

Quasi-elastic and inelastic neutron-scattering studies of superconducting $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$

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We review the results of recent neutron-scattering studies of the spin fluctuations in samples of $\text{La}_{1.89}\text{Sr}_{0.11}\text{CuO}_4$ which are ~80% superconducting with $T_c = 10$ K. The structure factor, $S(\vec{Q})$, reflects three-dimensional modulated spin correlations with an in-plane correlation length of order 18 ± 6 Å. The fluctuations evolve with temperature from being predominantly dynamic at high temperatures to mainly quasi-elastic ($|\Delta E| < 0.5$ meV) at low temperatures. No significant differences are observed in the normal and superconducting states.

1. Introduction

Experiments indicate that the CuO_2 lamellar superconducting materials exhibit novel but complicated

magnetism [1-3]. Recent neutron experiments in $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ with $0.02 \leq x \leq 0.14$ show that as x increases, the Cu^{++} moment is preserved, but the antiferromagnetic correlations become progressively shorter-ranged [1]. These experiments, however, do not provide a definitive description of the magnetism in the superconducting state, since the samples studied there exhibit a Meissner fraction of at best 15%. Recently, however, two of us (Y. H. and T. M.) have made significant progress in the growth of single-crystal $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ of high crystalline perfection and with a large Meissner fraction (~80%) [4].

In this paper we review recent neutron-scattering studies of samples of $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ with $T_c = 10$ K and an 80% Meissner fraction [5]. The high quality of the samples has allowed a much more thorough study of the spin correlations than was previously possible. A number of new results have therefore been found. First, the static structure factor $S(\vec{Q})$ exhibits a complicated three-dimensional incommensurate structure with a characteristic two-dimensional (2D) correlation length of order 18 ± 6 Å. The low-energy ($|\Delta E| < 0.5$ meV) part of $S(\vec{Q})$ appears to exhibit pronounced *three-dimensional* correlations at all temperatures (5 K to 350 K). The low-energy part of the response function evolves with temperature from being predominantly inelastic ($|\Delta E| > 0.5$ meV) at high

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temperatures to mainly quasi-elastic ($|\Delta E| < 0.5$ meV) at low temperatures, $T \leq 50$ K. The integrated intensity is, however, preserved; further, it is close to that observed under identical spectrometer conditions for pure La_2CuO_4 [6].

2. The energy-integrating experiments

The experiments were carried out on the H7 and H4M triple-axis spectrometers at the Brookhaven high flux beam reactor. As is discussed below, the experiments proved to be rather difficult and thus required a novel approach to data collection. Specifically, it was discovered that there was a striking thermal evolution in the distribution in energy of the scattering, so that it was essential to separate the quasi-elastic ($|\Delta E| < 0.5$ meV) and integrated inelastic ($|\Delta E| > 0.5$ meV) contributions to $S(\vec{Q})$. This was accomplished with a filtering technique described elsewhere [5].

Two crystals of $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$, labeled NTT-30 and NTT-35, were studied. The samples measured $\sim 2 \times 2 \times 0.2$ cm, with the thin direction along the orthorhombic $\vec{b}$, perpendicular to the CuO_2 planes ($\vec{a}-\vec{c}$). For both crystals the tetragonal-orthorhombic structural phase transition occurred at 265 ± 10 K; from Figure 3 of [1], this implies $x = 0.11 \pm 0.02$ —close to, but slightly less than, the chemical analysis value of $x = 0.14 \pm 0.02$. The new crystal-growth technique is described elsewhere [4]; as discussed there, this technique produces large single crystals with an 80% Meissner fraction. As a check, the Meissner fraction was measured via both the zero-field and field-cooled susceptibility in fields of order 10 Oe on a piece $6 \times 6 \times 2$ mm broken from NTT-35; the sample was found to be at least 80% superconducting, with $T_c = 10$ K. These data will be discussed in more detail in [7]. We also confirmed directly, using a neutron depolarization technique, that both samples become superconducting at 10 K. We suspect that this low T_c is due to oxygen vacancies, and indeed these presumed vacancies may play an important role in the spin-fluctuation spectrum.

Closely similar results were obtained in both samples; however, because our data for NTT-30 are much more extensive, we review primarily those results in this paper. The sample was mounted with the orthorhombic $\vec{a}$ (or $\vec{c}$ because of twinning) axis vertical (see [1, 6] for diagrams of the real-space lattice), first in a closed-cycle refrigerator ($12 \text{ K} \leq T \leq 350 \text{ K}$) and second in a pumped helium cryostat ($1.9 \text{ K} \leq T \leq 20 \text{ K}$). The sample was carefully masked in order to minimize parasitic peaks from the sample container, multiple scattering, etc. The crystals themselves are of very high quality, so that there is little or no contamination from powder lines, flux inclusions, or unreacted material.

Representative scans across the 2D magnetic ridge are shown in Figures 1 and 2. As discussed in [1, 6], these scans are along the direction $(h, h - 0.45, 0)$, which for $E_i = 14.7$

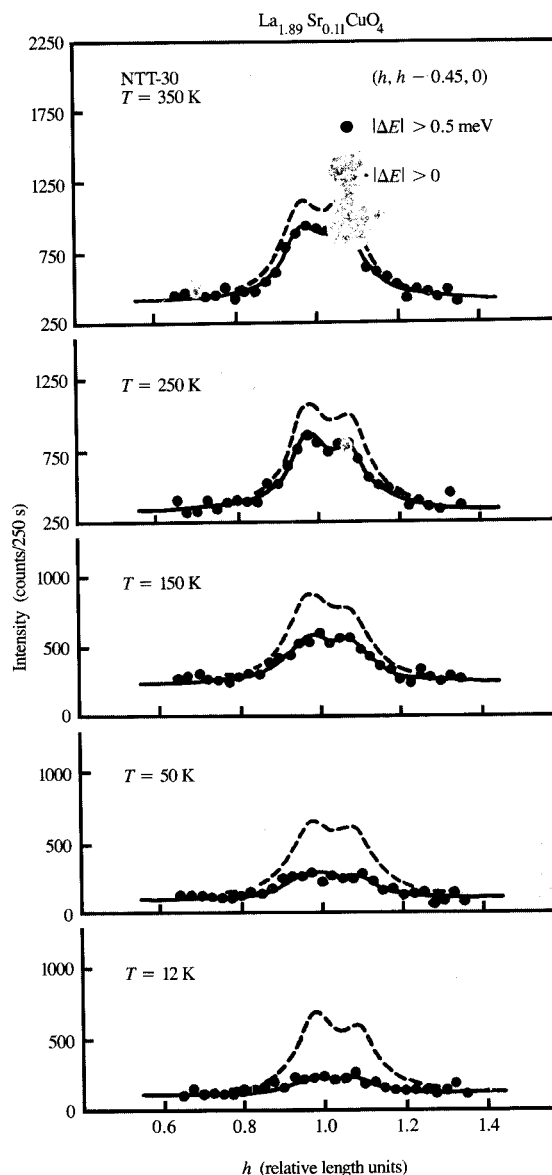


Figure 1

Integrated inelastic ($|\Delta E| > 0.5$ meV) scattering for scans across the magnetic ridge along $(h, h - 0.45, 0)$; $E_i = 14.7$ meV and the collimator configuration is $40'-40'-40'-80'$. The solid lines are the results of fits to two displaced 2D Lorentzians, as discussed in the text. The dashed lines are the result of the best fits to the total scattering, elastic plus inelastic.

meV has the feature that the outgoing neutron direction $\vec{k}_f/|k_f|$ is along $-\vec{b}^*$; that is, it is always perpendicular to the CuO_2 planes. The two-axis scan then automatically collects neutrons with all values of $|k_f|$ and thus integrates over

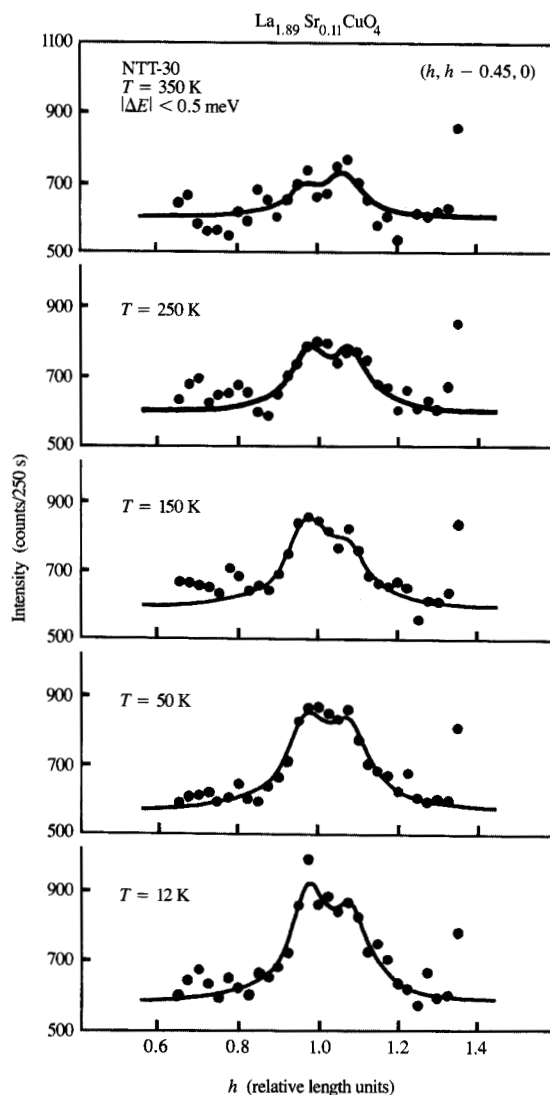


Figure 2

Three-axis ($|\Delta E| < 0.5$ meV) scans across the magnetic ridge along $(h, h - 0.45, 0)$; $E_i = 14.7$ meV and the collimator configuration is $40^\circ-40^\circ-80^\circ$. The solid lines are the results of fits to two displaced 2D Lorentzians with width and positions held fixed at the values determined from fits to the total cross section, elastic plus inelastic.

energy without varying the 2D momentum transfer $\vec{Q}_{2D}$. For the total scattering the energy integration range is from $-kT$ (neutron energy gain) to $+E_i$ (energy loss). This is illustrated in the uppermost panel of **Figure 3**, which shows an integrated inelastic scan (with $|\Delta E| > 0.5$ meV) with the 2D momentum transfer $\vec{Q}_{2D}$ held fixed at $1.05 \vec{a}^*$ and $\vec{Q}_\perp = k\vec{b}^*$ varied. This scan shows a well-defined peak at

$\vec{Q}_\perp \approx 0.6 \vec{b}^*$, at which point for $E_i = 14.7$ meV the condition that all outgoing neutrons propagate to the CuO planes is satisfied. This verifies that there is a substantial inelastic 2D

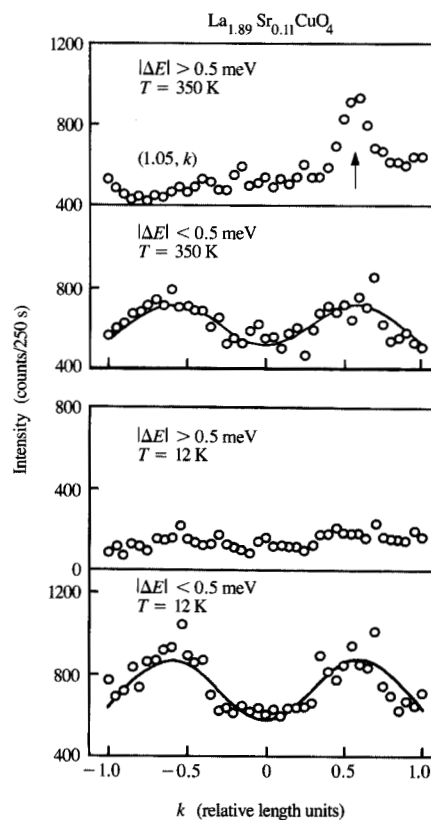
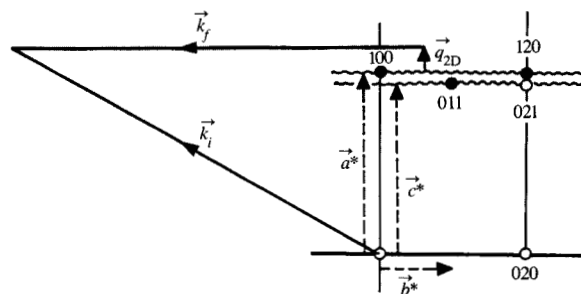


Figure 3

Top: Superposition of $\vec{a}^*-\vec{b}^*$ and $\vec{c}^*-\vec{b}^*$ reciprocal lattice planes together with a representative scattering diagram for $E = 14.7$ meV neutrons. Bottom: Elastic ($|\Delta E| < 0.5$ meV) and integrated inelastic ($|\Delta E| > 0.5$ meV) scans perpendicular to the CuO_2 sheets for $\vec{Q}_{2D} = 1.05 \vec{a}^*$. The arrow gives the position at which the outgoing neutron wavevector $\vec{k}_f$ is perpendicular to the CuO_2 sheets, that is, along $-\vec{b}^*$.

cross section at 350 K. This is confirmed by direct inelastic measurements, discussed below.

Figures 1 and 2 show the integrated inelastic ($|\Delta E| > 0.5$ meV), quasi-elastic ($|\Delta E| < 0.5$ meV), and fitted total cross sections for the $(h, h - 0.45, 0)$ scans across the 2D ridge at various temperatures. Two features are immediately evident. First, the scattering is broad and flat-topped, with some indication of a two-peaked structure. This incommensurate two-peaked structure was suggested in previous experiments [1] but was not established definitively. Second, the total cross section as measured in this particular cut through reciprocal space varies only weakly with temperature from 350 K to 12 K.

However, the low-energy spin fluctuations appear to change from being predominantly inelastic at 350 K to predominantly quasi-elastic at 12 K. We confirmed that the integrated intensity at room temperature is identical to within the errors ($\sim 20\%$) to the integrated 2D magnetic cross section for a sample of pure La_2CuO_4 ($T_N = 240$ K) measured under identical spectrometer conditions. Since the scattering near $h = 1$ comes predominantly from low energies, this implies that the full Cu^{++} moment or a significant part thereof is preserved in the superconducting samples. Figure 4 shows pure two-axis scans along $(h, h - 0.4, 0)$ across the ridge at $T = 20$ K (normal) and $T = 5$ K (superconducting), together with a background scan along $(h, -0.2, 0)$. It is evident that any change in the static structure factor, $S(\vec{Q})$, between the normal and superconducting states is below our limit of detectability.

We consider next the geometry of the quasi-elastic scattering. Its full Q -dependence is experimentally accessible, albeit with considerable uncertainty in the background. As is evident in Figure 2, the quasi-elastic scattering intensity increases gradually with decreasing temperature. Further, the quasi-elastic lineshape for the $(h, h - 0.45, 0)$ scan across the ridge is closely similar if not identical to that for the total cross section, and thence $S(\vec{Q})$. The overall geometry in $\vec{Q}$ -space, however, turns out to be quite elaborate. Figure 3 shows quasi-elastic scans at 12 K and 350 K in which $\vec{Q}_{2D}$ is held fixed at the peak position, $1.05 \text{ } \bar{a}^*$, and the momentum transfer perpendicular to the CuO_2 planes, $\vec{Q}_\perp = k\vec{b}^*$, is varied. As is evident in Figure 3, the quasi-elastic peak intensity exhibits a sinusoidal modulation perpendicular to the CuO_2 sheets at both 12 K and 350 K. The period of modulation is about two La_2CuO_4 unit cells. If this scattering is indeed magnetic, we would conclude that, even at temperatures as high as 350 K, the low-energy spin fluctuations in $\text{La}_{1.89}\text{Sr}_{0.11}\text{CuO}_4$ are fully three-dimensional in character. This contrasts markedly with the fluctuations in pure La_2CuO_4 , which are essentially two-dimensional above the Néel temperature [5]. Scans with $\vec{Q}_\perp \sim 0.5 \vec{b}^*$ and $\vec{Q}_{2D}$ varied typically give either a flat-topped or a double-peaked structure, with the incommensurability varying from ~ 0.05

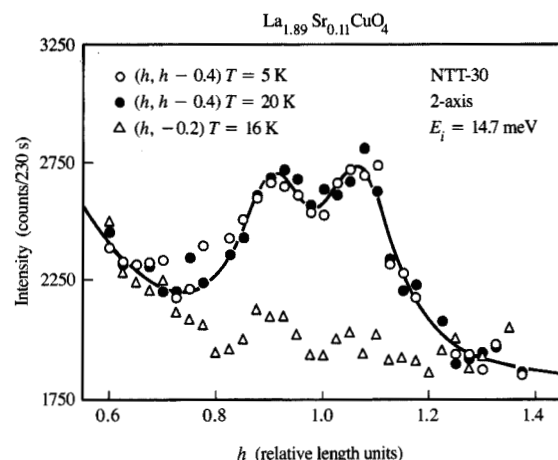


Figure 4

Pure two-axis scans along $(h, h - 0.4, 0)$ at $T = 5$ K and 20 K and along $(h, -0.2, 0)$ at $T = 16$ K. The spectrometer had one pyrolytic graphite filter and no analyzer. The solid line is the result of fits to two displaced 2D Lorentzians together with a background function determined from the $(h, -0.2, 0)$ scan.

$\bar{a}^*$ to $\sim 0.2 \bar{a}^*$ depending on the exact value of $\vec{Q}_\perp$. On the other hand, scans of $\vec{Q}_\perp$ at varying $\vec{Q}_{2D}$ all give the sinusoidal variation discussed above. Thus, at low temperatures the Cu^{++} structure factor in superconducting $\text{La}_{1.89}\text{Sr}_{0.11}\text{CuO}_4$ apparently corresponds to a slowly fluctuating ($< 10^{-11}$ s) 3D modulated spin fluid or glass. We note that recent μSR studies [2] on a sample prepared identically to ours indicate that the entire volume freezes magnetically below $T \sim 4$ K. Similar results in $\text{YBa}_2\text{Cu}_3\text{O}_{6.4}$ had been inferred earlier but could not be established definitively [2].

In order to compare these results with previous measurements [1], the data were fitted to several simple cross sections. The solid lines in Figures 1, 2, and 4 are the results of fits to two displaced 2D Lorentzians. Clearly this simple model works well, although it certainly is not unique. For the total scattering, the peak positions, intensities, and width, as well as the background, were all varied. For the quasi-elastic and integrated inelastic components, the peak positions and widths were fixed at the values determined from the fits to the total scattering, and only the intensities and background were allowed to vary. The quasi-elastic and integrated inelastic components turn out to be well described separately by the parameters characterizing the total cross section. The 2D instantaneous spin-spin correlation length is of order $18 \pm 6 \text{ } \text{\AA}$ independent of temperature from 350 K to 5 K. The 2D incommensurability from these fits is of order $0.05 \text{ } \text{\AA}^{-1}$ (Figure 2) to $0.08 \text{ } \text{\AA}^{-1}$ (Figure 4), although, as noted above, larger values are obtained from quasi-elastic

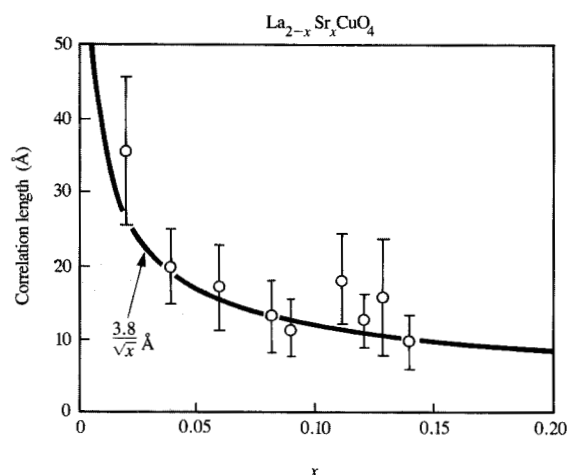


Figure 5

Instantaneous spin correlation length vs. temperature in $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$. The lengths are deduced from fits of two Lorentzians with identical widths symmetrically displaced about $\hbar = 1$. The solid line is the function $3.8/\sqrt{x}$ Å, which is just the average separation between the holes introduced by the Sr^{2+} doping.

scans perpendicular to the rod, so that the exact value of the incommensurability should be treated cautiously. At 350 K the total cross section, which corresponds to an integral from $-kT$ to $+14.7$ meV, is predominantly ($\sim 75\%$) inelastic, while at 12 K the $|\Delta E| < 0.5$ meV component accounts for $\sim 75\%$ of the observed scattering. We note that all of the data reported in [1] are also well described by the two-Lorentzian lineshape. As shown in Figure 5, the 2D correlation lengths deduced by this fitting procedure agree well with the average separation of the O^- holes, as suggested in [1].

3. Inelastic measurements

As reported in more detail in [7], we have made preliminary direct inelastic measurements of the spin excitations. Representative results at 6 meV are shown in Figure 6. The lineshape for excitation closely mirrors the integrated response (Figures 1, 3). Thus, these represent excitations out of the slowly fluctuating modulated ground state. The excitation creation intensity is independent of temperature between 300 K and 5 K; this spans the region $kT \gg \hbar\omega$ to $kT \ll \hbar\omega$. The excitation annihilation cross section is related to the creation process by the detailed balance factor $e^{-E/kT}$, as is required by time-reversal symmetry. It has been verified [7] that there is no dispersion in the $\tilde{b}$ direction at 6 meV; that is, the excitations are confined to the CuO_2 planes. This contrasts with the

fluctuations with $|E| < 0.5$ meV which appear to exhibit 3D sinusoidal correlations, as shown in Figure 3.

If the spin excitations were *bosons*, as in pure La_2CuO_4 below T_N [1], the intensity for $E = +6$ meV would have changed by a factor of 5 between 300 K and 5 K. On the other hand, the temperature dependence of the excitation intensity is only consistent with *Fermi* statistics if the chemical potential is much larger than 25 meV. However, since the correlation length is so short, the spin excitations are not well defined, and their statistics may therefore be complicated.

Limited measurements have also been performed [7] for energies varying between 4 meV and 18 meV. The lower limit of 4 meV is set by background considerations, while above 18 meV phonon-scattering processes dominate. The data at this point are incomplete, but they do show that the excitation intensity depends only weakly on energy. Because of the broad distribution in energy, the scattering at any given energy is quite weak. The count rate in Figure 6 at 6 meV is 6 counts per minute; further, NTT-35 is a high-quality single-crystal 2 cm^3 in volume. It is not surprising, therefore, that similar inelastic neutron-scattering experiments have not yet been successfully executed in other high-temperature superconductors.

Finally, we consider the relationship between the dynamic (Figure 6) and instantaneous spin correlations (Figures 1–4). The integrated inelastic measurements cover the energy range from $-kT$ to $+E_i$, with the center 1 meV excluded. At 350 K this corresponds to ~ -30 meV to $+14.7$ meV. Further, the energy gain part is amplified by the kinematical factor $k_f/k_i = (1 - \hbar\omega/E_i)^{1/2}$. At 12 K the energy integration range is from ~ 0.5 meV to 14.7 meV. Thus, the diminution of the integrated inelastic scattering with decreasing temperature evident in Figure 5 is simply a manifestation of detailed balance combined with the weak energy and temperature dependence of $S(\vec{Q}, \omega)$ at $+\omega$. Heuristically, the thermal spin excitations seem to condense out, yielding a slowly fluctuating, modulated spin fluid or glass response. Further, the slow part of $S(\vec{Q}, \omega)$ appears to be correlated three-dimensionally.

From both the temperature dependence of the intensity and the dispersion, one may conclude that the inelastic scattering is consistent only with magnetic processes rather than lattice dynamical fluctuations. It has not, however, been proven that the quasi-elastic scattering shown in Figures 2 and 3 is magnetic, and indeed there remains the possibility that part, if not all, of this scattering actually arises from complicated multiple-scattering events. However, the continuous trade-off intensity evident in Figure 1 between the dynamic and static fluctuations with decreasing temperature, as well as the closely similar lineshapes, is suggestive. Certain theories, such as that of [8], require a gap in the spin excitation spectrum in the superconducting state. Our results seem to contradict these predictions. However,

the low T_c value of 10 K in our samples implies considerable disorder in the CuO_2 planes, probably due to oxygen vacancies. It is possible that the quasi-elastic scattering originates from this disorder. Only future experiments on more perfect samples with T_c near 40 K can remove this ambiguity.

4. Conclusions

Clearly, the magnetism in these samples of superconducting $\text{La}_{1.89}\text{Sr}_{0.11}\text{CuO}_4$ with $T_c = 10$ K is quite intricate. The superconductivity occurs in the presence of a slowly fluctuating Cu^{++} spin fluid; the 2D correlation length is of order 18 ± 6 Å, while the 3D correlations appear to be sinusoidal in character, with a period of ~ 2 La_2CuO_4 unit cells. It seems clear heuristically that this novel spin state is generated by the O^- holes [9–11], which also carry the supercurrent. So far we have not succeeded in observing a direct manifestation of the superconductivity in the spin fluctuations. The gradual freezing of the Cu^{++} spin fluid as the temperature is decreased from 350 K to 5 K is, in our view, one of the most remarkable features of these results. It is important to emphasize that these experiments have confirmed unambiguously that there is a substantial Cu^{++} moment in superconducting samples, thence strengthening the case for a magnetic mechanism for the superconductivity in the lamellar CuO_2 superconductors.

Acknowledgments

We would like to thank our colleagues at MIT, Brookhaven National Laboratory, Tohoku University, and NTT for many helpful discussions of these results, and R. B. Laughlin for invaluable critical comments. This work was supported by the U.S.–Japan Cooperative Neutron Scattering Program, and a Grant-In-Aid for Scientific Research from the Japanese Ministry of Education, Science, and Culture. The work at MIT was supported by the National Science Foundation under Contract No. DMR85-01856 and Contract No. DMR87-19217. Research at Brookhaven National Laboratory was supported by the Division of Materials Science, U.S. Department of Energy, under Contract No. DE-AC02-CH00016.

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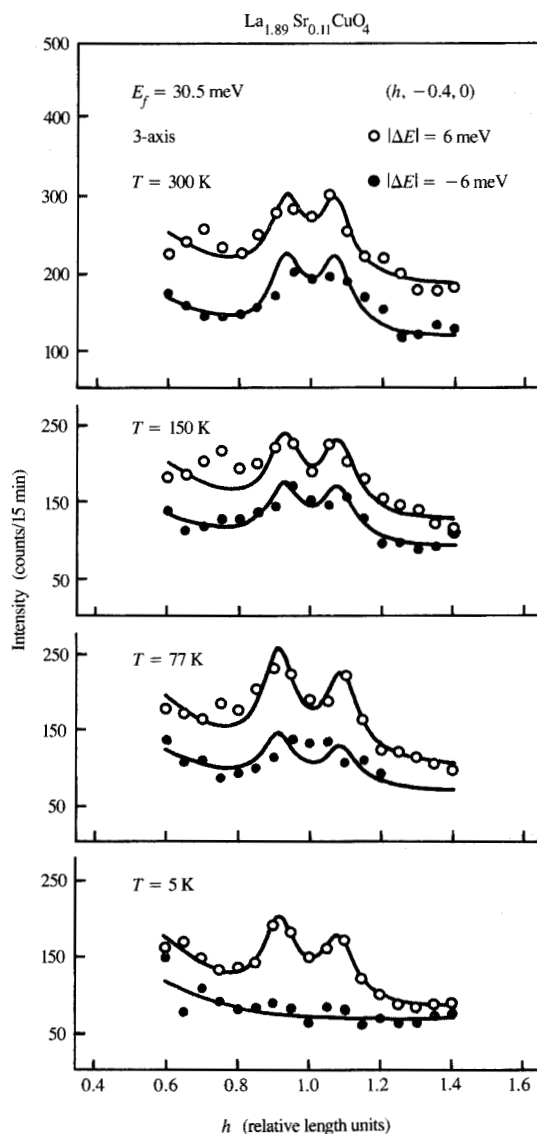


Figure 6

Constant energy scans across the 2D rod along $(h, -0.4, 0)$ in $\text{La}_{1.89}\text{Sr}_{0.11}\text{CuO}_4$. The outgoing neutron energy was 30.5 meV and the collimation was $40^\circ\text{--}40^\circ\text{--}80^\circ$. The lines for the +6 meV data are the results of fits to two symmetrically displaced Lorentzians. The lines for the -6 meV data are calculated from the +6 meV fits assuming detailed balance. The open circles represent excitation creation and the closed circles excitation annihilation scattering processes.

Received November 11, 1988; accepted for publication January 9, 1989

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