Stripe structure of the CuO_2 plane in $Bi_2Sr_2CaCu_2O_{8+y}$ by anomalous x-ray diffraction

A. Bianconi and M. Lusignoli
Dipartimento di Fisica, Università di Roma ''La Sapienza,'' P. A. Moro 2, 00185 Roma, Italy

N. L. Saini
Istituto Nazionale di Fisica Nucleare, Dipartimento di Fisica, Piazza A. Moro 2, 00185 Roma, Italy

P. Bordet
Laboratoire de Crystallographie, Centre National de la Recherche Scientifique, Avenue des Martyrs, F-38043 Grenoble, France

Å. Kvick
European Synchrotron Radiation Facility, B.P. 220, F-38043 Grenoble, France

P. G. Radaelli
Institut Laue Langevin, Avenue des Martyrs, F-38043 Grenoble, France
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The modulated structure of the CuO_2 plane in $Bi_2Sr_2CaCu_2O_{8+y}$ superconductor has been solved by Cu K-edge anomalous x-ray diffraction. The displacement of Cu ions along the **c** axis shows the largest amplitude and anharmonic character. The results show flat stripes of CuO_2 lattice of width $L=15.5$ Å with long Cu-Bi distances that are separated by stripes of bent CuO_2 plane and shorter Cu-Bi distances. In each CuO_2 bilayer the flat stripes in a first layer are close to the bent stripes in the second layer, suppressing the electronic coupling between the two adjacent planes. [S0163-1829(96)03929-X]

I. INTRODUCTION

In most of the high-T_c superconductors the local structure of the CuO_2 lattice deviates from the average crystallographic structure.[1] Models for mechanisms of pairing in high-T_c superconductors based on the coexistence of two components have been proposed[2–6] where a first component is made of a gas of itinerant charges while the second one is made of relatively localized charges. Recently a model has been proposed[7] where the two components are spatially separated in a superlattice of undistorted stripes (U stripes) alternated with distorted stripes (D stripes). In this model the superconducting phase coexists with a one-dimensional anharmonic modulation of the CuO_2 plane.

There is growing evidence that an incommensurate modulation with superstructure vector of the type $\mathbf{q}_s=\beta\mathbf{b}^*+(1/\gamma)\mathbf{c}^*$ (in the orthorhombic notation where $b^*=1/(\sqrt{2}d)$ and d is the average Cu-Cu distance) is a common feature of optimally doped cuprate superconductors. It has been observed in $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi2212) with $\beta\sim0.21$ and $\gamma=1$;[8,9] in $Bi_2Sr_2CuO_{6+\delta}$ (Bi2201) with $\beta\sim0.21$ and γ varying from sample to sample in the range 1.6–3;[10–12] in $La_2CuO_{4.1}$ (LCO) with $\beta=0.22$ and $\gamma=3$ (Ref. 13) or $\beta=0.2$ and $\gamma=3$;[14] in $Tl_2Ba_2CaCu_2O_8$ (Tl2212) (Ref. 15) and in $Tl_2Ba_2Ca_2Cu_3O_{10}$ (Tl2223),[16] with $\beta\sim0.2$; and in $La_{2-x}Sr_xCu_2O_4$ (LSCO), for $x\sim0.075$ with $\beta\sim0.16$ and $\gamma\sim3.5$.[17] Recently a similar superstructure has been seen for $x=0.1$, $x=0.15$ and, much more weakly, in the overdoped sample $x=0.2$.[18] The **c**-axis period changes from one system to another and it can be assigned to different staging of dopants; on the contrary the in-plane (**b**-axis direction) modulation seems to be a common feature. However, the satellite reflections are often weak and diffused, indicating ordering over small domains, and better resolved at temperatures lower than 200–100 K. The best-characterized situation, in this respect, is that of bismuth-based superconductors,[19] in which the modulation gives rise to sharp and intense satellite peaks up to high temperatures.

The modulated structure of the Bi2212 system has been studied by several groups using electron, x-ray, and neutron-diffraction techniques.[8,9,19–27] The solution of the anharmonic incommensurate superstructure of Bi2212 is at the limit of the capabilities of diffraction methods, due to the large number of parameters needed in the structural refinement. Electron diffraction[12] has pointed out that the main lattice modulation involves the BiO layers. Bi L_3-edge anomalous diffraction has shown the distribution of Bi atoms over Ca and Sr sites.[24] The BiO rocksalt layers form blocks of undistorted rocksalt lattice (U blocks) alternated by blocks of stretched BiO lattice (D blocks) in the **b** direction. The latter are characterized by (1) low Bi density, (2) variation of the BiO coordination geometry, (3) presence of interstitial oxygen, and (4) deviations from the stoichiometry. Although various experiments[8,9,23,25–27] report modulation of the Cu sublattice mainly along the **c** axis, the results disagree on the amplitude and anharmonic character of the modulation.

In the present paper we report a study of the modulation of the CuO_2 plane in Bi2212 by x-ray anomalous scattering, taking advantage of tunability of synchrotron radiation.[28] Due to the strong variation of the copper scattering factor near the Cu K threshold, the technique provides direct and selective information on the sublattice of copper atoms. Furthermore, the data were collected in a selected region of the reciprocal space with Bragg reflections of Miller indices $\ell \gg h\sim k$. In this case, the diffracted intensities are mainly sensitive to displacements along the **c** axis. As a result the z

coordinate of the Cu atom in the structure and its modulation were measured accurately. The results indicate that the Cu position is anharmonically modulated in the out-of-plane direction with strong contribution from the second harmonic. The one-dimensional (1D) modulation results in two different stripes of distorted (bent Cu plane) and undistorted (flat Cu plane) Cu lattice running along the **a** axis. In this heterostructure the distorted stripes would act as potential barrier between the stripes formed by undistorted lattice.

II. EXPERIMENTAL AND DATA ANALYSIS

The experiment has been performed at the European Synchrotron Radiation Facility (ESRF, Grenoble, France) on the wiggler beam line ID 11-BL2. The measurements were made on a well-characterized single crystal of $0.75\times0.75\times0.015$ mm^3, with $T_c=84$ K, grown by the traveling-solvent-floating-zone method.

The sample was given 24° oscillation around the **b** axis, the diffraction images were recorded on a 35×43-cm imaging plate detector. The high x-ray beam intensity allowed us to record the intensity variation of first- and second-order satellite spots up to high momentum transfer along the **c** axis [$\sin(\theta)/\lambda<0.41$ Å^{-1}] in the range of h, k, l, and m in reciprocal space: $10<\ell<23$, $h=0,\pm1,\pm2$ and $k=0,\pm1$ $m=0,\pm1$, ±2. Out of 81 indexed spots, 37 first-order and 23 second-order superstructure peaks were identified. This geometry has permitted a precise evaluation of the amplitude and anharmonic content of the modulation of the z component of Cu atoms. The diffraction patterns have been recorded at 28 different temperatures in the range 30–300 K. Independent diffraction patterns at each temperature have been collected with short and long exposure times to increase the counting rate for the weak spots, taking under control saturation effects, and for reproducibility.

For the anomalous diffraction two patterns were recorded sequentially at two wavelengths chosen to have a large variation in the real part of the Cu anomalous scattering factor ($\Delta f'=6.2$ electrons) with no variation in the imaginary part f'': $\lambda_1=1.3788$ Å, at the rising edge of the Cu K threshold, and $\lambda_2=1.4086$ Å, about 200-eV below the edge. The difference of the structure factors $\Delta F=F(\lambda_2)-F(\lambda_1)$ at the two wavelengths is equal to the variation of the Cu contribution only, as the scattering factors of the other atoms are almost constant in this range ($\Delta f<0.1$ electrons). The peak indexing was made using the four-dimensional approach for incommensurate 1D modulations,[29] considering that for each Bragg reflection the diffraction vector **H** can be written as $\mathbf{H}=h\mathbf{a}^*+k\mathbf{b}^*+1\mathbf{c}^*+m\mathbf{q_s}$ [$\mathbf{q_s}=\beta\mathbf{b}^*+(1/\gamma)\mathbf{c}^*$, $\gamma=1$]. The superstructure period (given by $1/\beta$) has been found to be temperature independent ($\lambda_p=4.73\pm0.01$). The data were corrected for the Lorentz factor, for polarization, and for absorption. The structure factor was calculated in the four-dimensional approach using the $N^{Bbmb}_{1,1,1}$ superspace group and isotropic thermal factors. In this situation, having an almost centrosymmetric structure, the measurable quantity $\delta_F=|F(\lambda_2)|-|F(\lambda_1)|$ may be assumed equal to the variation of the Cu contribution (real part) only.[28] It is given, for permitted reflections, by the formula

$$\delta_F=8\Delta f'_{\mathrm{Cu}}e^{-B_{\mathrm{Cu}}(\sin\theta/\lambda)^2}\int_0^1\cos\left(2\pi h x_{\mathrm{Cu}}(t)+\pi\frac{k}{2}\right)\times\cos[2\pi(k+m\beta)y_{\mathrm{Cu}}(t)]\times\cos\left(2\pi(l+m)z_{\mathrm{Cu}}(t)-\pi\frac{k}{2}\right)\times\cos(2\pi mt)\,dt.$$

The particular symmetry of the metal sites requires $z(t)=z(-t)$.[8] Considering two harmonics, we have refined

TABLE I. The z coordinates and modulation parameters for the metal atoms determined in this work considering $z(t)=c_0+c_1\cos(2\pi t)+c_2\cos(4\pi t)$, where $t=y/\lambda_p$ and λ_p the modulation period in the **b**-axis direction. The results of this work are compared with other diffraction works (Refs. 8, 9, 10, and 18). [Kan and Moss (Ref. 18) consider a sinusoidal component in the modulations; Petricek *et al.* (Ref. 25) include this component only for Bi atoms but it is very small.]

		This work ($T<100$ K)	Yamamoto *et al.*	Petricek *et al.*	Kan-Moss	Beskrownyi *et al.*
Cu	$c0$	0.1953(2)	0.1955(3)	0.1965(1)	0.1962(2)	0.1974(2)
	$c1$	−0.0090(2)	−0.0102(4)	−0.0086(3)	0.0094(5)	−0.0093(4)
	$c2$	−0.0042(5)		−0.0022(3)	−0.003(1)	
Sr	$c0$	0.1409(2)	0.1401(2)	0.1408(1)	0.1395(2)	0.1416(4)
	$c1$	−0.0071(2)	−0.0091(5)	−0.0065(3)	0.0086(4)	−0.0071(6)
	$c2$	−0.0019(3)		−0.0022(6)	−0.0015(6)	
Bi	$c0$	0.05177(6)	0.0520(1)	0.0523(9)	0.0514(1)	0.0518(2)
	$c1$	−0.0062(2)	−0.0052(2)	−0.0049(3)	0.0040(5)	−0.0078(6)
	$c2$	−0.0017(4)		−0.0019(3)	−0.0058(7)	
Ca	$c0$	0.25	0.25	0.25	0.25	
	$c1$	−0.0077(5)	−0.0099(8)	−0.0080(3)	0.008(1)	−0.0104(8)
	$c2$	0		0	−0.002(2)	
c (Å)		30.651(2)	30.6490(1)	30.871(5)	30.88(2)	30.716(3)
λ_p		4.734(4)	4.721(2)	4.76(7)	4.717	4.75

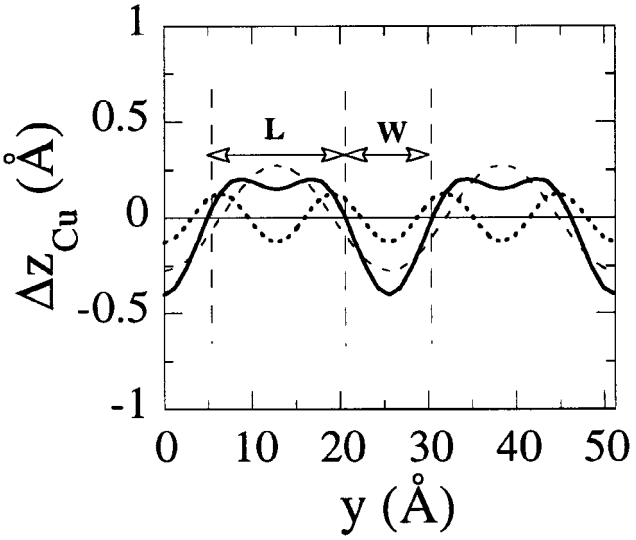

FIG. 1. Modulation along the **b** axis of the z component of the Cu ions in the CuO_2 layers of Bi2212, determined by anomalous x-ray diffraction. The dashed and dotted lines are the first and second harmonic contributions, respectively.

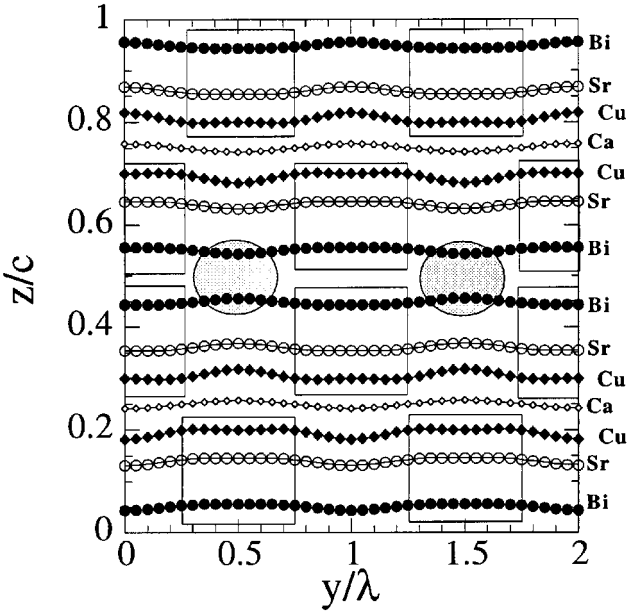

FIG. 2. The modulation of z/c (where $c=30.653\pm0.002$ Å) for the heavy atoms over two periods of the superstructure. The squares indicate sections of wires of undistorted CuO_2 stripes and BiO rocksalt blocks (U blocks). The modulations of two neighbor parallel CuO_2 planes are out of phase, so that flat stripes are surrounded by bent stripes.

the z-component values of Cu atoms independently from the structural parameters of all other atoms, fitting δ_F values with $z_{\mathrm{Cu}}(t)=c_0+c_1\cos(2\pi t)+c_2\cos(4\pi t)$ (where $t=\beta y$). The value of $\Delta f'_{\mathrm{Cu}}$, an overall scale factor, and a correction factor which account for small differences in the relative scale of the λ_1 and λ_2 data sets, were refined as part of the refinement. In general, the Fourier coefficient of $x_{\mathrm{Cu}}(t)$ and $y_{\mathrm{Cu}}(t)$ as well as the B_{Cu} thermal parameter should also be refined. As already mentioned, however, the intensity of the Bragg reflections collected during our experiments are poorly sensitive to displacements along either the **a** or **b** axes. In addition, due to the limited reciprocal space range, B_{Cu} is strongly correlated with the scale factor. Therefore, all these parameters were fixed to the values obtained by Yamamoto *et al.*[8] As expected, attempts to refine these parameters did not improve the fit. The refined values of the parameters are reported in Table I. As a self-consistency check, the agreement factor between F_{calc} and $F_{\mathrm{obs}}(\lambda_2)$ (the observed structure factor away from the edge) was minimized with respect to the z coordinates and modulation of the heavy atoms. As we see from the Table I, our results are in good agreement with the published values, in spite of the small number of reflections that were employed.

III. RESULTS AND DISCUSSION

Figure 1 shows the modulation of the Cu z component determined at low temperature in the superconducting phase ($T<T_c$). The amplitude of the first-harmonic component, $c_1=-0.009$ ($c_1=-0.276$ Å) is comparable with previous results, as shown in Table I. On the other hand, the amplitude of the second harmonic component, $c_2=-0.0042$ ($c_2=-0.128$ Å), is comparatively higher than the reported values. In fact the neutron-diffraction results[8,9] do not consider second harmonics, and the x-ray-diffraction results show lower values. The strong amplitude of the second harmonics gives rise to an anharmonic modulation with portions of Cu lattice having an almost constant z coordinate. These are separated by portions of largely bent lattice where the z coordinate differs up to 0.6 Å. This 1D anharmonic modulation results in flat stripes of Cu lattice of width $L=15.5$ Å running along the **a** axis. Therefore the Cu lattice can be described as a superlattice of undistorted stripes (U stripes) with period 25.5 Å. The U stripes are separated by the stripes of bent lattice of width $W=10$ Å (D stripes).

The modulations of copper and the other heavy atoms along the **b** axis are shown in Fig. 2. The amplitude of the modulation of the Cu plane along the **c** axis is larger than that of the other atomic species. It decreases going from Cu to Bi, and therefore cannot be simply assigned to a steric effect due to the Bi layer. The large **c**-axis displacements of the Cu plane are correlated with the strong **b**-axis modulation of the BiO layers. Figure 2 highlights the spatial distribution of the flat and bent Cu lattices. The projection of the Bi2212 crystal in the y-z plane is shown over an area of 51 Å (along the **b** axis) times 30.65 Å (along the **c** axis). The rectangles indicate the section of wires of undistorted BiO rocksalt lattice and flat CuO_2 stripes (U stripes). They are separated by distorted blocks of stretched BiO lattice and bent CuO_2 plane (D stripes).

This complex structure can be described as wires of distorted lattice, where dopants (substituted and/or interstitial atoms) are located, giving negative localized charges (acceptors) indicated as shadowed circles, and wires of undistorted lattice indicated by squares in Fig. 2. In each Cu bilayer the U stripes in a first layer are close to the D stripes in the second layer. This arrangement has interesting implications for the electronic properties of the compound: in fact the coupling between two CuO_2 planes in a bilayer is expected to split the main Cu $3d_{x^2-y^2}$–O $2p_{x,y}$ band in two bands

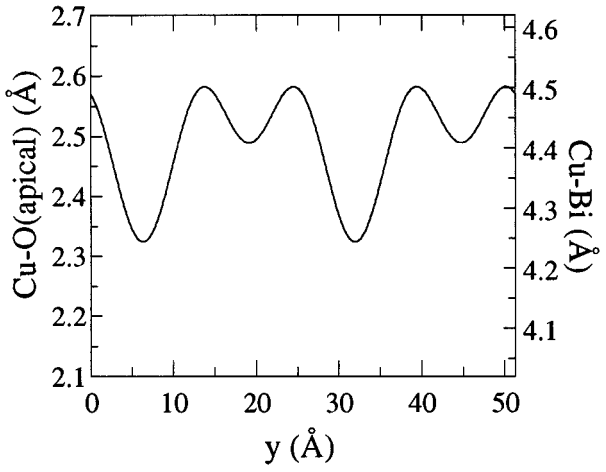

FIG. 3. The measured modulation of the Cu-Bi bonds [taken to be the same as that of the Cu-O (apical) bonds for a fixed Bi-O (apical) bond].

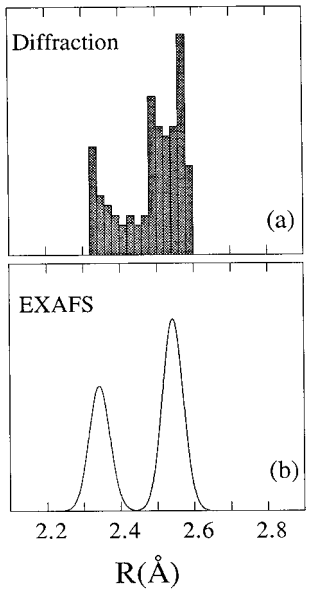

FIG. 4. The pair distribution function (PDF) of the Cu-O (apical) bonds [taken to be the same as that of the measured Cu-Bi bonds for a fixed Bi-O (apical) bond] obtained by the present diffraction work [panel (a)], compared with the PDF Cu-O (apical) bond determined by polarized Cu *K*-edge EXAFS (Ref. 33) [panel (b)].

having even and odd symmetries. However, angle-resolved photoemission has ruled out the presence of two bands.[30] This unexpected experimental result can be understood by looking at the spatial distribution of the lattice distortions in Fig. 2. The *U* stripes in the first plane are close to the *D* stripes of the second plane of the bilayer. This decreases the electron hopping between the two planes, suppressing the band splitting.

Large attention has been paid to the coexistence of two different Cu-O (apical) bonds in cuprate superconductors observed by extended x-ray-absorption fine structure (EXAFS) and assigned to dynamic fluctuations in a double-well potential for the apical oxygen[31] or to a spatial nonhomogeneity due to stripe formation.[32] The comparison of the EXAFS results and diffraction results have been the subject of considerable discussion.[1] In order to solve this open problem it should be recalled that while x-ray diffraction measures the difference between time-averaged atomic positions, EXAFS measures the instantaneous distances between pairs of atoms. Therefore the two Cu-O (apical) bonds detected in EXAFS can be assigned both to the modulation of Cu as well as of O (apical) positions. In Fig. 3 we report the modulation of the Cu-Bi distance. The main contribution to the modulation shown in Fig. 3 is due to the out-of-plane modulation of Cu; in fact, the in-plane modulation of Bi has a minor effect on the bond distances. Because the Bi-O (apical) bond length is not modulated,[8,9] this modulation is expected to be the same as that of the Cu-O (apical) bond. In the present aperiodic structure the CuO_5 pyramids take an infinite number of different conformations that can be described by the statistical distribution of interatomic distances. The pair distribution function (PDF) for the Cu-O pairs extracted from our refined structure is shown in Fig. 4. The results show a distribution of the Cu-O (apical) bond from 2.3 to 2.6 Å. The results are compared with the Cu-O (apical) PDF of Bi2212 determined by extended x-ray-absorption fine structure.[33] In spite of the intrinsic differences of the two techniques the results are in good agreement with each other. In conclusion the presence of the anomalous short Cu-O (apical) bond (0.2 Å shorter) observed in EXAFS is clearly due to the modulation of the Cu *z* coordinate. The distribution of the Cu-O (apical) distance measured by EXAFS can be assigned to the anharmonic displacement of Cu.

In summary, the modulation of the Cu plane has been determined by anomalous diffraction. The contribution of the second harmonic is such that the resulting structure appears to be made of flat and bent stripes. The electronic structure of the two types of stripes is expected to be different due to the large amplitude of the distortions. In this quantum heterostructure the distorted stripes (bent CuO_2 plane) would act as a potential barrier between the stripes formed by undistorted lattice (flat CuO_2 plane). Therefore this work supports the proposed models considering two electronic components for the mechanism of high-T_c superconductivity.[1–6]

One of these electronic components is expected to be confined in the superlattice of quantum wires formed by the plurality of *U* stripes. The measured width of *U* stripes $L=15.5$ Å is such that the bottom of the second and third subbands of the superlattice are close to the Fermi energy. This tuning of the Fermi level has been reported to be a possible mechanism for amplification of the superconducting transition temperature.[7,34]

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