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Low temperature *Pmmm* and $C2/m$ phases in $Sr_2CuO_{3+\delta}$ high temperature superconductor*

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A new low temperature *Pmmm* (120 K) phase was found in high temperature superconductor $Sr_2CuO_{3+\delta}$, which was indicated as a pure electronic phase by resonant x-ray diffraction at Cu *K*-edge. As shown by x-ray absorption fine structure (EXAFS) and x-ray absorption near edge structure (XANES) at Cu *K*-edge, the strong charge density redistribution and local lattice fluctuations around Cu site at the onset of phase transition were due to the occurrence of superconductive coherence, the redistribution and fluctuation finished at T_c. Finally, the electron–lattice interaction was mainly elaborated to understand the superconductivity of $Sr_2CuO_{3+\delta}$.

Keywords: resonant x-ray diffraction, x-ray absorption fine structure, modulated phase, superconductivity

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1. Introduction

Despite the discovery of high-temperature superconductors more than two decades ago, the relationship between their superconductivity and their microstructure still remains a mystery.[1–3] With growing evidence for the existence of different kinds of microstructures in the high T_c superconducting cuprates, complex microstructures and their evolution were considered to be highly correlated with the superconductivity of the cuprates.[4–11] The rich modulated phases in the cuprates with either oxygen interstitials or vacancies were a case in point, the most striking feature was the appearance of charge modulation generally coexisting and playing a suppressive role in superconductivity of doped La-214 cuprates, such as $La_{2-x-y}Nd_ySr_xCuO_4$, $La_{2-x}Ba_xCuO_4$, and $La_{1.8-x}Eu_{0.2}Sr_xCuO_4$. The competitive characteristics between superconductivity and charge order have been reported in $YBa_2Cu_3O_y$. The charge modulation seemed to play a key role in affecting the superconductivity of cuprates. Furthermore, the anomalous lattice response to the onset of charge modulation and T_c was also reported to be important in explaining the superconductivity of cuprates. Several recent works on doped La-214 cuprate superconductors revealed the intimate relationship among the charge modulation, local lattice fluctuation, and high-T_c superconductivity.[12–20]

$Sr_2CuO_{3+\delta}$ is a single-CuO_2-layer cuprate with similar structure to La_2CuO_4 (Fig. 1(a)). With La replaced by Sr which contributed one electron less to the system, a significant amount of oxygen vacancies were introduced to stabilize the material, which could only be grown under high pressure. Surprisingly, even with significant defects in the system, when compared with La_2CuO_4, $Sr_2CuO_{3+\delta}$ showed much higher T_c (maximum reported was 95 K). In the past few years, great efforts were made to reveal the evolution of the superstructures and its relationship with the superconductivity in $Sr_2CuO_{3+\delta}$. Liu *et al.* reported the coexistence of $C2/m$ and *Fmmm* phases in as-synthesized $Sr_2CuO_{3+\delta}$.[21,22] They also found that the $C2/m$ phase transited to the *Cmmm* and *Pmmm* phases when the annealing temperature decreased to 150 ℃ and 250 ℃, and the corresponding T_c shifted from 75 K to 89 K and 95 K, respectively. Due to no change of the *Fmmm* phase with the shift of T_c, the *Fmmm* phase was believed to have no correlation with the superconductivity. However, Wang *et al.* claimed that the *Fmmm* phase was superconductivity-related because only the *Fmmm* phase was observed in $Sr_2CuO_{3+\delta}$ sample annealed at different temperatures all the time, and moreover, its coherent length varied with the annealing temperature.[23] Since these studies were all carried out at room temperature, the phase evolution at low temperature is considered more important to understand the superconductivity of $Sr_2CuO_{3+\delta}$.

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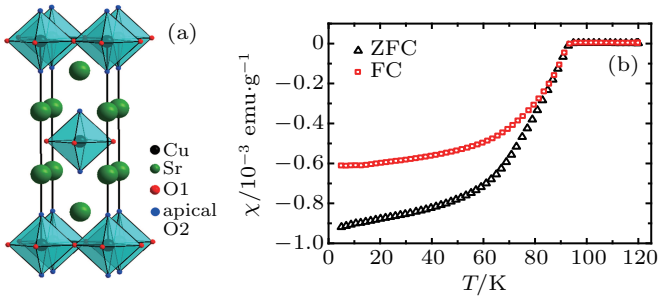


Fig. 1. (a) A sketch of $Sr_2CuO_{3+\delta}$ unit cell in space group $I4/mmm$. This was used as the average structure in the following structural refinement. (b) Temperature dependence of the DC magnetic susceptibility in both the zero-field-cooling and field-cooling modes for $Sr_2CuO_{3+\delta}$, revealing the superconducting transition with $T_c \sim 95$ K. (Ref. [18] copyright).

2. Methods

To follow up the phase evolution of $Sr_2CuO_{3+\delta}$ at low temperature, systematic temperature-dependent x-ray diffraction (XRD) and x-ray absorption fine structure (XAFS) studies were carried out at Cu K edge. The results showed that two low-temperature phases, *Pmmm* and $C2/m$, emerged around 120 K and 60 K and the former may highly correlated with the superconductivity because it appeared around the superconducting critical temperature.

$Sr_2CuO_{3+\delta}$ polycrystalline specimens with $\delta = 0.4$ were fabricated by using high temperature high pressure solid state synthesis technique. The temperature-dependent DC magnetic susceptibility curve showed a superconducting transition at 95 K. Details of the sample preparation and magnetic susceptibility measurement have been reported in Ref. [24]. The temperature-dependent synchrotron radiation x-ray diffraction and extended x-ray absorption fine structure (EXAFS) at Cu K edge were carried out with beamlines of 14B1 and 14W1 at the Shanghai Synchrotron Radiation Facility. The photon energy (x-ray wavelength) was calibrated with a Cu foil.

3. Results and discussion

As shown in Fig. 2, the XRD patterns of $Sr_2CuO_{3+\delta}$ at different temperatures (13 K, 35 K, 50 K, 65 K, 70 K, 75 K, 80 K, 90 K, 95 K, 100 K, 115 K, 120 K, 130 K, 160 K, 190 K, 230 K, 300 K) converted from the Debye rings were recorded by a two-dimensional x-ray detector. The data was processed with FIT2D software. Figures 2(a) and 2(b) show the collected XRD patterns at 8.500 keV (far below the Cu K edge) and 8.990 keV (in the Cu K edge range), respectively. Details of the Debye rings can be found in the supplementary materials. Except for the diffraction peaks from the reported *Fmmm* phases,[21,22] three new diffraction peaks were found when the temperature decreased to around 120 K and 60 K. This result indicates (through indexing) that two new modulated phases developed at low temperature. To determine the symmetry of the low temperature phases, indexing of the diffraction peaks was carried out with results listed in Table 1. The space groups (parameters of unit cell) determined from the indexing are *Pmmm* ($a_p = 5.883$ Å, $b_p = 16.020$ Å, $c_p = 6.080$ Å) and $C2/m$ ($a_c = 16.800$ Å, $b_c = 6.660$ Å, $c_c = 9.881$ Å, $\beta = 119.5°$), respectively. The *Pmmm* phase appeared at around 120 K, while the $C2/m$ phase came out from about 60 K. The volume of the *Pmmm* phase modulation structure was $\sqrt{2}a_t \times 3\sqrt{2}a_t \times c_t/2$, while the volume of the $C2/m$ phase modulation structure was $3\sqrt{2}a_t \times c_t/2 \times 2\sqrt{2}a_t$. Here, a_t and c_t are the lattice constants in the unit cell. At the same time, the diffraction intensity of the *Pmmm* phase appearing at 120 K was stronger than that of the $C2/m$ phase at 60 K. More importantly, we found that the diffraction peaks (5.48 Å and 3.26 Å) from the low temperature *Pmmm* phase showed up only in Cu K absorption edge range (anomalous scattering), while diffraction peaks from other phases showed no obvious intensity change with photon energy. This indicates that the modulation in the low temperature *Pmmm* phase was purely an electronic behavior of the Cu valence state, while modulation in other phases should come from the displacement of ions or ordering of oxygen vacancy. In other words, the modulation in the low temperature *Pmmm* phase might come from Cu charge ordering, such as charge density wave of Cu 3d electron. This result coincided with the recent reports on La-214 cuprates that a charge density wave appeared in the proximity of T_c.[25–30]

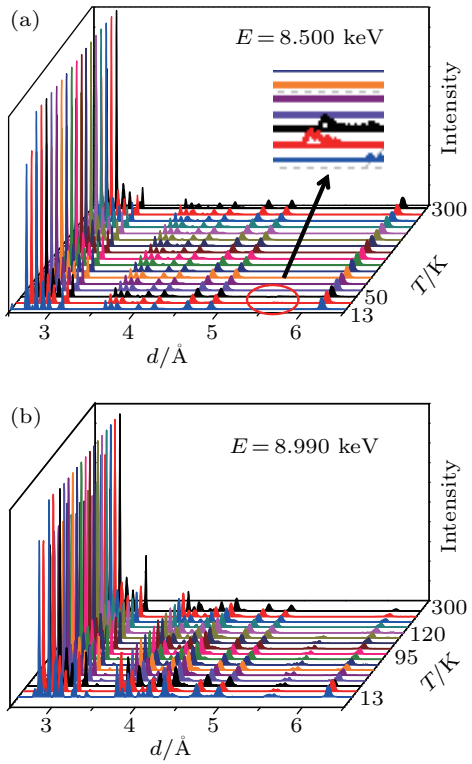


Fig. 2. X-ray diffraction result of $Sr_2CuO_{3+\delta}$ as function of temperature and incident x-ray energy. (a) and (b) The x-ray diffraction patterns obtained at varied temperatures with x-ray energy of (a) 8.500 keV and (b) 8.990 keV. Some modulated structures emerge in the low temperature region.

Coincidently, the local lattice fluctuation on Cu site has been reported at the onset of charge density wave.[26–30] The

coincidence indicates that the electron–lattice interaction is an important element for superconductivity of doped cuprates.

Table 1. The modulated phases of $Sr_2CuO_{3+\delta}$ superconducting specimen obtained from indexing the diffraction peaks. The lattice parameters for tetragonal structure $Sr_2CuO_{3+\delta}$ are $a_t = b_t = 3.785$ Å and $c_t = 12.425$ Å.

T/K	Modulated phases	
	Space group	Lattice constants
300	$Fmmm$	$a = b = 5\sqrt{2}a_t,\ c = c_t$
300	$Pmmm$	$a = b = 4\sqrt{2}a_t,\ c = c_t$
120	$Pmmm$	$a = \sqrt{2}a_t,\ b \approx 3\sqrt{2}a_t,\ c = c_t/2$
60	$C2/m$	$a = 3\sqrt{2}a_t,\ b \approx c_t/2,$ $c = 2\sqrt{2}a_t,\ \beta = 119.5°$

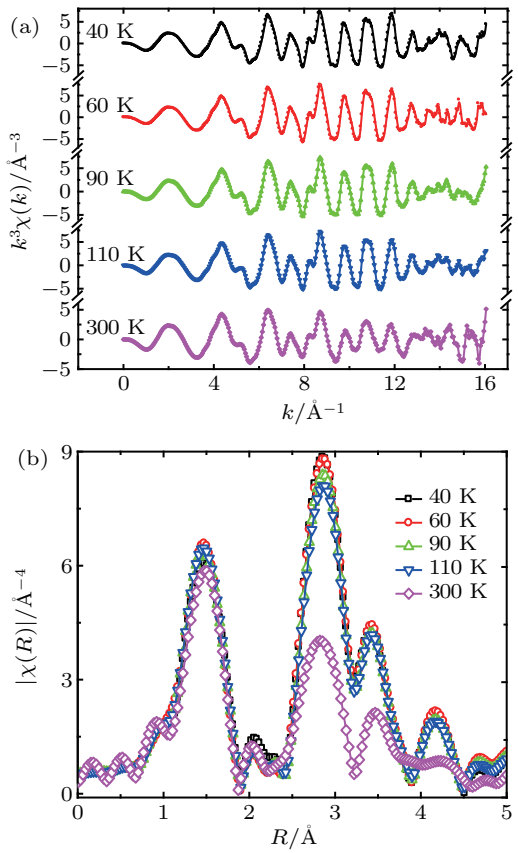


Fig. 3. X-ray absorption fine structure spectroscopy result of $Sr_2CuO_{3+\delta}$ as a function of temperature. The temperature dependence of Cu K-edge (a) EXAFS oscillations in moment space (weighted by k^3) and (b) FT magnitude function in real space for $Sr_2CuO_{3+\delta}$.

To verify if this local lattice fluctuation around Cu site correlated with the modulation of Cu valence state in $Sr_2CuO_{3+\delta}$, temperature-dependent EXAFS measurement at Cu K-edge was performed. Figure 3(a) shows five representative k^3-weighted EXAFS oscillations of $Sr_2CuO_{3+\delta}$ at the temperatures of 300 K, 115 K, 90 K, 60 K, and 40 K; and the corresponding radial-structure-functions (Fourier transform of the EXAFS oscillations) are shown in Fig. 3(b). Details for the EXAFS data processing can be found in the supplementary materials. The first three peaks in Fig. 3(b) can be attributed to the Cu–O, Cu–Sr, and Cu–O–Cu coordinations, respectively. The location of the peak was determined by the coordination distance, and the intensity of the peak was determined by the coupling strength of the coordination bonds. According to the coordination structure around Cu atom, the O atoms for CuO_6 octahedron were nearest to the Cu atom, the first peak in Fig. 3(b) was due to the Cu–O coordination. The second peak was due to the Cu–Sr coordination. The third peak came from the Cu–O–Cu coordination.

In the La-214 cuprates, for example, $La_{2-x}Sr_xCuO_4$, $La_{1.48}Sr_{0.12}Nd_{0.4}CuO_4$, $La_{1.875}Ba_{0.125}CuO_4$, XAFS studies revealed an upturn on the Debye–Waller factor around T_c.[19,25,26] This upturn was interpreted as the local lattice instability.[19,25,26] Similar behavior was also observed in $Sr_2CuO_{3+\delta}$. As shown in Fig. 4(a), the Debye–Waller factor of the Cu–O coordination, as the function of the temperature, was denoted as the mean-square relative displacement of O around Cu. It was believed to be relevant with low temperature modulated phases in doped 214 cuprates.[19,20,31–34] As shown in Fig. 4(a), the anomalous upturns appeared around 115 K and 60 K as the temperature decreased. Combined with the anomalous XRD result, it is believed that the upturn is caused by the critical fluctuation of the phase transition.[33,34] The Debye–Waller factor was a measure of the mean-square relative Cu–O bond.[33,34] At the critical temperature of phase transition, nucleation of new phase caused lattice fluctuation and a sudden increase of Debye–Waller factor. In order to observe the instability clearly, we performed fitting for the temperature dependent Debye–Waller factor of Cu–O bond of $Sr_2CuO_{3+\delta}$ by using the Debye model. The fitting curve is shown as the circles in Fig. 4(a). The clear difference between experimental result and fitting can be seen, which was attributed to the occurrence of local lattice instability below 115 K. However, as soon as the phase transition finished, the Debye–Waller factor returned to the normal value, it might be attributed to the cooper pairing.

The anomalous tendency was also observed in x-ray absorption near edge structure (XANES). The upper inset of Fig. 4 (b) shows a typical Cu K-edge XANES of $Sr_2CuO_{3+\delta}$ ($\delta = 0.4$) at 20 K and its best fitting result. For CuO_6 octahedral coordination, there are four small peaks in XANES marked with A1, A2, B1, and B2. A1 and B1 originated from the $1s \rightarrow 4p_z$ and $1s \rightarrow 4p_{x,y}$ transitions, which came from the multiple scattering of ejected photoelectrons by oxygen atoms in apical and in-plane sites, respectively. A2 was caused by the scattering of photoelectrons from Sr atoms in the rock-salt layer, while B2 included multiple scattering contribution. Factor $R = (b_1 - a_1)/(b_1 + a_1)$, where a_1 and b_1 are the intensitie of A1 and B1 peaks, could be used to represent the local lattice fluctuation and electronic density inhomogeneity around Cu atoms.[33,34] The temperature dependence of R for $Sr_2CuO_{3+\delta}$ is shown in Fig. 4(b), the value increased monotonically with the decrease of temperature. This indicates that the charges transferred gradually from the apical site to in-plane site. This result was in accordance with the

charge transfer picture of other layered cuprates. Similar to the evolution of Debye–Waller factor of Cu–O bond, the R factor also showed two anomalous drops at about 60 K and 115 K, indicating significant local lattice distortion. Due to the strong coupling interaction between Cu and O in CuO_2 plane, the charge density of Cu ions can be easily modulated by local lattice instability. The inhomogeneous charge density distribution may be the origin of the *Pmmm* modulated phase at low temperature. Furthermore, exact coincidences between the start of the first glitch (∼115 K) and the formation of the low temperature *Pmmm* phase, between the endpoint (∼95 K) and the superconducting transition temperature T_c were found. The above results suggested that the *Pmmm* phase may be the superconducting related phase and electron–lattice interaction might play a key role in affecting the superconductivity of $Sr_2CuO_{3+\delta}$.

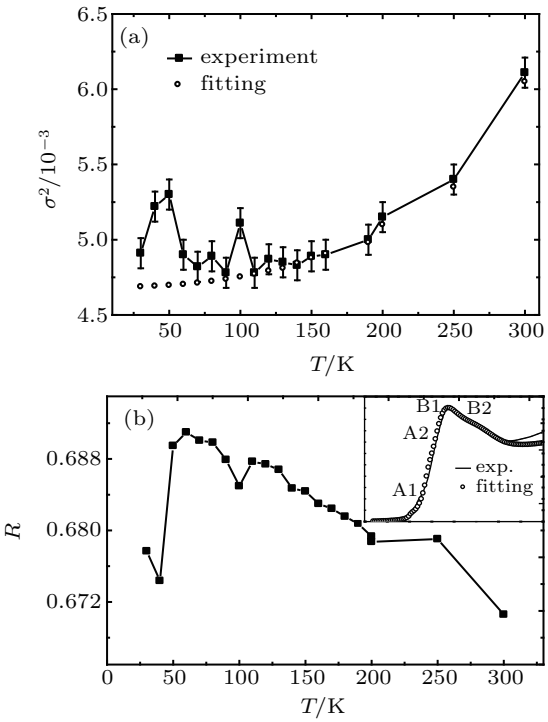


Fig. 4. (a) Temperature dependence of Debye–Waller factor of Cu–O coordination in $Sr_2CuO_{3+\delta}$, showing anomalous change around 115 K and 60 K. The circles in the Fig. 4(a) are the fitting result using Debye model. (b) Temperature evolution of the peak intensity ratio $R = (b_1 - a_1)/(a_1 + b_1)$ obtained from the XANES spectra of $Sr_2CuO_{3+\delta}$, also showing clear changes around 115 K and 60 K. The inset is a typical Cu K-edge XANES of $Sr_2CuO_{3+\delta}$ ($\delta = 0.4$) at 20 K and its best fitting result.

4. Conclusion

In summary, we reported two low temperature phases *Pmmm* (120 K) and $C2/m$ (60 K) in layered cuprate high temperature superconductor $Sr_2CuO_{3+\delta}$ ($\delta = 0.4$). The *Pmmm* phase was suggested highly correlated with the superconductivity. Strong charge density redistribution and local lattice fluctuations around Cu site occured with the decrease of temperature. The coincidence between the behavior of local lattice instability and *Pmmm* modulated phase indicated that the electron–lattice interaction could never be neglected for understanding the superconductivity of $Sr_2CuO_{3+\delta}$.

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