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Abstract
The effect of Cu doping on the stripe-ordered state in La$_{1.67}$Sr$_{0.33}$NiO$_4$ has been studied by means of electrical resistivity and magnetic measurements and transmission electron microscopy observation. The investigations reveal that Cu doping at Ni sites does not change the modulation pattern of the charge ordering, but clearly reduces the charge-ordered-transition and spin-ordering temperatures. Moreover, Cu on the NiO$_2$ planes tends to melt the stripe lattice and reduce the charge and spin stripe correlation lengths. These effects could arise from the cooperative action of carrier mobility enhancement, strong interplay of Cu spins with the host Ni spin–lattice system, and the disordering effect of Cu dopants.

1. Introduction

Both La$_2$CuO$_4$ and La$_2$NiO$_4$ belong to the class of charge-transfer insulators, characterized by an oxygen 2p-like valence band and a 3d-like upper Hubbard conduction band which results from the strong correlation among the 3d electrons [1]. Although the two oxides have the same crystal structure, different behaviour is manifested when hole carriers are introduced into their lattice. Introduction of a few holes into the CuO$_2$ plane induces high-temperature superconductivity in La$_{2-x}$Sr$_x$CuO$_4$, but La$_{2-x}$Sr$_x$NiO$_4$ remains insulating to very high doping levels [2]. However, charge ordering (CO) is reported to exist over a rather wide hole doping range $0.2 < p < 0.7$ in La$_{2-x}$Sr$_x$NiO$_4$ [3–9]. In fact, this kind of CO at low temperature is a real-space ordering of holes and spins in a static stripe form. Some evidence of stripe formation has also been found in the La$_2$CuO$_4$ family compounds [10–20] and YBa$_2$Cu$_3$O$_{6+\delta}$ [21–23]. These experimental facts lead to speculation that there is a relation between stripe ordering and high-T_c superconductivity.

It is well known that high-T_c superconductivity in $La_{2-x}Sr_xCuO_4$ arises from the CuO_2 plane and that the stripe ordering in $La_{2-x}Sr_xNiO_4$ is situated on the NiO_2 plane. For high-T_c superconductors, studies of substitution for the CuO_2 plane have revealed many important facts—such as the importance of the antiferromagnetic spin fluctuation. Like these studies of substitution for the CuO_2 plane, for $La_{2-x}Sr_xNiO_4$ studies of substitution for the NiO_2 plane are helpful; they enable us to infer the dynamics of ordered hole carriers, the interaction between holes and spin systems, and the nature of the stripe ordering. In particular, it has been suggested that a static stripe phase is antipathetic to superconductivity [10, 12, 24, 25] while a dynamic stripe phase or dynamic fluctuation of the stripe phase is favourable for high-T_c superconductivity [16, 24, 26–28]. Recently, searching for more evidence of stripe phases in high-T_c superconductors and investigating the relationship between the stripe phase and high-T_c superconductivity has constituted a new 'hot spot' in the high-T_c superconducting field. For $La_{2-x}Sr_xNiO_4$, by investigating the effect of Cu substitution for the NiO_2 plane it may be possible to gain some understanding of the correlation between stripe ordering and high-T_c superconductivity and, furthermore, the transition from the static stripe phase to superconductivity.

In this paper, we present systematic electrical resistivity and magnetic susceptibility measurements performed on $La_{1.67}Sr_{0.33}Ni_{1-x}Cu_xO_4$ ($0 \leqslant x \leqslant 0.3$). We found that introduction of Cu into the NiO_2 plane reduces rapidly the charge- and spin-ordering characteristic temperatures of $La_{1.67}Sr_{0.33}NiO_4$.

2. Experimental details

The polycrystalline specimens of $La_{1.67}Sr_{0.33}Ni_{1-x}Cu_xO_4$ with $x = 0$–0.3 were synthesized by conventional solid-state reaction in air, starting from high-purity La_2O_3, $SrCO_3$, NiO, and CuO powders. La_2O_3 was dried at 850 °C for 15 h before weighing. The appropriate mixture of these powders was well ground and sintered in air at 1085 °C for five days with several intermediate grindings. The loose powder was reground and pressed into rectangular bars. The bars were sintered in the temperature range of 1250 and 1300 °C for 30 h, then cooled down to room temperature at the rate of 100 °C h^{-1}.

X-ray diffraction (XRD) analysis was carried out with an MXP 18-AHF diffractometer using Cu Kα radiation at room temperature. Resistivity was measured using a standard four-probe method in a liquid-helium Dewar. Magnetic susceptibility measurements were made with fields of 1 T between 5 and 300 K using a Quantum Design SQUID magnetometer. Transmission electron microscopy (TEM) observations were carried out with a Philips CM 200 field emission gun (FEG) TEM equipped with a Gatan imaging filter (GIF) and a liquid-nitrogen Gatan double-tilt cooling stage. The operating voltages were 200 kV.

3. Results

The powder XRD revealed that all the $La_{1.67}Sr_{0.33}Ni_{1-x}Cu_xO_4$ ($0 \leqslant x \leqslant 0.3$) samples have good K_2NiF_4-type single-phase properties. In fact, $La_{1.67}Sr_{0.33}Ni_{1-x}Cu_xO_4$ forms a solution series up to $x = 1$ [29]. All the Cu-doped samples in this series crystallize in the tetragonal lattice. The variations of the lattice parameters with Cu doping content are shown in figure 1(a). It is shown that lattice parameter a decreases continuously as a function of Cu doping content x, while the c-parameter increases. The lattice parameter changes clearly show that the doped Cu definitely substitutes for the Ni sites. From figure 1(b), we can see that the c/a ratio increases with increasing x. This indicates that Cu doping leads to an enhancement in the Jahn–Teller
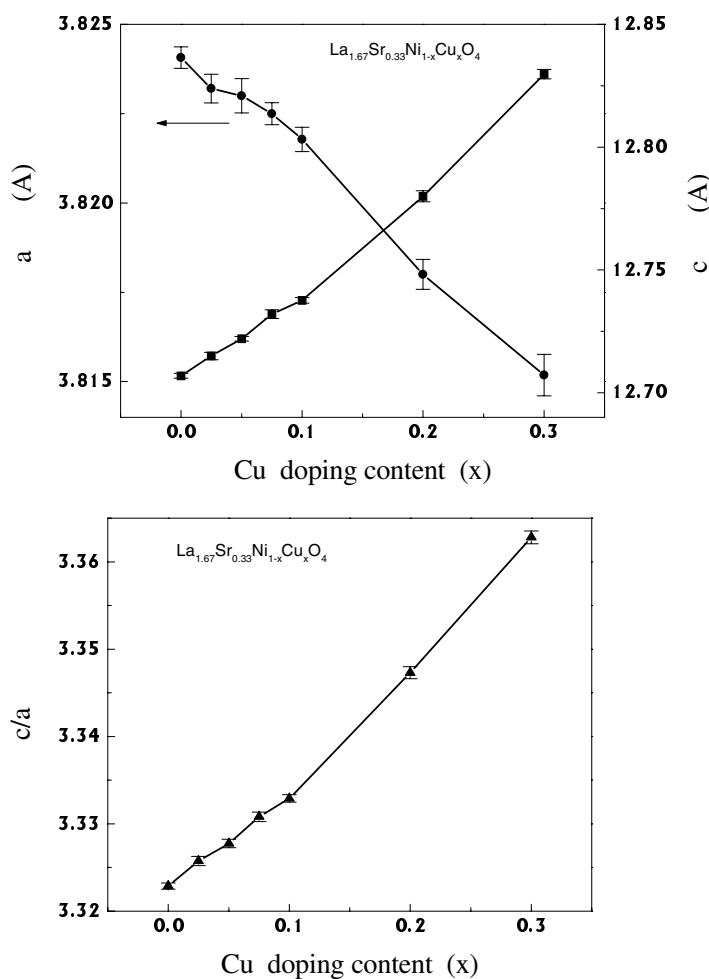
Figure 1. The lattice parameters a (circles) and c (squares) (a) and c/a (b) as a function of Cu doping content x for La$_{1.67}$Sr$_{0.33}$Ni$_{1-x}$Cu$_x$O$_4$. The short vertical lines are error bars.

distortion of the NiO$_6$ octahedra.

Shown in figure 2(a) is the temperature dependence of the resistivity ρ for the La$_{1.67}$Sr$_{0.33}$Ni$_{1-x}$Cu$_x$O$_4$ solid-solution series. In this figure, it is clear that ρ for $x = 0$ increases abruptly at $\sim$230 K with decreasing temperature T. The resistivity anomaly is attributed to the CO transition [6, 30]. The CO forms commensurate charge modulation in the NiO$_2$ plane with modulation wavevector $\delta = 1/3$. At the CO temperature, a sharp dip of the temperature derivative of $\log_{10}\rho$ appears as in figure 2(b). The dip feature may be used to identify the CO transition temperature T_{CO} as reported by Cheong *et al* [6]. But the dip feature is not well defined after Cu doping. So we take the inflection temperature, at which the variation of $\log_{10}\rho$ with T changes from a rapid decrease to a smooth decrease, as T_{CO} for the Cu-doped samples. Although the method for defining T_{CO} may introduce some error, the decreasing trend of T_{CO} with Cu doping is not affected. In figure 2(b), no dip feature can be observed when $x > 0.075$. The T_{CO} defined by the resistivity measurement is denoted as T_{CO}^R. T_{CO}^R decreases with increasing Cu doping, as plotted in figure 3.
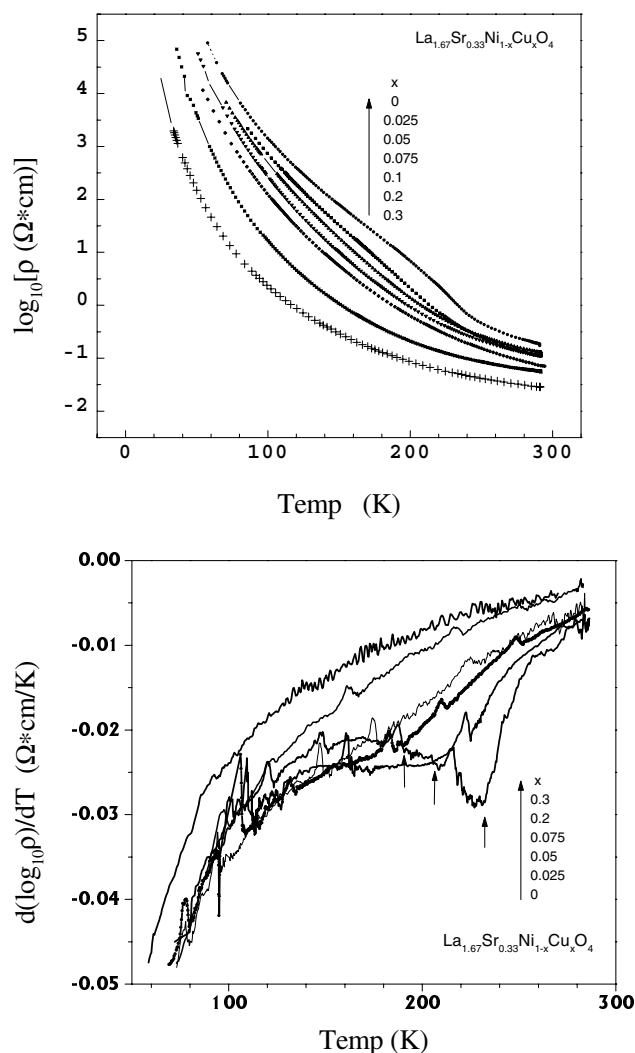
Figure 2. The logarithm of the resistivity (a) and the temperature derivative of $\log_{10} \rho$ (b) versus temperature for $La_{1.67}Sr_{0.33}Ni_{1-x}Cu_xO_4$. The short arrows show the inflection points of the curves corresponding to the CO transition.

The magnetic susceptibilities $\chi(T)$ for seven samples, measured between 5 and 300 K in 1 T, are displayed in figure 4(a). Cheong *et al* [6] found that $\chi(T)$ above 200 K is field independent. And the local minimum of χ at $\sim$235 K is an indication of the CO transition mentioned above. From the peak position of the temperature derivative of $\chi(T)T$ for $La_{1.67}Sr_{0.33}Ni_{1-x}Cu_xO_4$, T_{CO} can be determined. Shown in figure 4(b) is the temperature dependence of the temperature derivative of $\chi(T)T$ for $La_{1.67}Sr_{0.33}Ni_{1-x}Cu_xO_4$ with $0 \leqslant x \leqslant 0.3$. We can see that the peaks in the derivative gradually broaden with Cu doping. But each peak has a clear maximum. The peak feature is invisible when $x \geqslant 0.2$. This makes it easier for us to determine T_{CO} than in resistivity measurements. We denote T_{CO} derived from magnetic measurements as T_{CO}^M. The variation of T_{CO}^M with x is shown in figure 3. The results in this figure indicate that the CO phase transition moves to low temperature with
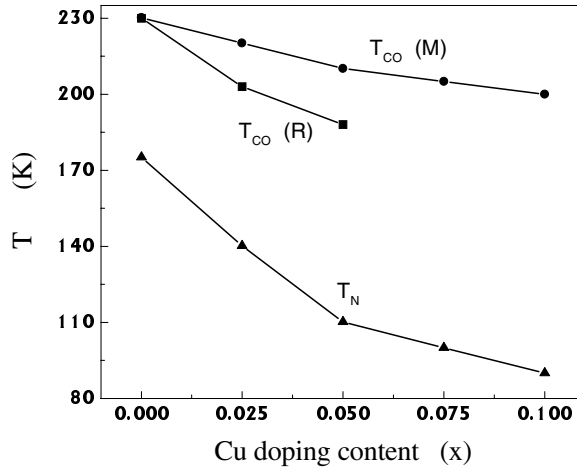
Figure 3. The CO temperatures T_{CO}^R (from resistivity) and T_{CO}^M (from magnetic susceptibility) and the spin-ordering temperature T_N versus Cu doping content x for La$_{1.67}$Sr$_{0.33}$Ni$_{1-x}$Cu$_x$O$_4$.

increasing Cu doping. Unlike the case for pure La$_{2-x}$Sr$_x$NiO$_4$, all the Cu-doped samples have a discrepancy between T_{CO}^R and T_{CO}^M. It is evident that stripes with higher resistivity have a finite length(s) and some percolation paths are introduced into the areas of stripes by Cu doping. Therefore electronic transport occurs in a nonhomogeneous media. The substantial change of resistivity is blurred by the generation of more metallic paths. The discrepancy between T_{CO}^R and T_{CO}^M most probably arises from signal smearing in the resistivity response to stripe phase in Cu-doped samples.

By analysing the data in figure 2(a), we found that the resistivity of the samples above a temperature of about 250 K follows a thermal activation law. The resistivity in the thermally activated conduction process shows the following temperature dependence:

$$\rho(T) = \rho_0 \exp(\epsilon_a / k_B T). \tag{1}$$

Figure 5 displays resistivity on a logarithmic scale versus $1/T$ for La$_{1.67}$Sr$_{0.33}$Ni$_{1-x}$Cu$_x$O$_4$ ($0 \leqslant x \leqslant 0.3$) samples above about 250 K. The thermal activation energy ϵ_a derived from the slope of the fitted straight lines in this figure is 133, 119, 107, 100, 112, 62, and 57 (meV) for $x = 0$, 0.025, 0.05, 0.075, 0.1, and 0.3 respectively. It generally shows that ϵ_a decreases from 133 meV with $x = 0.0$–56.76 meV with $x = 0.3$. It is noteworthy that an anomalously high ϵ_a appears at $x = 0.1$. Although with an unclear origin, it seems to indicate some speciality at this doping point. In addition, figures 2(b) and 4(b) also display $x = 0.1$ generally as a 'crossover' point making distinct existence from nonexistence of static stripe ordering character.

In view of these particular traces displayed at $x = 0.1$, and in order to obtain some microscopic information on stripe phase in Cu-doped La$_{1.67}$Sr$_{0.33}$NiO$_4$, we performed electron diffraction (ED) observations at 100 K for La$_{1.67}$Sr$_{0.33}$Ni$_{0.9}$Cu$_{0.1}$O$_4$. The selected-area electron diffraction (SAED) pattern along [001] axis is shown in figure 6. As in non-Cu-doped La$_{1.67}$Sr$_{0.33}$NiO$_4$ [5], four pairs of weak but well defined superlattice spots appear around each fundamental Bragg reflection along [100] and [010] direction. The modulation vector was measured to be $a^* \pm 1/3b^*$ with a period of ~8 Å. Clearly, the commensurability of charge ordering is not changed by Cu doping. Despite exactly same picture of charge ordering in La$_{1.67}$Sr$_{0.33}$Ni$_{0.9}$Cu$_{0.1}$O$_4$ as in La$_{1.67}$Sr$_{0.33}$NiO$_4$, the intensity of the superlattice spots is much weaker in the former than in the latter. Actually the picture in figure 6 was recorded
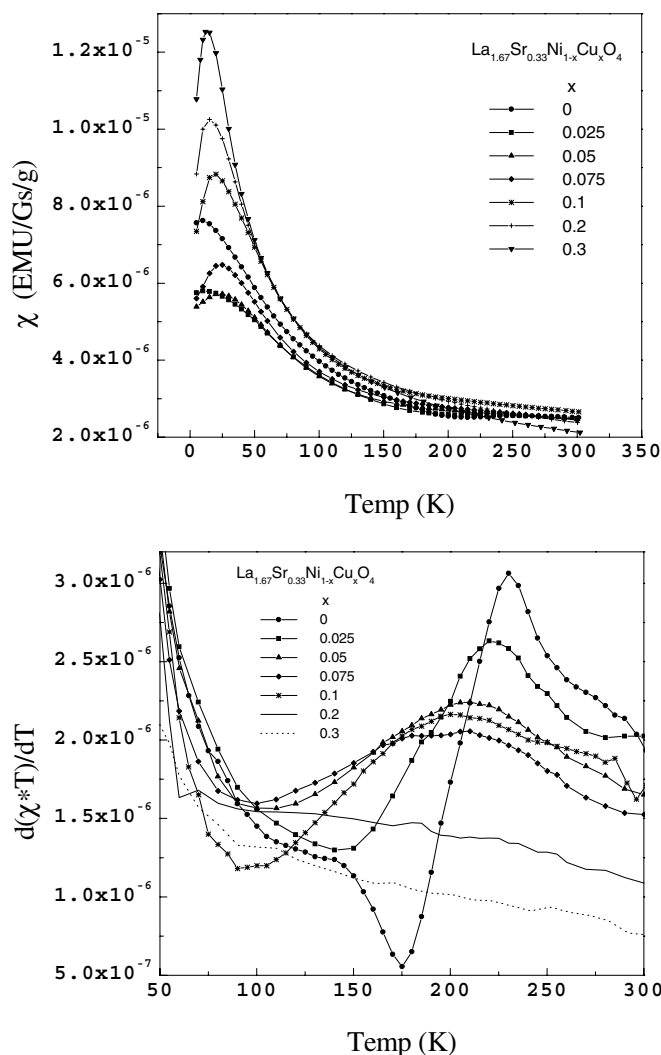
Figure 4. Magnetic susceptibility (a) and the temperature derivative of $\chi(T)T$ (b) versus temperature for $La_{1.67}Sr_{0.33}Ni_{1-x}Cu_xO_4$.

by slow-scan CCD camera. We found, even with CCD camera, these superlattice spots are so weak as to be difficult to identify them out.

4. Discussion

From our experimental observations, besides finding that charge-ordering temperature T_{CO} lowers under Cu doping, figure 2(a) shows generally that the resistivity lowers upon Cu doping at a fixed temperature. From large numbers of studies on $La_{2-x}Sr_xCuO_4$ and $La_{2-x}Sr_xNiO_4$ systems, Cu has the same valence state 2+ as Ni. Thus, Cu doping in $La_{1.67}Sr_{0.33}NiO_4$ does not change the nominal carrier density. It has been suggested that in La_2NiO_4 there exists stronger magnetic localization [31] than in La_2CuO_4. When hole carriers are introduced into La_2CuO_4
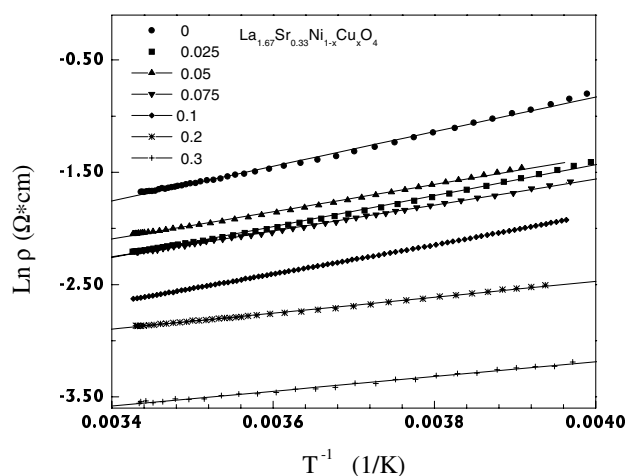
Figure 5. Resistivity on a logarithmic scale versus $1/T$ for La$_{1.67}$Sr$_{0.33}$Ni$_{1-x}$Cu$_x$O$_4$; each straight line is a least-squares fit to the data.
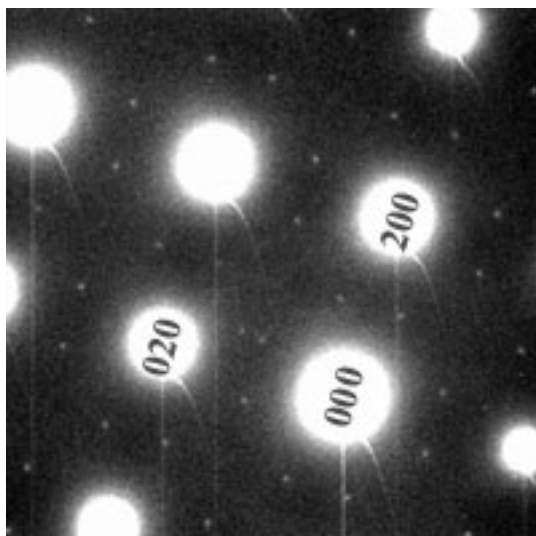
Figure 6. The [001] zone-axis ED pattern obtained at 100 K for La$_{1.67}$Sr$_{0.33}$Ni$_{0.9}$Cu$_{0.1}$O$_4$.

and La$_2$NiO$_4$, the holes predominantly have oxygen 2p character and interact with 3d spins [1]. The strong magnetic interaction make holes tend to be self-localized in La$_{2-x}$Sr$_x$NiO$_4$. While a weaker magnetic localization and higher oxygen 2p-like band than the lower Hubbard band of Cu 3d states make holes delocalized in La$_{2-x}$Sr$_x$CuO$_4$ [32]. It can be inferred that the resistivity lowering upon Cu doping must be due to an increase of the hole carriers mobility. However, as shown in figure 4(a), when 50 K $< T <$ 300 K, there seems to exist no monotonic variation of susceptibility upon Cu doping. This may be relevant to the complicated spin and charge ordering, magnetic correlation disturbation and disorder effect from Cu doping. Below 50 K, a peak generally shifts up with Cu doping. This behaviour is related to a spin–glass transition occurring at low temperature [33].

Up to now, we know that Cu doping in the stripe-ordered $La_{1.67}Sr_{0.33}NiO_4$ reduces the charge ordering temperature T_{CO}, enhances the mobility of carriers and induces spin–glass transitions at temperature less than 50 K. Simply, the enhanced carrier mobility (conductivity) may push down T_{CO}. In fact, enhancement of carrier mobility may just be the origin of T_{CO} reduction in a simple logic because stripes mean localized and well ordered electronic states. The enhanced carrier mobility tends to destroy localization and weaken the ordering degree of electronic states. Furthermore, as discussed below, increase of carrier mobility is intrinsically relevant to the change of magnetic correlation in NiO_2 plane under Cu doping.

The ED pattern shown in figure 6, with the same commensurability as in $La_{1.67}Sr_{0.33}NiO_4$ and very weak superlattice spots, indicates that Cu doping on NiO_2 plane in $La_{1.67}Sr_{0.33}NiO_4$ does not change the nature of charge ordering but clearly reduces the coherence length of charge ordering. This has a good agreement with broadening of the characteristic peaks in resistive and magnetic behaviour as shown in figures 2(b) and 4(b). The unchanged nature of charge ordering with Cu doping is compatible with the fact of the existance of Cu ions with the same valence state as Ni ions in NiO_2 planes. For $x \geqslant 0.2$, the absence of characteristic peaks in figures 2(b) and 4(b) can mean that the stripe ordering has disappeared. However, it is also possible that the stripes persist in a short-range or fluctuating (dynamic) form. Arising from Cu doping, reduction of coherence length could come from two aspects: disorder effect from random distribution of Cu^{2+} and magnetic effect from interaction of Cu with Ni spin lattice.

As regards the disorder effect, recently we noted that some papers have theoretically discussed the effect of disorder on the stripe phase in high-T_c superconductors and related materials. In these papers [17, 34–39], there are two basic points related to the present work. First, disorder is generally regarded as playing the role of pinning the stripe-ordering array and slowing down the dynamics, suppressing dynamic fluctuation of the stripe phase, and leading to the formation of a static CO. Second, disorder generated by randomly distributed impurities in the stripe phase induces stripe meandering and reduces the ordering of the stripe phase from quasi-long-range to short-range positional order. Despite mainly relating to dynamic stripe phases, as regards the length scale of the stripe ordering this conclusion seems similar to our present finding of reduction of the stripe coherence length in CO with disordered Cu doping. However, the first point seems not compatible with our experiments—because if disorder tends to slow down stripe dynamics and leads to it being more static, it is hard to understand the lowering resistivity and thermal activation energy with rising level of Cu doping into NiO_2 planes in stripe-ordered $La_{1.67}Sr_{0.33}NiO_4$. Hence, in our opinion, our present experiments cannot be fully interpreted in the framework of the disorder effect on the stripe phase. In fact, interaction of Cu spins with the host Ni spin lattice shows a large effect on the properties of the stripe phase in the Cu-doped $La_{1.67}Sr_{0.33}NiO_4$ as revealed below.

Recently, it has been found that spin ordering takes place below $T_N \sim 180$ K [27, 39] after CO in charge-ordered $La_{1.67}Sr_{0.33}NiO_4$. The temperature T_N is also a phase transition temperature at which stripe lattice states change into stripe glass states with heating [27]. In a measurement of the bulk susceptibility $d\chi(T)T/dT$, T_N is associated with the local minimum spot after the CO transition [6, 27]. In our experimental results, with Cu doping, the minimum spot moves gradually to lower temperature. This reflects T_N decreasing with Cu doping. It is also obvious that T_N decreases faster than T_{CO} with Cu doping, as shown in figure 3. This implies that introduction of Cu into NiO_2 planes strongly disturbs the spin/charge correlation within and between the NiO_2 planes and reduces the effective exchange interactions between Ni spins. These effects cause the suppression of T_{CO} and T_N with Cu doping. This also demonstrates that Cu doping leads to stronger perturbation of the spin system than the charge system in the $La_{1.67}Sr_{0.33}NiO_4$ stripe phase. A simple explanation is that Cu shows the same valence state as Ni but has only half the spin of Ni. In addition, referring to the results reported

by Lee and Cheong [27] and figure 4(b), we found that introduction of Cu into NiO$_2$ planes has a role of melting the stripe lattice and maintaining stripe glass states over a wider temperature region. This may possibly be ascribed to enhanced charge and spin stripe fluctuation with Cu doping. Therefore, like in the La$_{1.67}$Sr$_{0.33}$NiO$_4$, although no long-range spin ordering appears, spin-ordering fluctuation may exist in the stripe glass region in more highly (e.g. $x \geqslant 0.2$) Cu-doped La$_{1.67}$Sr$_{0.33}$Ni$_{1-x}$Cu$_x$O$_4$ [40]. Dynamics experiments, theoretical studies, and quantitative interpretation are needed to further understand the Cu doping effect on CO in La$_{1.67}$Sr$_{0.33}$Ni$_{1-x}$Cu$_x$O$_4$.

5. Conclusions

In summary, we have investigated the effect of Cu doping on charge and spin stripe ordering in La$_{1.67}$Sr$_{0.33}$NiO$_4$ by means of resistivity and magnetic susceptibility measurements and TEM observation. We found that Cu substitution for Ni on the NiO$_2$ planes does not change the nature of the CO but makes both the CO and spin-ordering transitions move to low temperature and reduces the stripe-ordering correlation lengths. The temperature regions of existence of stripe glass are gradually extended with increasing Cu doping. This reveals for La$_{1.67}$Sr$_{0.33}$NiO$_4$ that introduction of Cu into the NiO$_2$ planes strongly disturbs the spin/charge correlation within and between the NiO$_2$ planes and causes a reduction in the effective exchange interactions between Ni spins. And Cu doping leads to stronger perturbation of the spin system than the charge system in the La$_{1.67}$Sr$_{0.33}$NiO$_4$ stripe phase. It is inferred that these effects arise from the disordering effect of the random distribution of Cu^{2+} in NiO$_2$ planes and the magnetic spin interaction of the Cu spin with the Ni spin–lattice system.

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