Muon-spin-relaxation and neutron-scattering studies of magnetism in single-crystal $La_{1.94}Sr_{0.06}CuO_4$

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Combined muon-spin-relaxation (μ^+SR) and elastic magnetic neutron-scattering experiments on a large single crystal of $La_{1.94}Sr_{0.06}CuO_4$ are reported. μ^+SR data taken without an external field indicate that the Cu spins are fluctuating slowly or frozen below $T_f^{\mu} \approx 6.0$ K. At 3.9 K, measurements done with an applied longitudinal field show that the muon depolarization is almost completely due to static moments. Neutron measurements of the magnetic peaks $\mathbf{Q}=(1,0,0)$ and $(0,1,1)$ show that no three-dimensional long-range order is present down to 1.8 K. However, the quasielastic scattering intensity from the two-dimensional ridge $\mathbf{Q}=(1,k,0)$ at $k=-0.12$ increases sharply between 40 and 20 K. Below $T_f^N=20$ K the scattering intensity is roughly constant. The difference between these two freezing temperatures is explained in terms of the different range of fluctuation frequencies to which each of these probes is sensitive. The dependence of the freezing temperature on the frequency windows of different probes is similar to that seen in traditional spin-glass materials like *Cu*Mn.

I. INTRODUCTION

The magnetic properties of pure and doped La_2CuO_4 have been the subject of intense study since the discovery of superconductivity in $La_{2-x}Ba_xCuO_4$ by Bednorz and Müller.[1] Many authors have shown that adding holes to pure La_2CuO_4, by substituting Ba^{2+} or Sr^{2+} for La^{3+} or through the addition of oxygen, rapidly suppresses the antiferromagnetic transition temperature T_N. In the case of $La_{2-x}Sr_xCuO_4$, strontium concentrations as low as $x=0.02$ completely destroy the long-range ordered state.[2] However, NMR (Refs. 3 and 4) and muon-spin-relaxation[5-8] (μ^+SR) studies have shown that some sort of magnetic transition occurs in $La_{2-x}Sr_xCuO_4$ over a broad range of doping concentrations $0.02 \leq x \leq 0.15$. Of particular interest is the magnetic behavior of $La_{2-x}Sr_xCuO_4$ samples that evince neither superconducting or Néel states. In this border region, $0.02 \lesssim x \lesssim 0.06$, the phenomenological model proposed by Aharony *et al.*[9] predicts that magnetic frustration due to the presences of holes results in a spin-glass-like state. To investigate this behavior we have performed a number of measurements on a single large crystal of $La_{1.94}Sr_{0.06}CuO_4$ (labeled NTT-10 in Ref. 2) using both μ^+SR and neutron-scattering techniques. In this report we focus on the dynamics of the magnetism found in NTT-10.

II. CRYSTAL PREPARATION

The crystal studied in this paper was grown by Hidaka *et al.*[10] at the NTT Electrical Communications Laboratories using a CuO flux method. High-purity La_2O_3, $SrCO_3$, and CuO powders were mixed in a platinum crucible and heated at 1300 °C for 2 h and then slowly cooled at 3 °C/h. When a flux ratio of 80% was used, crystal formation was observed around ~1100 °C. After several hours of further cooling these crystal products were removed from the flux and cooled to room temperature at 100 °C/h. In the particular case of NTT-10 the resulting sample volume was approximately 0.5 cm^3. The strontium concentration x was determined by fluorescent x-ray analysis to be roughly 0.06.[10]

III. μ^+SR MEASUREMENTS

μ^+SR is an extremely sensitive method of measuring *local* magnetic fields at interstitial sites in solids.[11] Muons, initially 100% polarized, are implanted (range

≈ 170 mg/cm^2) in a target material. After coming to rest at a given site, each μ^+ precesses in the local magnetic field produced by both internal and external sources ($\omega = \gamma_\mu |\mathbf{B}_{\rm loc}|$, where $\gamma_\mu / 2\pi = 13.55$ kHz/G and $\mathbf{B}_{\rm loc} = \mathbf{B}_{\rm int} + \mathbf{B}_{\rm ext}$). The muon decay positrons, which are emitted preferentially in the direction of the μ^+ spin, can then be used to record the evolution of the μ^+ polarization as a function of time.

Magnetic phenomena in a solid can be characterized by applying the μ^+SR technique in the absence of an external field (ZF-μ^+SR). In ZF-μ^+SR, $\mathbf{B}_{\rm loc} = \mathbf{B}_{\rm int}$ and the resulting muon decay time spectra directly reflect the distribution of the internal fields seen at the various muon sites. For static moments the muon spectra yield information on both the magnitude of the local fields and the degree of randomness present in the local spatial order. If the distribution of internal fields at each muon site is sharply peaked, as in ferromagnetically or antiferromagnetically ordered materials, the muons at a given site precess coherently and contribute a long-lived oscillatory signal to the total spectrum. In materials in which the moments are frozen in a disordered spin configuration, so that a broad distribution of fields exits at each muon site, no coherent precession of the muon polarization occurs and the resulting spectrum decays rapidly.

ZF-μ^+SR is also sensitive to the presence of dynamic fields. For rapidly fluctuating moments the dynamic relaxation rate (corresponding to the T_1 relaxation of NMR) can be written as

$$\frac{1}{T_1} \sim \langle \omega^2 \rangle \tau_c \ , \qquad (1)$$

where $\langle \mathbf{B}(0)\mathbf{B}(t) \rangle \propto e^{-t/\tau_c}$ and $\omega = \gamma_\mu |\mathbf{B}|$. Since the relaxation rate is inversely proportional to the fluctuation frequency $\nu \sim 1/\tau_c$, fast fluctuations effectively "wash out" the randomness of the local-field distribution so that the resulting relaxation rate is small. On the other hand, the μ^+SR spectrum resulting from slowly fluctuating fields ($\nu \lesssim \omega$) is, at short times, nearly identical with that due to random static fields.[12] The two can be distinguished only at longer times, where the asymmetry in the presence of fluctuating moments continues to decay, whereas the asymmetry in the presence of static fields recovers to an asymptotic value, typically $\frac{1}{3}$ of the initial asymmetry. However, background levels, statistics, and the possible presence of other relaxation mechanisms can sometimes make this resolution difficult in practice. Hence, the muon polarization is sensitive to dynamic fields in a bounded frequency "window." Measurements of the muon precession rate in pure La_2CuO_4 well below T_N indicate that the internal field strength at the muon sites is on the order of 400 G.[13] With fields of this magnitude, only fluctuations in the range 10^6 Hz $\lesssim \nu \lesssim 10^{11}$ Hz produce observable depolarization within the time scale of μ^+SR measurements.

In the absence of either static or dynamic electronic moments, the muon polarization experiences only weak nuclear dipole magnetic fields which result in a comparatively small rate of depolarization that is easily distinguished from the relaxation associated with ordered or fluctuating electronic moments.

The μ^+SR experiments reported here were performed at the TRIUMF M20B surface muon channel. The sample was mounted, with the crystal **c** axis parallel to the initial muon polarization and momenta, in a He^4 gas-flow cryostat with mylar windows. Positron counters were positioned up and downstream of the sample volume. Muons that stop in the aluminum sampler holder and cryostat also contribute to the measured asymmetry. However, as the muon polarization is unaffected by aluminum, this background is flat and does not affect the shape of the experimental spectra. Measurements performed in a weak transverse field indicate that this background constitutes 20(1)% of the total asymmetry.

The ZF-μ^+SR spectra from NTT-10 at $T = 10$, 7.5, 6, and 3.9 K are shown in Fig. 1. In additional ZF data taken at 45, 35, and 25 K, a slow, temperature-independent relaxation of the muon polarization was found. As the depolarization due to fluctuating electronic moments is statistically independent from that due to nuclear dipolar broadening, the spectra at all three temperatures were fit with the product of a Gaussian signal, to account for the nuclear dipolar broadening, and an exponential signal, $A_0 \exp(-\lambda t)$, to reflect the relaxation due to fluctuating Cu moments. For all three spectra $\lambda \approx 0.02(2)$ μs^{-1}, which indicates that the fluctuation frequencies in this temperature regime are not within the range of the muon's dynamic sensitivity. At 15 and 10 K similar fits resulted in a slightly larger depolarization rate, $\lambda = 0.04(1)$ μs^{-1}, suggesting a shift in the spectral weight of the field fluctuations into the upper range of the muon's dynamic depolarization window. The relaxation rate starts to change noticeably at 7.5 K, where $\lambda = 0.09(1)$ μs^{-1}. Assuming the local-field strength in $La_{1.94}Sr_{0.06}CuO_4$ is of the same order of magnitude as that found in pure La_2CuO_4, a rough estimate of the fluctuation rate at 7.5 K, using Eq. (1), yields a value of $\nu \sim 10^{10}$ Hz. In the 6 K data the sharp initial relaxation and the gentle relaxation at long times cannot be attributed to dynamic (T_1) effects alone. The shape of the 6 K spectrum strongly suggest that some static fields are present. In the following we refer to the μ^+SR freezing temperature, $T_f^\mu \approx 6.0$ K, loosely defined as the temperature below which evidence of a contribution from static moments to the ZF depolarization rate can be seen.

In the data taken at 3.9 K, the absence of long-lived oscillations, and the dip and recovery seen in the first $\frac{1}{2}$ μs, are quite similar to features of the Kubo-Toyabe relaxation function for a Gaussian distribution of random static fields[14]

$$G_z(t,\Delta) = \tfrac{1}{3} + \tfrac{2}{3}(1-\Delta^2 t^2) e^{-(1/2)\Delta^2 t^2} \ , \qquad (2)$$

where

$$\rho(B_i) = \frac{\gamma_\mu}{\sqrt{2\pi}\Delta} \exp\left[-\frac{\gamma_\mu^2 B_i^2}{2\Delta^2}\right], \quad i = x,y,z \ .$$

This suggests that the depolarization at 3.9 K is due mostly to the presence of static moments, and that these moments are spatially disordered. This is in strong contrast to the ZF-μ^+SR signal found in pure La_2CuO_4

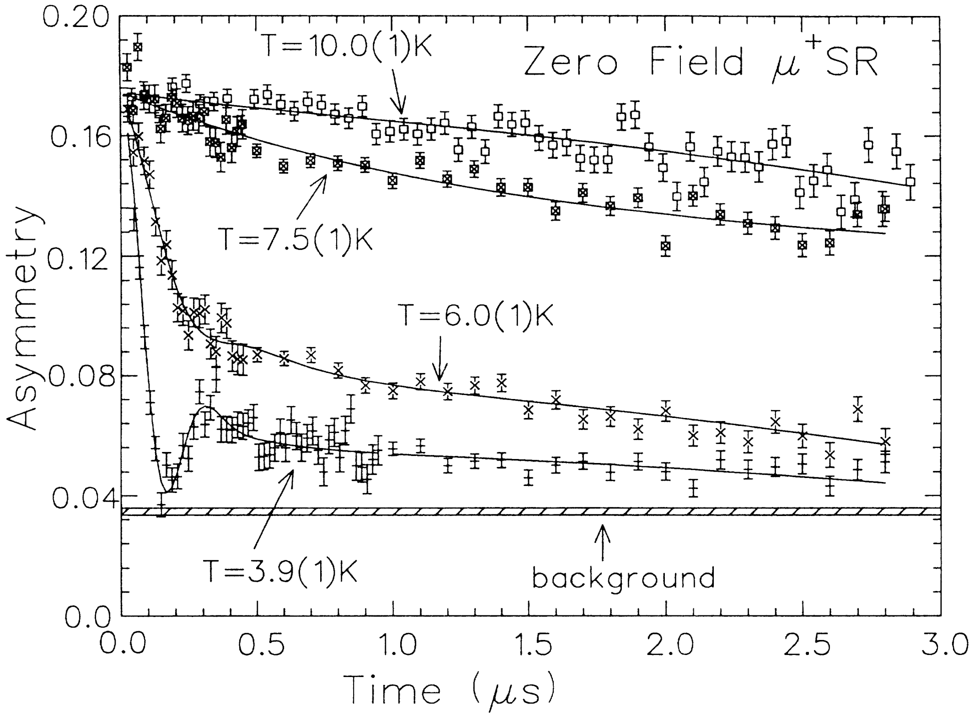

FIG. 1. Zero-field muon-spin-relaxation spectra in NTT-10. The relaxation at 10 and 7.5 K can be attributed to fluctuating local fields. The increase in the relaxation rate at $T_f^\mu=6.0$ K is due to the onset of spin freezing. By 3.9 K, fits indicate that the muon depolarization is mostly due to static fields.

where long-lived oscillations have been seen at temperatures below T_N.[13] The 3.9 K data was fit using the product of Eq. (2) and a plain exponential (for any depolarization due to fluctuating moments), plus a constant signal to account for the aluminum background. A value of 122(3) G for $\Delta/\gamma_\mu=\langle B_i^2\rangle^{1/2}$ produced the best fit to the data, which, with the above field distribution, implies a static field strength of

$$\langle|B_{\mathrm{loc}}|\rangle=\sqrt{8/\pi}\Delta/\gamma_\mu=196(7)\ \mathrm{G}\ .$$

The difference between this result and the field strength measured in La_2CuO_4 may be due to both the magnetic frustration introduced by Sr^{2+} doping and the fact that, as $T=3.9$ K is still relatively close to the freezing temperature, the order parameter (defined by the single-spin time correlation[12]) has not yet reached its ground-state value.

To resolve better the static and the dynamic behavior of the magnetism in NTT-10 at 3.9 K, data were taken with an external field applied parallel to the initial spin polarization. This longitudinal-field geometry (LF-μ^+SR) allows the depolarization due to dynamic or fluctuating moments to be decoupled from that due to static moments. The dependence of the decoupling on the applied field strength provides a measure of the magnitude of the internal static random fields.[15] The spectra from LF-μ^+SR on NTT-10 at 3.9 K for $|\mathbf{B}_{\mathrm{ext}}|=0$, 290, 550, and 2100 G are shown in Fig. 2. As can be seen in the figure, approximately half the asymmetry at early times is recovered when $|\mathbf{B}_{\mathrm{ext}}|=290$ G. This indicates that most of depolarization, but a comparison with data taken at temperatures well above T_f^μ indicates that some residual dynamic depolarization, due to fluctuating moments, remains present at 3.9 K. Fits of the 290 and 550 G data, to the product of the appropriate relaxation function for a Gaussian distribution of random fields centered about an applied longitudinal field [Eq. (10) in Ref. 15] and an exponential to account for depolarization due to fluctuating fields, resulted in values for Δ/γ_μ of 232(23) and 222(68) G, respectively, roughly consistent with the value of Δ/γ_μ found above.

IV. NEUTRON-SCATTERING MEASUREMENTS

Quasielastic magnetic neutron scattering provides information on the dynamics of magnetic fluctuations in a different frequency range from that measured by μ^+SR. By adjusting the spectrometer energy resolution ΔE the frequency range of the magnetic fluctuations contributing to the quasielastic scattering intensity can be chosen via the relation $\nu_{\mathrm{qe}}\lesssim\Delta E/h$. In addition, as neutron scattering is a Q-space probe, one can also study the actual spatial spin structure of materials.

A great deal of neutron work has been done on the $La_{2-x}Sr_xCuO_4$ system.[16] In the temperature and Sr concentration regime of interest, $La_{2-x}Sr_xCuO_4$ is orthorhombic with space-group Cmca. Early work on the isostructural compound K_2NiF_4 (Ref. 17) showed that

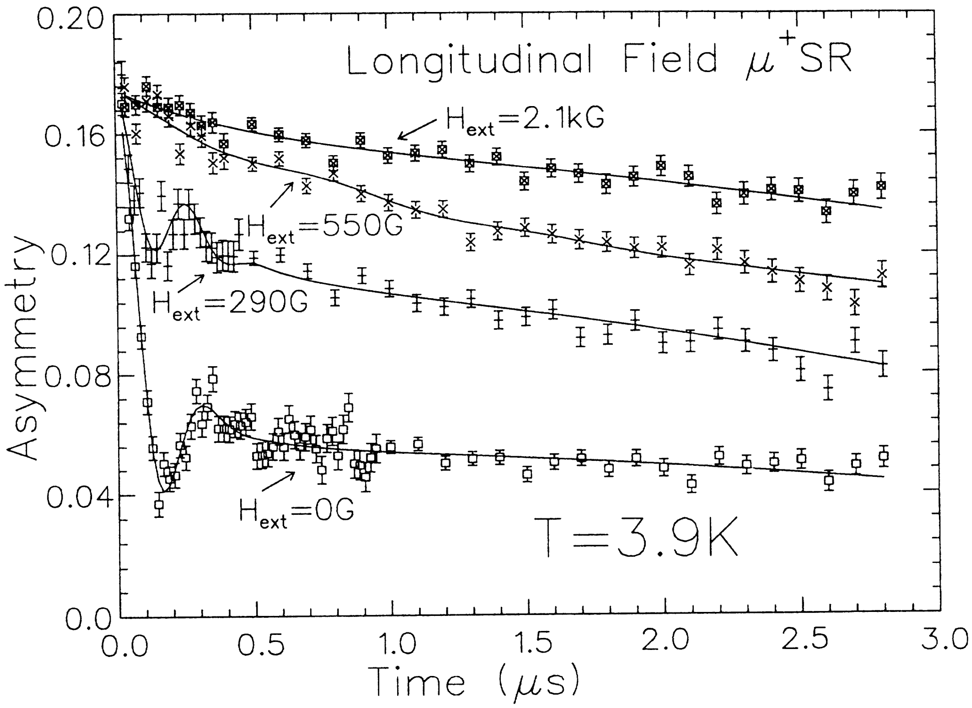

FIG. 2. Longitudinal-field muon-spin-relaxation spectra in NTT-10 at 3.9 K. The application of an external pinning field decouples the depolarization due to static and dynamic fields. Fits to the 2900 and 550 G data resulted in values for Δ/γ_μ (see text) of 232(23) and 222(68) G, respectively.

strong two-dimensional (2D) spin fluctuations were present at temperatures down to the Néel temperature T_N, below which a transition to a three-dimensional (3D) ordered state occurs. As soon as large single crystals became available Shirane *et al.*[18] found similar 2D behavior above T_N in La_2CuO_4. A diagram of the $\mathbf{a}^*(\mathbf{c}^*)$-$\mathbf{b}^*$ scattering plane illustrating the location of 2D magnetic scattering rods and the 3D magnetic and nuclear peaks in La_2CuO_4 is shown in the inset in Fig. 3.

Of particular importance to this paper are the studies done by Birgeneau *et al.*[2] on antiferromagnetic spin correlations in NTT-10 (in addition to other $La_{2-x}Sr_xCuO_4$ crystals). In this work the authors reported an increase in the "two-axis" ($\int dE$) scattering intensity due to instantaneous 2D spin correlations with decreasing temperature. In order to investigate the relationship between these 2D correlations and our μ^+SR results we have done an extended study of the quasielastic component of this 2D scattering as a function of temperature.

The differential cross section for the magnetic scattering of neutrons[19] can be written as

$$\frac{\partial^2\sigma}{\partial\Omega\,\partial E}=A(\mathbf{k},\mathbf{k}')\sum_{\alpha\beta}(\delta_{\alpha\beta}-\hat{Q}_\alpha\hat{Q}_\beta)S^{\alpha\beta}(\mathbf{Q},\omega)\ ,$$

where

$$S^{\alpha\beta}(\mathbf{Q},\omega)=\frac{1}{2\pi}\int_{-\infty}^{\infty}dt e^{i\omega t}\sum_{\mathbf{r}}e^{i\mathbf{Q}\cdot\mathbf{r}}\langle S_0^\alpha(0)S_{\mathbf{r}}^\beta(t)\rangle\ ,$$

and

$$A(\mathbf{k},\mathbf{k}')=\frac{N}{\hbar}(r_0\gamma)^2\frac{k'}{k}|f(\mathbf{Q})|^2\ .$$

To a first approximation, the quasielastic intensity with energy window $\pm\Delta E$ is given by

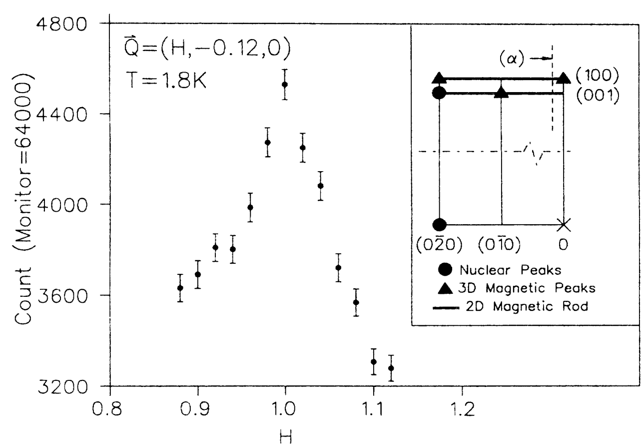

FIG. 3. (Inset) A diagram of the $\mathbf{a}^*(\mathbf{c}^*)$-$\mathbf{b}^*$ scattering plane illustrating the location of the 2D magnetic scattering rods and the 3D magnetic and nuclear peaks in La_2CuO_4. Because of twinning the ⟨100⟩ and ⟨001⟩ axes are simultaneously present. The gap between $\mathbf{a}^*$ and $\mathbf{c}^*$ has been exaggerated to illustrate better the various magnetic features found in La_2CuO_4. (Main) Results of a quasielastic scan across the (2D) Bragg rod $\mathbf{Q}=(h,-0.12,0)$, the dashed line α at 1.8 K. This ridge reflects the presence of strong, slowly fluctuating or static (2D) spin correlations.

$$\left.\frac{d\sigma}{d\Omega}\right|_{\Delta E} \sim S^{\alpha\beta}_{\Delta E}(\mathbf{Q}) = \int_{-\Delta E/\hbar}^{\Delta E/\hbar} S^{\alpha\beta}(\mathbf{Q},\omega)d\omega \ .$$

Hence, the quasielastic magnetic scattering provides a direct measure of the spin order which is static on the time scale of $t \sim \hbar/\Delta E$. In materials with only 2D spin-spin correlations the magnetic cross section is independent of the momentum transfer normal to the ordered planes so that the quasielastic magnetic scattering is distributed along Bragg "rods" in reciprocal space. If a transition to 3D long-range order occurs this scattering intensity is transferred to discreet Bragg peaks.

Here we report our measurements of the quasielastic scattering from the 2D Bragg rod $\mathbf{Q}=(1,k,0)$ in NTT-10 for temperatures between 1.8 and 100 K. The measurements were done at the Brookhaven High Flux Beam Reactor using the H7 triple-axis spectrometer with 40′-40′-40′-80′ collimation, pyrolytic graphite (PG) monochromator and analyzer, and PG filters both before and after the sample to remove $\lambda/2$ contamination. All measurements were made using 13.7-meV neutrons. With this energy and collimation, the H7 spectrometer has a HWHM energy resolution of $\Delta E = 1$ meV over the range of Q space studied. Consequently, all fluctuations with frequencies $\nu \lesssim 1$ meV$/h \sim 10^{12}$ Hz contributed to the quasielastic scattering intensity. The crystal was mounted in an aluminum container filled with helium-exchange gas, and attached to the cold finger of a pumped He^4 cryostat.

Results of an elastic scan across the 2D rods $\mathbf{Q}=(1,-0.12,0)$ and $(0,-0.12,1)$ [corresponding to the dashed line (α) in Fig. 3 (inset)] at our base temperature of 1.8 K are shown in Fig. 3. As mentioned above, this quasielastic ridge reflects the presence of static or slowly fluctuating 2D correlations. The broad width in Q space of the 2D magnetic scattering peak indicates that the in-plane spin-spin correlation length is quite short as observed previously in Ref. 2. Below we discuss the temperature dependence of the peak and off-peak intensities. More detailed studies of the width of the 2D quasielastic scattering ridge, or equivalently the 2D static correlation length, as a function of temperature are in progress.

In Fig. 4 we show the measured scattering intensity from both the peak of the 2D elastic ridge, $\mathbf{Q}=(1,-0.12,0)$, and a point significantly below the peak, $\mathbf{Q}=(0.88,-0.12,0)$, for temperatures between 1.8 and 100 K. As can be seen in Fig. 4, the off-peak scattering remains roughly temperature independent, consistent with previous results that no major change in the width of the spectral shape in Q space, or the correlation length, occurs over the temperature range studied. In contrast, the peak intensity increases sharply between 40 and 20 K. This shows that the spectral weight of the 2D fluctuations starts to move below 10^{12} Hz around 40 K. Below 20 K the peak scattering intensity is roughly constant. This motivates the definition of a neutron freezing temperature, $T_f^N=20$ K, since the moments in NTT-10 are frozen with respect to the quasielastic scattering cutoff frequency, 10^{12} Hz, at temperatures below T_f^N. In combination with the absence of any elastic scattering intensity at 1.8 K from the 3D magnetic Bragg peaks $\mathbf{Q}=(1,0,0)$ and $(0,1,1)$, this result argues that while significant 2D freezing is evident below T_f^N, no transition to a 3D long-range ordered state occurs above 1.8 K; the quasielastic spin correlations exist only within the CuO_2 planes.

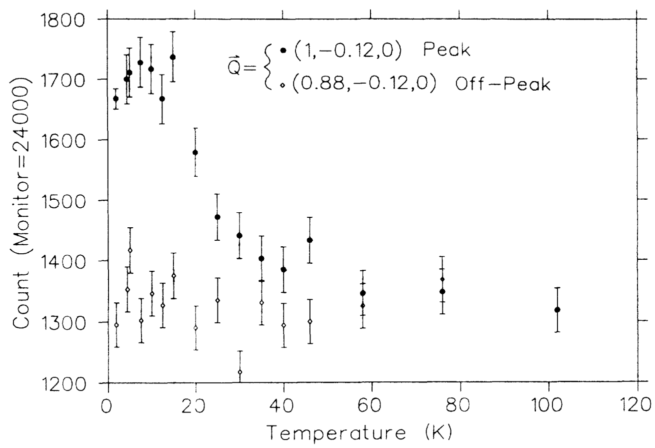

FIG. 4. The quasielastic scattering intensity from both the peak of the 2D scattering ridge, $\mathbf{Q}=(1,-0.12,0)$, and a point below the peak, $\mathbf{Q}=(0.88,-0.12,0)$, as a function of temperature. The rapid increase in the peak intensity between 40 and 20 K reflects the onset of 2D spin freezing. Below $T_f^N=20$ K the fluctuations have frozen with respect to the neutron quasielastic scattering window of $\sim 10^{-12}$ Hz.

V. DISCUSSION AND CONCLUSIONS

The difference between T_f^N and T_f^μ, the neutron and μ^+SR transition temperatures in NTT-10, can be explained in terms of the different frequency windows associated with these measurements and the gradual temperature dependence of the slowing down and/or freezing of the spin fluctuations. Between 40 and 20 K our neutron data indicate that the distribution in frequency space of the 2D spin fluctuations shift from being totally outside to being totally inside the quasielastic scattering window bordered by 10^{12} Hz. As the temperature is further reduced the spin fluctuations continue to slow down so that, at 3.9 K, a majority of the fluctuations have frozen with respect to the muon static cutoff frequency, 10^6 Hz. This gradual slowing down of spin fluctuations over a wide temperature range in NTT-10 is quite similar to the temperature dependence of the spin fluctuations seen in spin-glass materials like *Cu*Mn.[12,20] The slow freezing of magnetic fluctuations is generally considered to be a fundamental feature of the dynamics of classic spin-glass systems. An equally important characteristic of canonical spin glasses is the temperature independence of the spectral shape in reciprocal space.[21] In the present experiment our preliminary results on the peak and off-peak scattering from the 2D elastic ridge of the NTT-10 crystal as well as previous results suggest that the correlation length in $La_{1.94}Sr_{0.06}CuO_4$ also in not strongly temperature dependent.

In principle, results similar to the present μ^+SR data might be seen in an inhomogeneous antiferromagnet with a broad range of Néel temperatures. However, the absence of 3D magnetic Bragg scattering in our neutron data demonstrates that this is not possible. We may also conclude that the data cannot be explained by a broadened freezing into a nearly static short-range ordered magnetic state since, in the present measurements, this would have resulted in similar muon and neutron freezing temperatures. Therefore, the difference between T_f^μ and T_f^N in our results clearly shows that the magnetism in NTT-10 does not reflect a simple spatial inhomogeneity of the Sr or oxygen concentrations.

In conclusion, the combined μ^+SR and neutron-scattering results on the $La_{1.94}Sr_{0.06}CuO_4$ crystal studied here have demonstrated that the spin dynamics of a compound in the compositional region between the antiferromagnetic and superconducting phases of $La_{2-x}Sr_xCuO_4$ has features characteristic of spin-glass systems.

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