Differential resistance measurements on nanofabricated steps [3]
CDW Shear strain	5×10^{-4}	X-ray microdiffraction, [5] and this report
Shear modulus	$1.8 \times 10^7 \text{ N/m}^2$	

5. CONCLUSIONS

A novel method, microbeam diffraction, is presented as a tool for studying dynamics of elastic and plastic deformation in driven CDWs. This method yields values for the CDW shear strain and shear modulus in NbSe₃. The ratio of shear and longitudinal moduli is consistent with the measured electronic anisotropy of NbSe₃ [9].

Acknowledgments

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References

- [1] J. P. Pouget, “*Structural and dynamical aspects of the CDW instability, in Physics and Chemistry of Low-Dimensional Inorganic Conductors*”, eds. C. Schlenker, M. Greenblat, J. Dumas, and S. van Smaalen (NATO-ASI, vol. 254, Plenum, New York, 1996).
- [2] Y. Li *et al.*, Phys. Rev. Lett. **83**, 3514 (1999).
- [3] K. O’Neill, K. Cicak, and R. E. Thorne, Phys. Rev. Lett. **93**, 066601 (2004).
- [4] P. G. Evans *et al.*, Science **295**, 1042 (2002); Y. Soh *et al.*, J. Appl. Phys. **91**, 7742 (2002).
- [5] A. F. Isakovic *et al.*, submitted for publication (2005), manuscript available upon request.
- [6] J. W. Brill and N. P. Ong, Solid State Comm. **25**, 1075 (1978).
- [7] DiCarlo *et al.*, PRL **70**, 845, Adelman *et al.*, PRB **53**, 1833, Requardt *et al.*, PRL **80**, 5631.
- [8] N. P. Ong and J. W. Brill, Phys. Rev. B **18**, 5265 (1978); E. Slot and H. S. J. van der Zant, J. Phys. IV **12** (Pr9), 103 (2002).