Anelastic relaxation and $^{139}$LaNQR in La$_{2-x}$Sr$_x$CuO$_4$ around the critical Sr content x=0.02

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Abstract. Anelastic relaxation and $^{139}$La NQR relaxation measurements in La$_{2-x}$Sr$_x$CuO$_4$, for Sr content x around 2 and 3 percent, are presented and discussed in terms of spin and lattice excitations and ordering processes. It is discussed how the phase diagram of La$_{2-x}$Sr$_x$CuO$_4$ at the boundary between the antiferromagnetic (AF) and the spin-glass phase (x = 0.02) could be more complicated than previously thought, with a transition to a quasi-long range ordered state at $T = 150$ K, as indicated by recent neutron scattering data. On the other hand, the $^{139}$LaNQR spectra are compatible with a transition to a conventional AF phase around $T = 50$ K, in agreement with the phase diagram commonly accepted in the literature. In this case the relaxation data, with a peak of magnetic origin in the NQR relaxation rate around 150 K at 12 MHz and the anelastic counterparts around 80 K in the kHz range, yield the first evidence in La$_{1.98}$Sr$_{0.02}$CuO$_4$ of freezing involving simultaneously lattice and spin excitations. This excitation could correspond to the motion of charged stripes.

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1 Introduction

From a variety of recent experiments and theoretical descriptions (mostly motivated by the search of the microscopic mechanism underlying high-temperature superconductivity), it has been realized that the electron system in doped two-dimensional (2D) quantum Heisenberg antiferromagnets (AF) exhibits complicated ordering phenom-
ena. On cooling from high temperatures, first a kind of phase separation is expected to occur, causing the formation of charged stripes separating mesoscopic AF domains \[1,2,3\]. In cuprates in general the stripes should exist only dynamically, with slowing down of their fluctuations on cooling. At lower temperatures the spin degrees of freedom associated to the AF patches \[4\] between the stripes are known to freeze, generating a cluster spin-glass state \[5,6,7\]. Charge and spin freezing both involve a complex spin dynamics. While the stripe dynamic is slow, the motion of the holes along the stripe is much faster than the fluctuation of the stripe itself.

There is also evidence of unusual coupling of the lattice to charge and spin excitations [3]. For instance, the \[^{139}\text{La}NMR\] line broadening, for \(T \leq 40 \text{ K}\), in \(\text{La}_{2-x}\text{Sr}_x\text{CuO}_4\) (LSCO) for \(x = 0.12\) (a signature of modulated magnetic order) is accompanied by softening of sound velocity [3]. Neutron diffraction, for \(0 \leq x \leq 0.3\), indicates local tilts of octahedra, interpreted as evidence of charged stripes [10], the local tilt decreasing with increasing \(x\). The local tilts give also rise to tunneling systems, observed by acoustic experiments for \(x \leq 0.03\), and relaxation rate strongly depends on doping [11].

Charge localization along stripes and spin freezing have been studied, in \(\text{La}_2\text{CuO}_4\)-based compounds, mostly by means of NMR-NQR and \(\mu\)SR spectroscopies, which probe the low frequency excitations through the relaxation times and the modifications in the spectra [3]. In particular, it has been argued [13] that, in the underdoped regime of LSCO, when diffraction experiments indicate complete ordering, the stripes are still fluctuating at low frequencies. LSCO at \(\text{Sr}\) content around 0.02 is interesting, being at the boundary between the 3D-AF and the spin-glass phase (see Ref. [4] for a review). A recent neutron scattering study has shown that quasi-3D magnetic ordering occurs below about 40 K in the spin-glass state, with a spin structure related to the diagonal stripe structure [5]. This new intermediate magnetic state is believed to result from partial freezing of 2D spin fluctuations existing at high temperatures, the spin-glass state being described as a random freezing of quasi-3D spin clusters with anisotropic spin correlations.

Motivated by this scenario of interrelated lattice and spin fluctuation effects, we have undertaken a comparative study of LSCO at \(x = 0.02\) and \(x = 0.03\) based on anelastic relaxation, \[^{139}\text{La}NQR\] relaxation and NQR spectra.

We first qualitatively recall how slowing down of spin fluctuations and ordering are expected to affect nuclear and mechanical relaxation. Single holes or charged stripes motions cause a time dependence in the hyperfine field \(\mathbf{h}(t) = \sum_i \mathbf{A}_i \mathbf{S}_i(t)\) at the nucleus (\(\mathbf{S}_i\) spin operator at the \(i\)-th ion, \(\mathbf{A}_i\) hyperfine coupling tensor). When a characteristic frequency \(\omega_s\) of the fluctuating stripes becomes of the order of the measuring quadrupole frequency \(\omega_m\), a maximum in the spin lattice relaxation rate \(T_1^{-1} = W\) driven by the local time dependence of \(\mathbf{h}(t)\) is expected. Below this temperature the stripes move very slowly, or are "pinned", and an "anomalous" magnetic moment is induced, associated to the 2D patches of AF correlated ions in between stripes [3]. The randomly distributed mag-
netic moments $\mu_i$ experience cooperative slowing down and a second relaxation mechanism sets in, related to the field at the nuclear sites due to $\mu_i$’s. In the correspondent relaxation rate a correlation function of the form $(\sum_{i,j} \mu_i(0)\mu_j(t)) = \sum_t \langle \mu_i(0)\mu_i(t) \rangle$ is involved.

One can empirically write this correlation function as $\mu^2 \exp[-t/\tau_f(x,T)]$, with an average correlation time $\tau_f$ which increases on decreasing temperature. At the temperature $T_g$ where $\tau_f$ becomes of the order of $\omega_m^{-1}$ another peak appears in $T_1^{-1}$, with non-exponential recovery law, a signature of disordered systems. Below $T_g$ one speaks of spin freezing. In a long-range ordered AF matrix $T_g$ should increase about linearly with $x$, due to the increased strength of the interaction among the $\mu_i$’s. On the contrary, for an amount of doping $x$ which destroys the long range AF order, with the onset of a cluster spin-glass phase, $T_g$ decreases with increasing $x$ [12,16,17].

In particular, in LSCO, for $x \leq 0.02$ the peaks in $^{139}$LaNQR $W$ have been shown [1] to follow the law $T_g = T_f = bx$, indicating spin freezing in an AF matrix. For $x \geq 0.02$ (cluster spin-glass phase) the peaks at $T_g(x)$ have been attributed to the spin freezing of the magnetic moments in short-range AF correlated islands [13].

The stripe localization has been detected indirectly from the wipe-out effect on the $^{63}$CuNQR signal in Nd and Eu doped LSCO at $x = 0.12$, at $T_{charge} = 65$ K (and in the underdoped regime of LSCO), with a wipe out fraction having a temperature behavior similar to the charge and spin order determined by neutron scattering [13,18]. It should be remarked that in principle a wipe out effect should be accompanied, at higher temperature, by a marked enhancement in the spin lattice relaxation rate, when $\omega_s = \omega_m$. Finally, $\mu$SR and $^{139}$LaNQR measurements [7,19] pointed out a magnetic transition to a spin-glass like phase well extending into the superconducting regime.

As regards the effects expected in the anelastic relaxation, one notes that the elastic energy loss coefficient $Q^{-1}$, measured by exciting flexural vibrations, is directly proportional to the imaginary part of the mechanical susceptibility $\chi''(\omega)$. Thus $Q^{-1}$ is related to the spectral density $J_{latt}(\omega)$ of the motions causing dissipation, according to the law $Q^{-1} \propto \chi'' \propto \omega J_{latt}$.

Since the motions of the stripes involve sizeable lattice effects, when a characteristic frequency decreases down into the kHz range they can be detected as maxima in $\chi''/\omega$. Thus, from a combination of anelastic relaxation and of magnetic NQR relaxation, one can in principle probe the lattice and the spin fluctuations associated to the stripe motions. The investigation reported here was aimed at this purpose.

### 2 Experimentals results and discussion

Two LSCO ceramic samples grown by standard solid state reaction [20] have been investigated. According to x-ray diffraction the final amounts were $x = 0.022$ and $x = 0.032$ respectively. A more precise estimate of $x$ was derived by detecting the orthorhombic-tetragonal transition through anelastic relaxation. The relationship of the transition temperature $T_0$ to the amount of Sr was taken as $T_0$...
(x) = 535 [1 - (1/0.235) x] (Refs. 14 and 21). From the step in the Young’s modulus (Fig.1) at the transition normalized in temperature and amplitude, one as $T_0 = 495$ K and $T_0 = 468$ K, corresponding to the Sr amounts $x = 0.019 \pm 0.0015$ and $x = 0.030 \pm 0.001$ (hereafter called samples 2 and 3 percent). As it is noted the structural transition appears even sharper than in pure La$_2$CuO$_4$ (where some broadening may be attributed to non-perfect oxygen stoichiometry). SQUID magnetization measurements yield a magnetic susceptibility as a function of temperature qualitatively typical of a spin-glass phase. Small differences between the field cooled and zero-field cooled data below about 140 K have been attributed to magnetic impurities present in the powders used for the preparation. These impurities do not affect the $^{139}$LaNQR and anelastic relaxation measurements.

In Fig.2 the quantity $J_{tatt} = T Q^{-1}/\omega$ is reported in the temperature range of interest, in correspondence to the measuring frequencies $\omega/2\pi = 1.29, 6.9$ and 17.2 kHz, in LSCO at $x = 2$ percent. The thermal depinning of the stripes should have cooperative character. For the moment we neglect their frequency distribution. According to the data in Fig. 2, the spectral density of the motion responsible of the dissipation has a diffusive character:

$$J_{tatt}(\omega_m) = \frac{2\omega_s}{\omega_s^2 + \omega_m^2}$$

From the temperature where the maxima are observed one can deduce the values of the characteristic frequency $\omega_s$. A good fit of the data (Fig.3) is obtained on the basis of a temperature behavior of $\omega_s$ of the form

$$\omega_s = \omega_0 \exp(-E/T)$$
Fig. 3. Characteristic frequencies of the motions as estimated from the maxima in Fig. 2. The solid line is the fitting behavior according to Eq. 2 in the text and correspondent to $\omega_0 = 4.5 \times 10^{12} \text{s}^{-1}$ and $E = 1650 \text{K}$. The empty circle indicate the frequencies deduced from the maxima in the $^{139}\text{LaNQR}$ relaxation rate around $T=160 \text{K}$ (see Fig. 5 and text).

with $\omega_0 = 4.5 \times 10^{12} \text{s}^{-1}$ and $E = 1650 \text{K}$, consistent with the idea of thermal activation of the stripe fluctuations.

As regards $^{139}\text{LaNQR}$ relaxation, the recovery laws for the $+5/2 - +7/2$ line at $3\nu_Q$ and the $+3/2 - +5/2$ at $2\nu_Q$, are multiexponential. However in the first decade they differ only little from a single exponential. The correspondent effective decay rate $\tau_e^{-1}$ can be related to the magnetic relaxation rate $W_M$ or to the quadrupolar relaxation rate $W_Q$ due to the time dependence of the electric field gradients at the La site, in the following way [22]:

$$3\nu_Q \quad \tau_e^{-1} = (67/21)W_Q = 23W_M$$

$$2\nu_Q \quad \tau_e^{-1} = (64.5/21)W_Q = 41.3W_M$$

For $T \geq 250 \text{K}$ the NQR relaxation rate (Fig. 4) are frequency independent and follow the law $\tau_e^{-1} \propto T^2$, features characteristic of the relaxation process driven by underdamped phonons [23]. Also an order of magnitude estimate corroborates this conclusion, since for this process one expects [24] $W_Q = 5 \times 10^{-4} T^2 \text{s}^{-1}$. Around $280 \text{K}$ some evidence of a quadrupole contribution due to overdamped phonon modes (tilting of the oxygen octahedra in a double well potential [25]) is present [26]. Below $250 \text{K}$ the relaxation rates depart from the behavior described above. For temperatures lower than about $180 \text{K}$ the comparison of the data for the $3\nu_Q$ and $2\nu_Q$ lines indicates the insurgence of a magnetic relaxation mechanism. The temperature behavior of $\tau_e^{-1}$ for $T \leq 230 \text{K}$ is analyzed in detail in Fig. 5, after subtraction of the background of quadrupole character. Assuming that the modulation of the hyperfine magnetic field $\mathbf{h}(t)$ is due to the same motion causing the mechanical relaxation, then for

$$2W_M = \frac{1}{2} \gamma^2 \int \langle h_+(0)h_-(t) \rangle e^{-i\omega t} dt$$

one writes

$$(\tau_e^{-1})_{3nQ/3nQ} = aW_M = a\frac{1}{2} \gamma^2 h \left[2\omega_s/(\omega_s^2 + \omega_m^2)\right]$$

with $a = 23$ for the $3\nu_Q$ line and $a = 41.3$ for the $2\nu_Q$ line, $\omega_m = 3\omega_Q$ and $\omega_m = 2\omega_Q$ respectively. In Fig. 5 the experimental data for $aW_M$ are compared with the theoretical behaviors for the relaxation rates according to Eq.s 3 and 4, having used for $\omega_s$ the expression derived from the anelastic relaxation (Eq. 4). The maxima in $W_M$ are well reproduced. The departures of the experimental data from the theoretical expressions in the temperature range
corresponding to slow motions, \( \omega_s \leq \omega_m \), are likely to be due to the simplifying assumption of a monodispersive process. A distribution in \( \omega_s \) implies a flattening in the relaxation rate around the maximum and a departure from the behavior for monodispersive process more marked in the low temperature range, as it is observed in the Figure. The relevant fact is that the temperature dependence of \( \omega_s \) deduced from anelastic relaxation in the kHz range seems to justify quantitatively the magnetic NQR relaxation rate in the MHz range. On the other hand, the maxima in \( W_M \) around 150 K could reflect the slowing down of the spin dynamics on approaching the transition to an ordered state. Information in this regard can be obtained from the NQR spectra (Fig.6), since the insurgence of a static field \( \langle h \rangle \) at the La site is signaled by a splitting \( \Delta \) of the resonance line, proportional to the sublattice magnetization \( \langle S \rangle \). A clear splitting of the line is noticeable only below about 50 K, close to the temperature indicated by neutron scattering \[15\]. If the width \( \delta \) of a single component is kept temperature independent the fitting of the spectra with two gaussian lines yields an ordering temperature \( T_N \), where \( \Delta \) goes to zero, of about 140 K. The temperature dependence of the order parameter \( \langle h \rangle \propto \langle S \rangle \) would turn out quite different from the one of a canonical phase transition to the AF phase, experimentally observed \[27\] in pure \( \text{La}_2\text{CuO}_4 \). On the other hand, if the maxima in \( W_M \) at 150 K are taken as an indication of stripe motion at frequency around \( \omega_Q \), then an NQR line broadening must be expected below the temperature at which the frequency becomes of the order of the line width it-
An experimental support to the hypothesis that the intrinsic linewidth $\delta$ is temperature dependent comes from the comparison of the spectra at $2\nu_Q$ and at $3\nu_Q$ (Fig. 6). The ratio $\delta_{2\nu_Q}(T = 77 \text{ K}) / \delta_{2\nu_Q}(T = 177 \text{ K}) = (210 \text{ kHz}) / (183 \text{ kHz}) = 1.15$ is the same of the one for the $3\nu_Q$ line: $\delta_{3\nu_Q}(T = 77 \text{ K}) / \delta_{3\nu_Q}(T = 177 \text{ K}) = (320 \text{ kHz}) / (280 \text{ kHz}) = 1.15$. One also has $\delta_{3\nu_Q} = (3/2) \delta_{2\nu_Q}$. These data are not compatible with an effect due to a magnetic field $\langle h \rangle$, that would cause an extra broadening of the same amount for both the $2\nu_Q$ and the $3\nu_Q$ lines. Thus, if a moderate (of the order of 15-20%) temperature dependence for the single component linewidth $\delta$ is allowed, then the NQR spectra indicate $T_N \simeq 50 \text{ K}$. This value is in substantial agreement with the phase diagram commonly accepted in literature \[14\]. One could speculate that at this temperature a small bump is observed in the relaxation rates (Fig. 4), consistent with the slowing-down of the spin dynamics, just above the temperature range where the drastic increase of $W_M$ due to the spin freezing occurs. On cooling, the relaxation rate exhibits a maximum at a temperature around $T = 9 \text{ K}$. Also the recovery plots, showing evidence of departure from an exponential recovery towards the $t^{1/2}$ law, support the conclusion that the spin-glass quasi-freezing temperature $T_f$ has been reached. We compare now the experimental findings in the sample at the boundary between the AF and the spin-glass phase with the one at $x = 3 \text{ percent}$, well within the latter phase. The anelastic relaxation shows a temperature behavior similar to the one for $x = 2 \text{ percent}$.

**Fig. 6.** Typical La NQR spectra (obtained by Fourier transforming the signal as a function of the irradiation frequency) in LSCO at $x = 0.02$ for the $2\nu_Q$ and the $3\nu_Q$ lines. (Fig.7). Both the peaks attributed to the stripes motion and to collective tilting of the octahedra (not shown) are attenuated by a factor of about two. The $^{139}$LaNQR relaxation rates indicate the typical freezing of the spin fluctuations in a cluster spin-glass (Fig. 8). The maximum in $W$ occurs at $T = 8 \text{ K}$, in good agreement with the phase diagram by Cho et al \[16,21\].

The relaxation rate measured at $2\nu_Q$ reaches a value about twice the one reported in previous measurements \[16,28,29\] at $3\nu_Q$, consistent with a magnetic relaxation mechanism. It is noted that for $x = 0.03$ magnetization measurements provide direct evidence of the occurrence of a canonical spin-glass state \[30\].

The temperature dependence of $W$ can be discussed in terms of the behavior expected for the effective correlation time. For magnetic moments coupled to a Fermi gas of carriers \[31\], from Eq. in the fast fluctuations regime
Fig. 7. Spectral densities of the motions responsible of the elastic energy loss in LASCO at \(x=0.03\), for three measuring frequencies. One would have \(W \propto 1/\omega_s = \tau_f = h/\pi J^2 kT\), where \(J\) is the exchange coupling to the band and \(\rho\) the density of states at the Fermi level. From Fig. 8 one sees that \(W\) diverges, on decreasing temperature, more rapidly than \(T^{-1}\), in a way close to the law \(\tau \propto \exp[\tau_0/kT]\) at least for the temperature range where the fast motions condition, namely \(\tau_c \ll 1\), holds (see inset in Fig. 8). Below \(T \approx 10\text{K}\) one notes the behaviour of the relaxation rate expected for a glassy system.

3 Conclusions

Anelastic and NQR relaxation and NQR spectra have been combined in the attempt to derive insights on spin and lattice excitations, possibly stripes motions, driving the ordering processes in LSCO. The experimental findings have been discussed within two interpretative frameworks. On one side it could be possible that the phase diagram around the Sr content \(x = 0.02\) separating the AF and the spin-glass phases is more complex that previously assumed, with a quasi-long range ordering temperature as high as \(150\text{K}\), corroborating recent neutron scattering measurements and qualitatively agreeing with the extrapolation at \(x = 0.02\) of a magnetization study carried out in samples at \(x = 0.03, 0.04\) and \(0.05\). The order parameter of such a transition would be characterized by an unconventional temperature dependence.

On the other hand, the NQR spectra can be interpreted as indicating a conventional transition to the AF phase around \(T = 50\text{K}\), in substantial agreement with the phase diagram commonly accepted. In this case the anelastic and La NQR relaxation rates around \(T = 80\text{K}\) and \(T = 160\text{K}\) respectively, are the first direct experimental evidence of low frequency motions of stripes simultaneously involving spin and lattice excitations. The thermal de-
pinning barriers and the characteristic diffusive frequencies are then derived, and they do not differ much in the sample at Sr content 0.03.

In the low temperature range a spin freezing process is detected, with a dramatic increase of the $^{139}$LaNQR relaxation rate on cooling.

These experimental findings could be, at least in part, explained with a kind of distribution of transition temperatures $T_N$, $T_f$ and $T_g$ resulting from a spread in the Sr content around the critical amount $x = 0.02$. However the tetragonal-orthorhombic transition appears sharp and hence the Sr content seems to be well defined and close to that value.

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