Diagonal static spin correlation in the low temperature orthorhombic $Pccn$ phase of $La_{1.55}Nd_{0.4}Sr_{0.05}CuO_4$

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Elastic neutron-scattering measurements have been performed on $La_{1.55}Nd_{0.4}Sr_{0.05}CuO_4$, which exhibits a structural phase transition at $T_s \sim 60 \text{ K}$ from the low temperature orthorhombic $Bmab$ phase (labelled LTO1) to the low temperature orthorhombic $Pccn$ phase (labelled LTO2). At low temperatures, well below $T_s$, elastic magnetic peaks are observed at the “diagonal” incommensurate (IC) positions $(0, 1\pm0.055, 0)$, with the modulation direction only along the orthorhombic $b$-axis just as in Nd-free $La_{1.95}Sr_{0.05}CuO_4$. In the present study, the one-dimensionality of the IC modulation, which is naturally explained by a stripe model, is clearly demonstrated with our “single-domain” crystal. The temperature dependence of the IC peak intensity suggests a substantial contribution from the Nd$^{3+}$ spins below $\sim 3 \text{ K}$. Consistent with this, the $L$ dependence of the magnetic scattering is accurately accounted for by a model in which the contribution of the Nd$^{3+}$ spins is explicitly included.

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I. INTRODUCTION

The relationship between the microscopic magnetism and superconductivity is one of the central issues in the field of high-$T_C$ superconductivity. In particular, $La_{2-x}(Sr,Ba)_xCuO_4$ (LSCO, LBCO) and related compounds have received intensive attention because of their rich magnetic and transport properties. In addition, these materials have a simple layered structure with single CuO$_2$ planes composed of square Cu$^{2+}$ lattices; this facilitates the application of theoretical models. It is well known that superconducting LSCO samples exhibit dynamic incommensurate (IC) magnetic correlations modulated along the direction parallel to the Cu-O-Cu bonds in the low-temperature orthorhombic (LTO1, $Bmab$) structure. After the discovery of this IC nature, a systematic neutron-scattering study on the superconducting LSCO compounds revealed a linear relation between the hole concentration $x$ and the incommensurability parameter $\delta$ (Ref. 6) in the under-doped region $(0.06 \leq x \leq 0.12)$, suggesting a strong correlation between the superconductivity and the dynamic IC modulation.

On the other hand, several investigations have been performed at the specific hole concentration $x \sim 1/8$ where for many co-dopants the superconductivity is dramatically suppressed. This so-called $1/8$ anomaly was originally discovered in the LBCO system and found to be associated with a structural transition to the low-temperature tetragonal (LTT, $P4mm$) phase. A similar, but much smaller, suppression of $T_c$, as well as an enhancement of the static magnetic correlations, has been reported in the LSCO system, in which there is no transition to the LTT phase. [3] An important clue relevant to the $1/8$ anomaly was the observation in the $La_{1.48}Nd_{0.4}Sr_{0.12}CuO_4$ compound of very clear elastic magnetic peaks at the parallel IC positions around $(\pi, \pi)$ by neutron-scattering. [4] (The substituted Nd$^{3+}$ ions induce the LTT structure as well as the $1/8$ anomaly.) Note that Nd$^{3+}$ ions introduce no holes into the system.) On the basis of the stripe model, it was suggested that charge stripes along the Cu-O-Cu bond, which result in parallel IC peaks, might be pinned by the corrugation of the CuO$_2$ plane caused by the coherent tilt of the CuO$_6$ octahedra which is perpendicular to the stripes in the LTT structure. Hence, the elastic IC correlations are enhanced and the superconductivity is more suppressed than in the LTO1 structure of LSCO.

Recently Wakimoto et al. discovered the so-called “diagonal” IC peaks in insulating $La_{1.95}Sr_{0.05}CuO_4,$
which shows the LTO1 structure. The IC peak positions are shown in Fig. 1(a). In this case, the IC modulation is parallel to the orthorhombic b-axis, which is the same as the coherent tilting direction of the CuO$_6$ octahedra, and at 45° to the Cu-O bonds. Assuming that the magnetic peaks are associated with charge stripe order, the charge stripes would run parallel to the orthorhombic a-axis. Thus, similar to the case of La$_{2-x-y}$Nd$_y$Sr$_x$CuO$_4$ (LNSCO), the stripes may be pinned by the corrugation of the CuO$_2$ planes in the LTO1 phase; one might then speculate that the pinning is responsible for the insulating behavior below $\sim$ 100 K. Subsequent experiments by Matsuda et al. and by Fujita et al. have shown that this diagonal one-dimensional (1D) spin density wave state in LSCO extends across the entire spin-glass region $0.02 \lesssim x \lesssim 0.06$.

**II. SAMPLE PREPARATION AND EXPERIMENTAL DETAILS**

Single crystals of La$_{1.55}$Nd$_{0.4}$Sr$_{0.05}$CuO$_4$ were grown by the travelling-solvent floating-zone method. (Two crystals were prepared in the same manner as described below. Most of the data presented here are obtained from one of them, and the other one reproduced the magnetic and transport properties. Therefore, we do not distinguish two crystals in this paper.) Dried powders of La$_2$O$_3$, Nd$_2$O$_3$, SrCO$_3$ and CuO of 99.99 % purity were mixed and baked in air at 950°C and 1000°C for 24 hours with grinding between each baking. The powder sample so-obtained was confirmed to be a single 2-1-4 phase by X-ray powder diffraction. Feed rods were shaped in rubber tubing pressed by a hydrostatic press, and baked in air at 1100°C for 12 hours. Solvents with the composition of La$_{1.55}$Nd$_{0.4}$Sr$_{0.05}$CuO$_4$ : CuO = 30 : 70 in molar ratio were chosen based on the phase diagram for the pure La$_2$CuO$_4$ compound. The growth was performed in a four-ellipsoidal-mirror type image furnace in an oxygen atmosphere. The pelletized solvent was placed between the feed rod (attached to the upper shaft) and a seed crystal (on the lower shaft), and was melted at the focal point. A crystal was grown by moving the ellipsoidal-mirrors upward. During the high temperature operation of growth, there is vaporization of a small amount of CuO from the molten zone which causes a change in the solvent composition. To avoid this, extra CuO of $\sim$ 1 mol% was added into the feed rods to compensate for the loss of CuO vaporizing from the molten zone during the growth. Since the concentration of Nd and Sr in the crystallized crystals was controlled in the melt-grown process in an oxygen atmosphere, the as-grown crystal was annealed in an Ar atmosphere at 850 °C for 12 hours to purge any excess oxygen. After the treatment, the sample exhibits a spin-glass behavior below $\sim$ 5 K in the magnetic susceptibility...
The sample was mounted in either the (HK0) or (0KL) scattering plane in a pumped-helium cryostat. The lattice parameters were $a = 5.349$ Å, $b = 5.355$ Å and $c = 13.012$ Å at 1.5 K.

Figure 1(b) shows the scattering geometry in the (HK0) scattering plane of the present LNSCO crystal. The squares indicate the apparent nuclear Bragg peak positions determined by neutrons with a half wave length ($\lambda/2$), while the circles indicate the IC elastic magnetic peak positions. Properly, there is no nuclear or magnetic peak at the orthorhombic (100) or (010) positions. The $\lambda/2$ measurement is used to determine precisely the orientation of the IC peaks with respect to the reciprocal lattice (and possible twin domains).

The orthorhombic structure typically has two or more twin domains, as shown in Fig. 1(a), which indicates the nuclear and magnetic peak geometry in the Nd-free $x = 0.05$ crystal containing two twin-domains. However, in the present crystal, one of the domains is so dominant that one can treat it as effectively a single domain crystal. Indeed, only a single pair of the diagonal type IC peaks is clearly observed around the (010) position (see Sec. III and IV).

Since the orthorhombic crystallographic axes in both the LTO1 and LTO2 structures are defined by the diagonals of the distorted squares of the CuO$_2$ lattice, the orthorhombic $a^*$ and $b^*$ axes in reciprocal space are defined as shown in Fig. 1. Throughout the present paper, indices based on the orthorhombic $Bmab$ or $Pcmm$ crystallographic notation are utilized. Since clear IC magnetic peaks are observed only around (010), we utilized the (0KL) scattering plane to measure the $L$ dependence of the IC peak intensity so that the high-intensity peaks lie in the scattering plane.

Neutron scattering experiments were performed at the triple-axis spectrometer SPINS installed at the cold neutron guide at the NIST research reactor. The horizontal collimator sequence 32'-40'-S-40'-open and an incident neutron energy $E_i=5$ meV were utilized. Pyrolytic graphite (002) was used as both monochromator and analyzer. Contamination from higher-order neutrons was eliminated partially with a single Be filter, and completely with two Be filters. We confirmed that there is no significant multiple scattering around ($\pi, \pi$) with this incident energy in elastic scattering measurements. A crystal 40 mm in length for the neutron-scattering experiments was cut from the end part of the grown crystal. The sample was mounted in either the (HK0) or (0KL)
III. EXPERIMENTAL RESULTS

A. Structural transition to the LTO2 phase

Although one twin domain is dominant in the present crystal, there are some other minor twin domains whose volume is estimated to be less than 1/4 of that of the dominant domain. Therefore, we were able to observe the orthorhombic splitting between the (200) peak of the dominant domain and the (020) peak in a minor domain. Figure 3(a) shows the temperature dependence of the splitting. On cooling, the orthorhombicity decreases by about 30% between 65 K and 40 K. The intensity of the (110) peak, which is a superlattice peak of the Pccn structure but not of the Bmab structure, appears and increases in a complementary manner as shown in Fig. 3(b). These facts demonstrate that the system indeed exhibits a structural transition from the Bmab LTO1 phase to the Pccn LTO2 phase. The transition temperature of $\sim$ 65 K agrees well with that expected from the phase diagram previously reported by Crawford et al. This transition temperature is also in reasonable agreement with that determined from the resistivity measurement.

In the LTO1 structure above 65 K, the CuO$_6$ octahedra tilt along the orthorhombic $b$-axis. In the LTO2 structure, the octahedra tilt along a direction rotated within the plane away from the $b$-axis. From the change of the orthorhombic splitting in Fig. 3, the shift of the tilt direction from the $b$-axis is estimated to be $\sim$ 15 degrees.

Contour plots of the elastic neutron-scattering intensity around (010) and (100) are shown in Figs. 4(a) and 4(b), respectively. Since the crystal has a single dominant twin domain, the 1D nature of the IC modulation along the orthorhombic $b$-axis is clearly observable in Fig. 4(a); it is consistent with, but more obvious than, that first reported for the Nd-free $x = 0.05$ sample. An important difference from the Nd-free $x = 0.05$ compound is that clear magnetic peaks appear only around the (010) position; for the Nd-free sample, the intensity is strongest for the peaks split about (100). (The two cases are schematically summarized in Fig. 1.) A similar change of the magnetic peak position from (100) to (010) has been previously reported in Sr-free La$_{2-y}$Nd$_y$CuO$_4$. This feature is discussed in Sec.IV in terms of the spin orientation.

To analyze the IC peaks in detail, we made scans through the peak positions in the vicinity of (010) with higher statistics, achieved by optimizing the vertical focusing of the incident neutron beam. Figures 5(a) and 5(b) show the peak profiles along the trajectories $\alpha$ and $\beta$ indicated by arrows in Fig. 5(d). The incommensurate peaks are observed at the $(0, 1 \pm \epsilon, 0)$ positions. (The IC peak intensity in unit time is different from that in Fig. 4 due to the change in the vertical focusing of the incident beam; however, the vertical focusing does not affect the intrinsic characteristic values, such as the width, positions and temperature dependence of the IC peaks.)

The solid lines in Fig. 5 are fitted curves corresponding to a two-dimensional Lorentzian function convoluted with the instrumental resolution. The intrinsic peak half widths along the $a^*$ and $b^*$ directions are $\kappa_{a^*} = 0.053$ Å$^{-1}$ and $\kappa_{b^*} = 0.039$ Å$^{-1}$, respectively. The incommensurability parameter is $\epsilon = 0.055(\pm0.004)$, which is slightly
lower than that for Nd-free \( x = 0.05 \), where \( \epsilon = 0.064 \). Although in the contour plot of Fig. 4(b) the peak at \((0, 1 - \epsilon, 0)\) seems to be split additionally along the \( a^*\)-axis, the profile in Fig. 5(b) with higher statistics shows a single peak centered at \( h = 0 \).

The temperature dependence of the IC peak at \((0, 0.94, 0)\) is shown in Fig. 5(c). This measurement was done without optimizing the vertical focusing. The intensity gradually increases with decreasing temperature below \( \sim 5 \) K and rapidly increases below \( \sim 3 \) K, from previous experience with Nd-substituted samples, we expect that this additional increase of intensity below \( \sim 3 \) K is caused by an additional ordering of the Nd\(^{3+}\) spins.

![Graph showing the temperature dependence of the IC peak intensity](image)

**FIG. 6.** \( L \) dependence of the IC peak intensity in (a) La\(_{1.95}\)Nd\(_{0.05}\)Sr\(_{0.05}\)CuO\(_4\) at 1.5K, (b) La\(_{1.55}\)Nd\(_{0.45}\)Sr\(_{0.05}\)CuO\(_4\) at 3K, and (c) Nd-free La\(_{1.95}\)Sr\(_{0.05}\)CuO\(_4\) at 1.5K (obtained from Ref. 18). The back ground intensity at 30K have been subtracted. Solid lines in (a) and (b) are the results of fit by a model including contribution from Nd\(^{3+}\) spins and out-of-plane component of Cu\(^{2+}\) spins. (See text). Solid line in (c) is the fit with the same model without Nd\(^{3+}\) spin contribution and out-of-plane component of Cu\(^{2+}\), while dashed line is the fit without only Nd\(^{3+}\) spin component.

The \( L \) dependences of the intensity at the IC position \((0, 0.94, L)\) at 1.5 K and 3 K are shown in Figs. 6(a) and 6(b), respectively. For both results, the intensity at 30 K was subtracted as background so that the intensities shown are purely magnetic. We fit the data with a model introduced previously in a study of the Nd-substituted \( x = 0.12 \) compound. The model has the form:

\[
I \propto |F|^2 \frac{1 - t^2}{1 + t^2 - 2t \cos \phi}.
\]

This function consists of the magnetic structure factor \( F \) and an oscillating function with line width determined by \( t = \exp(-c/\xi) \), where \( \xi \) is a correlation length. In the analysis of the Nd-substituted \( x = 0.12 \) results, an oscillation period of \( L = 1 \) was utilized. This was an appropriate choice in that case because of the assumed rotation of the stripe orientation by 90° from one CuO\(_2\) plane to the nearest neighbor plane, which gives rise to correlations between the next nearest neighbor planes. However, in the present system with \( x = 0.05 \) the IC modulation is only along the orthorhombic \( b \)-axis, that is, a 90° rotation of the stripe orientation is not likely. Thus, we utilized the oscillation function with a period of \( L = 2 \); that is, \( \phi = \pi L \).

The structure factor is described as

\[
|F|^2 = |F_{\text{Cu}, \parallel}|^2 f_{\text{Cu}}^2 + |p_{\text{Cu}, \perp} f_{\text{Cu}} + y p_{\text{Nd}} f_{\text{Nd}} \cos (2\pi L z_{\text{Nd}})|^2.
\]

In this equation, \( f_{\text{Cu}} \) and \( f_{\text{Nd}} \) are \( Q \)-dependent magnetic form factors for the Cu\(^{2+}\) and Nd\(^{3+}\) spins which are taken from the literature. \( z_{\text{Nd}} \) is the distance between Nd and the nearest Cu in units of \( c \), which has been determined to be 0.36 by a neutron powder diffraction study. \( p \) is the spin component perpendicular to the scattering vector \( Q \), which relates to the ordered magnetic moment \( \mu \) by \( p = \vec{\mu} - \vec{Q} (\vec{Q} \cdot \vec{\mu}) \). The indices \( \parallel \) and \( \perp \) represent in-plane and out-of-plane components of the Cu\(^{2+}\) spin, respectively.

In this model the Nd\(^{3+}\) spins are assumed to be parallel to the \( c \)-axis which is expected to be the easy axis of the Nd\(^{3+}\) spins based on the magnetic susceptibility measurements for La\(_{1.92}\)Nd\(_{0.08}\)Sr\(_{0.05}\)CuO\(_4\). Therefore, the first term of Eq. (2) describes the contribution of the in-plane component of the Cu\(^{2+}\) spins while the second term describes contributions of the out-of-plane components of both the Cu\(^{2+}\) and Nd\(^{3+}\) spins. We assumed the spin direction of \( p_\parallel \) to be random because the system shows the features of a spin-glass. The fitting results are shown by the solid lines in Figs. 6(a) and 6(b), which agree with the experimental results.

| \( y \) and \( T(K) \) | \( \mu_{\text{Cu}, \parallel}/\mu_{\text{Cu}, \perp} \) | \( \mu_{\text{Nd}}/\mu_{\text{Cu}} \) | \( \xi/c \) |
|---|---|---|---|
| \( y = 0.4, T = 1.5 \) | 0.96 ± 0.25 | 4.1 ± 0.9 | 0.58 ± 0.11 |
| \( y = 0.4, T = 3 \) | 1.04 ± 0.37 | 1.1 ± 0.6 | 0.36 ± 0.18 |
| \( y = 0, T = 1.5 \) | 0 | 0 | 0.48 ± 0.05 |

(0.55 ± 0.05) (0) (0.44 ± 0.04)
In order to check the consistency of this function, we also fit the $L$ dependence of the IC peak intensity for the Nd-free $x = 0.05$ compound reported in Ref. 18 with the function presented above without the Nd$^{3+}$ spin contribution. Since the out-of-plane component of the Cu$^{2+}$ spins $p_{\text{Cu}, \perp}$ is assumed to be driven by the interaction with the Nd$^{3+}$ spins in the present model, we fit the data with $p_{\text{Cu}, \perp}$ fixed at zero. The fitted curve is shown in Fig. 6(c) by the solid line. For comparison, we also fit the data with $p_{\text{Cu}, \perp}$ as a fitting variable, as shown by the dashed line.

The parameters obtained by the fitting illustrated in Fig. 6 are summarized in Table 1. The values listed in the bottom row are obtained by fitting the Nd-free data with a $p_{\text{Cu}, \perp}$ component. In the Nd-substituted sample, the out-of-plane component of the Cu$^{2+}$ spins is larger than that in the Nd-free sample even if we assume the Nd-free sample also has an out-of-plane component. This indicates that the the out-of-plane component of the Cu$^{2+}$ spins is induced by the correlation with the Nd$^{3+}$ spins parallel to the $c$-axis. For the Nd-substituted sample, the ratio $\mu_{\text{Nd}}/\mu_{\text{Cu}}$ increases with decreasing temperature, consistent with the rapid increase of the IC intensity below 3 K in Fig. 5(c).

**IV. DISCUSSION**

The present paper reports the IC magnetic order observed by neutron-scattering experiments for the La$_{1.50}$Nd$_{0.4}$Sr$_{0.66}$CuO$_4$ compound. One of the important results is that the same type of 1D IC spin modulation as that reported for the Nd-free $x = 0.05$ compound is clearly observed in the almost single twin-domain sample of Nd-substituted $x = 0.05$. This demonstrates that the 1D IC modulation along the orthorhombic $b$-axis is common in the lightly Sr-doped spin-glass systems with both LTO1 and LTO2 structure.

A major difference from the Nd-free sample is that the clear IC peaks appear around the (010) position in the present LTO2 compound, while a more intense pair of peaks appears around (100) in the Nd-free $x = 0.05$. A similar change of the magnetic peak position from (100) to (010) has been reported by powder neutron-scattering for La$_{1.8}$Nd$_{0.2}$CuO$_4$ the magnetic commensurate peak at (100) in the LTO1 phase shifts to (010) below the LTO1-LTO2 transition temperature. Such a change of the magnetic peak position can be explained simply by a rotation of the spin direction that causes a change of the antiferromagnetic propagation vector from the $\hat{a}$ to the $\hat{b}$ direction. This change is schematically drawn in Fig. 7. The square represents the CuO$_2$ square lattice. The arrows at the corners are spins of the Cu$^{2+}$ ions on the same CuO$_2$ plane while the arrows at the center are spins on the nearest neighbor plane.

![FIG. 7. Schematic figure of the spin structure. The square represents CuO$_2$ square lattice. The arrows at the corners are spins of the Cu$^{2+}$ ions on the same plane and the arrows at the center are spins on the nearest neighbor plane. The spin structure shown by the dashed and solid arrows give the magnetic bragg peaks at (100) and (010), respectively.](image-url)
In the present system, the charge stripes are parallel to the orthorhombic a-axis and have the spacing nb as schematically shown in the upper panel of Fig. 8. (This figure corresponds to the spacing n = 7.) Therefore, the magnetic structure factor F can be described approximately as a function of k:

\[ F \propto 1 + 2 \sum_{j=1}^{n-1} (-1)^j \cos(\pi j k). \]  

The actual fitted curve using Eqs. (1) and (3) with fixed n = 7 is indicated by the solid line in Fig. 8. The only fitting parameter, \( \xi \), is \( \sim 31 \) Å. (The stripe spacing n = 7 is smaller than the expected value n = 8 from the incommensurability \( \epsilon \sim 0.06 \) reciprocal lattice unit (r.l.u.). However, in the fitting with n = 8, the damping by the magnetic structure factor is stronger than that for n = 7 and results in poorer agreement with the data.) Figure 8 demonstrates that this model gives a reasonable description of the experimental data. As noted in section III, in the comparison of the incommensurability parameter \( \epsilon \) determined by the Lorentzian function fit, the Nd-substituted sample has the incommensurability, \( \epsilon = 0.055 \), which is slightly smaller than that for Nd-free sample, \( \epsilon = 0.064 \). However, in the fitting using the stripe function, the fit with the fixed parameter n = 7 also agrees reasonably with the experimental data for Nd-free \( x = 0.05 \) with the fitting variable \( \xi \sim 47 \) Å. Thus the stripe structure can explain the IC peaks also in the lightly hole doped region with a stripe spacing of 7b for the \( x = 0.05 \) compounds.

As a future experiment, it would be interesting to see whether an in-plane anisotropy of the conductivity, associated with the 1D magnetic modulation, can be observed in an unwinned crystal with \( x \leq 0.05 \), where a unique orientation of the IC modulation has been observed.

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