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Crystal Structure Determination of Superconductors and Related Compounds by Combining High-Resolution Electron Microscopy and Electron Diffraction

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High-temperature superconducting compounds generally consist of very small crystalline grains. The two-stage image processing technique [1] based on the combination of high-resolution electron microscopy and electron diffraction is effective to determine the crystal structure for materials of such kind. In the first stage the high-resolution electron microscope image taken at an arbitrary defocus condition is transformed into the structure image by image deconvolution [2]. In general not all atoms can be seen in such obtained structure image, because the image resolution is limited by the resolution of the electron microscope. In the second stage the electron diffraction data is introduced to enhance the image resolution to the diffraction resolution limit by means of the phase extension technique using the direct method developed in X-ray crystallography [3]. Usually all atoms are resolved in the final projected potential map (PPM).

A series of crystal structures of high-temperature superconductors and related compounds were determined by this method [4-9]. Recently, the crystal structure of $(Y_{0.6}Ca_{0.4})(SrBa)(Cu_{2.5}B_{0.5})O_{7-\delta}$ has been studied. The electron diffraction patterns and images were taken with JEM-200CX and JEM-2010 electron microscopes, respectively. The diffraction intensities were collected with a slow scan CCD camera in a Tacnai F20 electron microscope.

Fig. 1 shows the electron diffraction patterns of [001] and [010] zone axes. It was determined that the crystal belongs to the orthorhombic system and lattice parameters are a = 3.85 Å, b = 3.86 Å and c = 11.5 Å. It is reasonable to recognize that the crystal structure is isomorphous to that of $YBa_2Cu_3O_{7-\delta}$. The present work is aiming at confirming the assumption that boron atoms substitute for those copper atoms, which are located at the Cu-O chain positions. Fig. 2 shows the [010] image corresponding to Fig. 1b. Fig. 3a shows the Fourier filtered image obtained from a thin area in Fig. 2. The rectangle indicates the projected unit cell. The symmetry average image obtained from Fig. 3a is given in Fig. 3b. Fig. 3c is the structure image obtained from Fig. 3b by maximum entropy image deconvolution with the defocus value –650 Å [10], where dark dots represent atoms, but not all atoms can be seen. For enhancing the image resolution, the phase extension in combination with diffraction intensity correction [11] was performed. In order to determine the position of boron atoms, two structure models were set up in the second cycle of diffraction intensity correction. In one of models boron atoms distribute randomly in all Cu positions, while in the other they are located at Cu-O chain positions. It turns out that the later model is correct. The determined PPM is given in Fig. 3d.

References

[1] F.H. Li, Proc. International Congress on Electron Microscopy, Paris (1994) Vol. 1, 481.
[2] F.S. Han et al., Acta Cryst. A, 42 (1986) 353.
[3] H.F. Fan et al., Ultramicroscopy, 36 (1991) 361.
[4] Z.Q. Fu et al., Ultramicroscopy, 54 (1994) 229.
[5] B. Lu et al., Ultramicroscopy, 70, (1997) 13.
[6] J. Liu et al., Materials Transaction, JIM, 39 (1998) 920.
[7] H. Jiang and F.H. Li, Micron, 30 (1999) 417.
[8] H. Jiang and F.H. Li, Proc. 8th Beijing International Conference on Instrumental Analysis (1999) A97.
[9] J. Liu et al., Acta Cryst. A, 57 (2001) 540.
[10] J.J. Hu, and F.H. Li, Ultramicroscopy, 35 (1991) 339.
[11] D.X. Huang et al., Acta Cryst. A, 52 (1996) 152.
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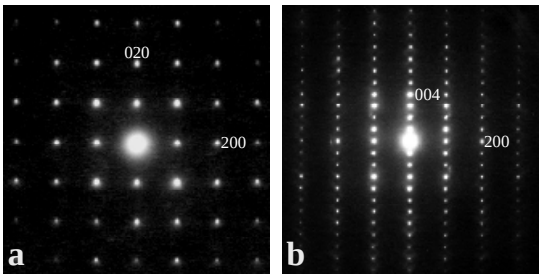

Fig. 1. Diffraction patterns of $(Y_{0.6}Ca_{0.4})(SrBa)(Cu_{2.5}B_{0.5})O_{7-}$

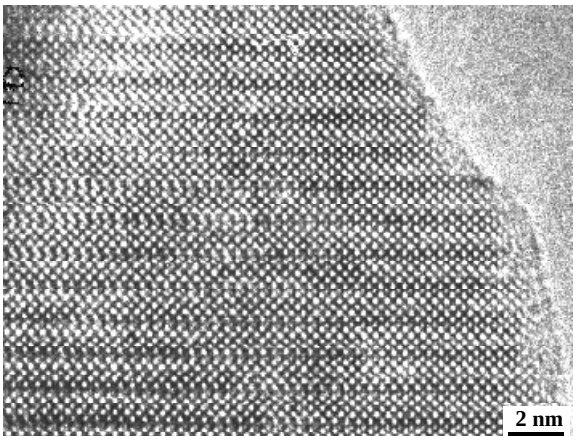

Fig. 2. [010] image corresponding to Fig 1b.

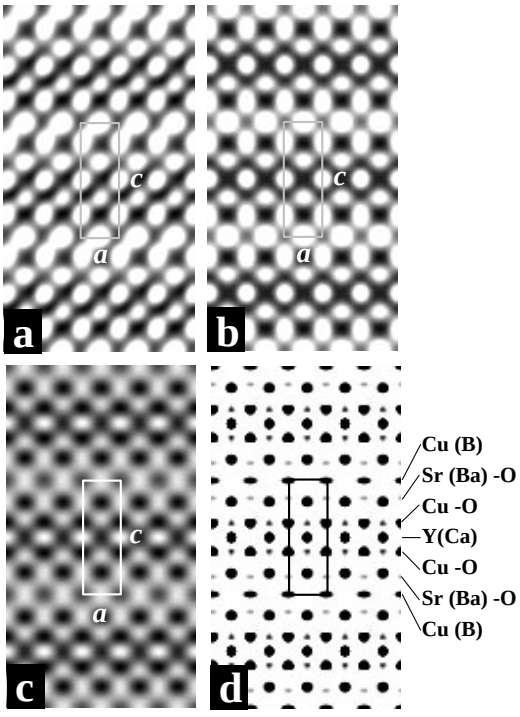

Fig. 3. (a) Fourier filtered image obtained from Fig. 2, (b) symmetry average image obtained from (a), (c) deconvoluted image obtained from (b) and (d) PPM obtained from (c).

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