

Observation of Ring-Like Lattice Modulation Domain in Single Crystal $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ Using X-ray Microscopy

Zhonghou Cai¹ and Yang Ding²

¹ Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois, USA

² Center for High Pressure Science & Technology Advanced Research, Beijing, P.R. China

* Zhonghou Cai, email_cai@aps.anl.gov

The crystal structure of the $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ or Bi-2212 was determined two decades ago when its high-temperature superconductivity emerged [1]. The structure contains copper-oxygen sheet and is further made up of alternating double Cu-O sheets and double Bi-O sheets. There are Ca^{2+} cations between the adjacent Cu-O sheets and Sr^{2+} cations between the adjacent Cu-O and Bi-O sheets (Fig. 1a). The variations in cell dimensions occur naturally in Bi-2212 taking the form of an incommensurate superstructure along the *b* axis that is believed to originate from a mismatch in preferred bond length between perovskite and rock-salt layer of the crystal. Based on the observations of strong variations in superconducting critical temperature among cuprates that have identical hole density but are crystallographically different outside of the Cu-O sheet, attention has also focused onto the out-of-sheet influence on the superconductivity, with particular attention, for example, on the distance between the apical oxygen and the Cu-O sheet plane [2]. The structure modulation perturbs atomic positions and displaces atoms from their mean locations with a functional form repeating about every 4.8 unit cells (Fig. 1b). The modulation creates periodic variations in crystallographic structure outside of the Cu-O sheet, and thus may have roles in Bi-2212 HTSC. Rather than concentrating on its effect on HTSC, we focus this study on spatial distribution of the structure modulation in single crystal Bi-2212.

The complexity arising from the multi-sheet structure in the Bi-2212 unit cell and its association with the incommensurate structure modulations results a large number reflections of fundamentals and various orders of satellites. Fig.2b is a diffraction pattern in transmission geometry of the single crystal Bi-2212 with the *c* axis along the incident beam. The diffraction pattern (Fig. 2a) becomes complicated while the sample is rotating along the *b* axis (ϕ angle) during exposure, probably due to the twinning that makes it appear that the superstructure is along both *a* and *b* axes. A sample rotation around *b* axis can also generate additional *L* indexed fundamentals and related satellites.

Spatial distribution of the structure modulations of Bi-2212 was investigated with the X-ray microscopy at APS 2-ID-D beamline [3]. The measurements used 20 keV X-ray radiation generated from APS Undulator A and selected with a Si(111) double crystal monochromator. The beam was focused by an Au zone-plate to a spot size of 400 nm. Sample areas were mapped with the integrated intensities of a series of ROIs that cover various reflections in Fig. 2a. We display in Fig 3 the maps obtained from three satellite reflections near (100), (210), and (300), referred as A, B and C in Fig. 2a. The existence of the satellite spots depends not only on the sample angle but also on the sample area. The satellite spot near a Bragg reflection is resulted from a constructive interference of the diffracted lights from the Bragg reflection that modulate spatially due to the superstructure modulation. Areas that contribute to the satellite diffraction spot form a ring, and the ring size increases continually as ϕ angle increases. The rings start to form at different angles for different satellites. For example, the ring corresponding to the satellite of ROI A forms at $\phi = 10.8^\circ$, that corresponding to the satellite of ROI B forms at $\phi = 10.45^\circ$, and that corresponding to the satellite of ROI C forms at an angle smaller than 10.4° . Spatial feature revealed through a satellite spot

at a sample angle can reappear through other satellite at a different angle. The spatial features in the maps obtained from the satellite A at 11.15° , the satellite B at 10.75° , and the satellite C at 10.50° are almost identical.

The diffraction maps shown in Fig. 3 reveal a novel geometric configuration of the structure modulation that haven't been seen before. The structure modulation that contributes to a satellite spot does not occupy entirely an area surrounded by an enclosed boundary, but varies continuously in space and forms an enclosed-contour-line 'field' so that the diffraction intensity of the satellite can be recovered from immediately adjacent area at a slightly different sample angle. Map series of satellite B and C illustrate the formation of a new ring almost at the same point where the first ring appeared about 0.4 degree apart, suggesting a vortex of the structure modulation. The size of the ring can be as small as $3\ \mu\text{m}$, and the ring size change can also be achieved by applying external pressure at a fixed sample angle. Further studies are needed to determine the origin of the ring-like domain, e.g. from lattice defect or from energy landscape of the system.

*The use of the Advanced Photon Source was supported by U.S. Department of Energy, the Office of Science and the Office of Basic Energy Sciences under the contract DE-AC02-06CH11357.6

[1] M. Subramanian *et al*, Science **239** (1988), p. 1015.

[2] J. Slezak *et al*, PNAS 105 (2008), p. 3203.

[3] J. Libera, Z. Cai *et al*, Rev. Sci. Instrum. **73** (2002), p. 1506.

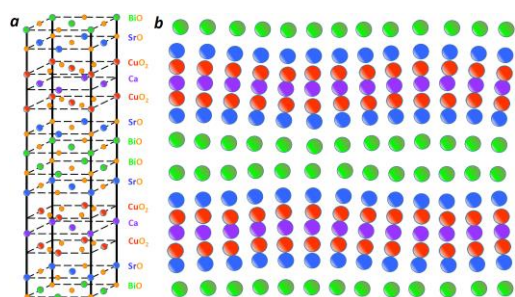


Fig 1. (a) Bi-2212 unit cell with $a = 5.413\text{\AA}$, $b = 5.408\text{\AA}$ and $c = 30.871\text{\AA}$. (b) Schematic view along a axis of the unit cell, displaying representative displacements of all metal cations with a modulation wavelength of 26\AA , or 4.8 unit cell along b axis.

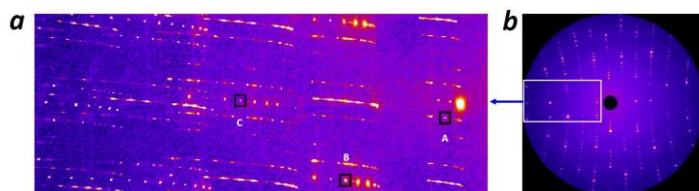


Fig 2. (b) Bi-2212 diffraction pattern obtained using a Mar-165 detector with the incident beam

normal to the sample along c axis. (a) Diffraction pattern within the highlighted detection area of (b) obtained using a Pilatus 100K. The sample was rotating around b axis within a range of 40 degrees during exposure.

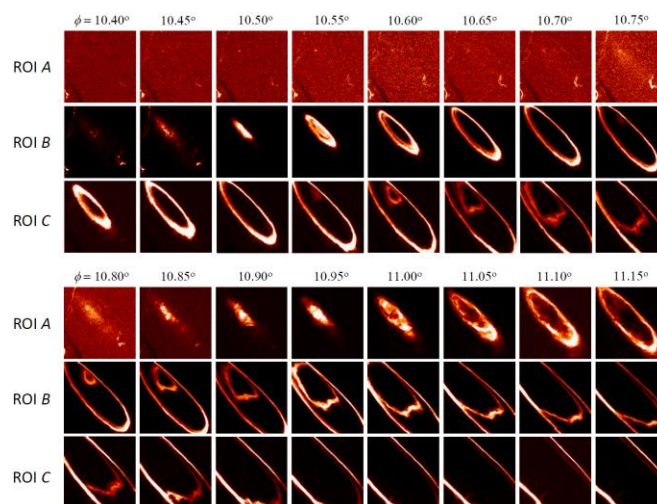


Fig 3. Area maps of the Bi-2212 with the integrated intensities of the ROIs A, B and C as a function of the ϕ angle from 10.40° to 11.15° . Each map covers an area of $100\ \mu\text{m} \times 100\ \mu\text{m}$.