Similar glassy features in the NMR response of pure and disordered La$_{1.88}$Sr$_{0.12}$CuO$_4$

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High $T_c$ superconductivity in La$_{2-x}$Sr$_x$CuO$_4$ coexists with (striped and glassy) magnetic order. Here, we report NMR measurements of the $^{139}$La spin-lattice relaxation, which displays a stretched-exponential time dependence, in both pure and disordered $x = 0.12$ single crystals. An analysis in terms of a distribution of relaxation rates $^{139}T_1^{-1}$ indicates that i) the spin-freezing temperature is spatially inhomogeneous with an onset at $T^\text{onset}_g = 20$ K for the pristine samples, and ii) the width of the $T_1^{-1}$ distribution in the vicinity of $T^\text{onset}_g$ is insensitive to an $\sim 1\%$ level of atomic disorder in CuO$_2$ planes. This suggests that the stretched-exponential $^{139}$La relaxation, considered as a manifestation of the systems glassiness, may not arise from quenched disorder.

I. INTRODUCTION

The coexistence of magnetic order with superconductivity is a prominent feature of La$_{2-x}$Sr$_x$CuO$_4$ [1] and other underdoped high $T_c$ cuprates [2,3], even in zero magnetic field. However, neither the origin of this static magnetism nor the reason for its glassy character are fully understood. An important school of thought focuses on stripe physics [4]. Indeed, the temperature of magnetic freezing, $T_g$, in La$_{2-x}$Sr$_x$CuO$_4$ is peaked in the vicinity of $x \simeq \frac{1}{2}$ (Fig. 1), and neutron scattering studies [5,6,7] reveal long-range antiferromagnetic order with the same typical modulation as in the materials presenting direct evidence for charge stripe order [8]. Another approach relies on electronic and magnetic inhomogeneities generated by quenched disorder [9]. Undoped or weakly hole-doped droplets may form the magnetic clusters which freeze at low temperature, as suggested by the recent nuclear magnetic resonance (NMR) evidence for a nanoscale inhomogeneity of the hole concentration in La$_{2-x}$Sr$_x$CuO$_4$ [11]. Furthermore, the importance of quenched disorder could be supported by the glassy features observed in superconducting samples ($x \geq 0.06$) [11]. These are reminiscent of the spin-glass behaviour, well documented for $0.03 \leq x \leq 0.05$. Spatial heterogeneity is particularly evident in a number of magnetic measurements, such as the stretched-exponential NMR relaxation of $^{139}$La nuclei. This heterogeneity develops as the magnetic fluctuations slow down over a substantial temperature range on cooling above the freezing temperature $T_g$, the value of which depends on the timescale of the measuring probe. These properties are typical of glassy systems.

Glassiness is, however, also present in materials where stripe order is well-defined and relatively long range [8]. Furthermore, from the $T$ dependence of an average $^{139}$La spin-lattice relaxation rate $T_1^{-1}$, Curro et al. [12] concluded that the distribution of magnetic correlation times is similar in several materials with very different hole or impurity contents. This has led to the suggestion that glassiness in these materials is not due to quenched disor-
order but is self-generated by the charge stripes [13]. Later, Hunt et al. [14] determined the distribution of correlation times in stripe-ordered materials in a more direct way than Curro et al. [12], i.e., from the recovery of the $^{139}\text{La}$ NMR signal. The stretched-exponential relaxation was thus better characterized, but its origin was not the focus of the discussion.

In this paper, we report $^{139}\text{La}$ NMR measurements in $\text{La}_{1.88}\text{Sr}_{0.12}\text{CuO}_4$, where $T_\theta$ is maximum, as illustrated in Fig. 1. We quantify the $^{139}\text{La}$ stretched-exponential relaxation and discuss the issues of inhomogeneity and atomic disorder.

II. SAMPLES

Single crystals, of typical size $2 \times 3 \times 4 \text{ mm}^3$, were cut from rods grown by the traveling solvent floating zone method. Growth at a rate of $\sim 1 \text{ mm/h}$ yielded “pristine” samples with the standard $T_c$ of 30 $\text{K}$ [5], while growth at 0.2 mm/h produced a “disordered” sample with a low $T_c$ of 10 $\text{K}$ ($T_{\text{onset}} = 12 \text{ K}$) [4]. Neutron scattering (NS) studies have shown that the two crystals have the same structure as well as an identical temperature of the transition from the high $T$ tetragonal (HTT) phase to the low $T$ orthorhombic (LTO) phase. These studies have also revealed an identical incommensurability of the magnetic peaks. Since these quantities depend strongly on $x$, the results demonstrate that the two crystals have the same doping $x = 0.12$. On the other hand, magnetic Bragg peaks appear below $T_{c}^{\text{NS}} = 30 \text{ K}$ [5] and 25 K [15] for the pristine and disordered samples, respectively. All of these properties, as well as an upturn of the zero-field in-plane resistivity below 80 K, are consistent with the presence of $\sim 1\%$ of non-magnetic defects in the disordered sample [10]. These defects likely correspond to Cu vacancies, which produce the same magnetic effects as non-magnetic impurities [17, 18].

III. NMR METHODS

As in [19], the applied magnetic field was tilted away from the $c$-axis by $\theta \sim 10^\circ$, in order to obtain a sharp peak on the high frequency edge of the $^{139}\text{La}$ central, $\langle \frac{1}{2} \leftrightarrow -\frac{1}{2} \rangle$; Zeeman transition. $T_1$ was measured on this peak, as shown in the inset of the sample spectra in the inset to Fig. 2. The $T$ dependence of $T_1$ is identical on other peaks in the central transition spectrum. Experiments were performed in fields of 9 T (for $x = 0.12$, $T_c = 10 \text{ K}$ sample) and 14 T (for $x = 0.12$, $T_c = 30 \text{ K}$, and for $x = 0.10$ samples).

In Fig. 2, we plot the temperature dependence of $^{139}\text{La}$ linewidth for the high frequency peak (shaded region in the inset) of $\langle \frac{1}{2} \leftrightarrow -\frac{1}{2} \rangle$ transition for $\text{La}_{1.88}\text{Sr}_{0.12}\text{CuO}_4$ ($T_c = 30 \text{ K}$ sample). The linewidth broadens with decreasing temperature indicating increasing distribution of local static magnetization as the temperature is lowered.

We remark that even at the lowest $T$ the signal from other central transition lines is insignificant at the frequency of the high frequency peak and its high frequency side, for this orientation of the applied field. Therefore, the temperature evolution of the measured relaxation rates is intrinsic to magnetism and not a result of signal overlap from other central transition lines.

We also point out that no sign of phase separation is detected in our NMR data, in apparent disagreement with $\mu$SR data showing a magnetic volume fraction of $\sim 20\%$ for $x = 0.12$ [20]. Since NMR was performed here in a high magnetic field, part of the discrepancy might be resolved by an increase of the magnetic volume fraction with the applied field [21, 22].

IV. ANALYSIS OF $T_1$ DATA

In Figure 3, we display the time ($t$) dependence of the $^{139}\text{La}$ longitudinal magnetization $M(t) = M_z(t)/M_z(\infty)$ after a comb of $\frac{T}{2}$ saturation pulses, in the pristine sample. Clearly, the data cannot be fitted by the theoretical formula [23], which corresponds to $\alpha = 1$ ("exponential" relaxation) in

$$M_\alpha(t, T^{-1}) = 1 - 0.714 e^{-\left(\frac{28}{\alpha} T\right)^\alpha} - 0.206 e^{-\left(\frac{15}{\alpha} T\right)^\alpha} - 0.068 e^{-\left(\frac{6}{\alpha} T\right)^\alpha} - 0.012 e^{-\left(\frac{T}{\alpha}\right)^\alpha}. \quad (1)$$

We point out that the above statement is true for data at any temperature below $\approx 80 \text{ K}$, as evident in Fig. 3, when $\alpha < 1$. In $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ materials, the stretched
relaxation, i.e. the deviation from \( M_{\alpha=1}(t, T_1^{-1}) \), has been attributed to a distribution of \( 1^{39}T_1^{-1} \) values. We shall comment on this interpretation below.

In an ideal case, one-to-one correspondence between dynamic and static inhomogeneities can be revealed by measuring \( T_1^{-1} \) as a function of position across the NMR line shape (see [11, 24] for example). However, despite the observation of a continuous line-broadening (shown in Fig. 2), no significant frequency dependence of \( T_1^{-1} \) was found across the \( 1^{39}\text{La} \) line. Thus, to quantify the inhomogeneities one must resort to alternative analysis of \( M(t) \) data, in two possible ways:

(i) First, a fit to Eq. 1 is made with the stretching exponent \( \alpha \neq 1 \). This provides a phenomenological account of the distribution of \( T_1^{-1} \) values (Fig. 3).

(ii) Second, the formula \( M_{\alpha=1}(t, T_1^{-1}) \), is convoluted with a chosen probability distribution function of \( T_1^{-1} \). We found that a good fit to the data is provided by the Gaussian distribution on a logarithmic (\( \log T_1^{-1} \)) scale:

\[
M_G(t) = \left( \sqrt{\pi/2} \sigma_{\log} \right)^{-1} \times 
\int \exp\left(-\frac{(\log R_1 - \log T_1^{-1})^2}{\sigma_{\log}^2}\right) M_{\alpha=1}(t, R_1) \, d(\log R_1). \tag{2}
\]

This fit is defined by only two parameters: the most probable relaxation rate \( T_1^{-1} \), i.e., the center of the Gaussian, and the width of the distribution \( \sigma_{\log} \) on a \( \log_{10} \) scale. Using the \( \log T_1^{-1} \) space is more physical when very broad distributions of relaxation rates are expected. This also naturally avoids the introduction of an artificial low \( T_1^{-1} \) cut-off needed to eliminate unphysical negative values encountered when the linear scale is used. The fit yields \( T_1^{-1} \) values equivalent to the stretched fit \( M_{\alpha}(T_1^{-1}) \), as confirmed from the analysis of our data shown in Fig. 3. Furthermore, the value of \( \sigma_{\log} \) directly shows over how many orders of magnitude spreads the distribution of relaxation rates. Direct insight into this parameter is the main advantage of this fit (see Fig. 4). The \( \sigma_{\log} \) also appears to be linearly related to the value of \( \alpha \), as demonstrated in Fig. 3, in agreement with predictions of the recent theoretical work of Johnston for \( \alpha \geq 0.5 \) [25]. We also remark that we cannot experimentally determine the unique/exact shape of the distribution function. Equally good fits of the data can be achieved by assuming, e.g., an asymmetric Gaussian or a Lorentzian distribution for \( T_1^{-1} \). Thus, we choose to analyze the data assuming a distribution function of the simplest form, a Gaussian. However, the results to be discussed below have been found to be insensitive to the choice of the exact form of the distribution.

V. TEMPERATURE DEPENDENCE OF THE SPIN DYNAMICS

In this section, we shall first give a qualitative description of the temperature dependence of the mean relaxation rate \( T_1^{-1} \), in order to define the freezing tempera-
ture \( T_g \) on the timescale of NMR. Then, tentative fits of the \( T \) dependence of \( T_1^{-1} \) will be described. This parametrization of the data enables primarily a comparison between different La\(_{2-x}\)Sr\(_x\)CuO\(_4\) samples, while it cannot provide a physical picture of the spin-glass freezing in the system. Furthermore, the shortcomings of such analysis based on the mean value of the distribution of \( T_1^{-1} \) values are alluded to in the next section.

The temperature dependence of \( T_1 \) may be understood, at least qualitatively, from the following expression:

\[
\frac{1}{T_1} = \frac{\tau_c^2}{\gamma_n} \left( \frac{h_\perp^2}{1 + \omega_n^2 \tau_c^2} \right),
\]

where \( \tau_c \) is the correlation time, \( h_\perp = (h_{xx}^2 + h_{yy}^2)^{1/2} \) the component of the hyperfine field perpendicular to the field direction, and \( \omega_n \) the NMR frequency [26]. At high temperatures the correlation time is short, that is the condition \( \tau_c^{-1} \gg \omega_n \) is satisfied. As the dynamics of the system slows down on cooling, \( \tau_c^{-1} \) decreases causing an increase of \( T_1^{-1} \). This occurs down to the temperature \( T = T_{g,NMR} \) at which the condition \( \tau_c^{-1} = \omega_n \) is reached. Upon further cooling, \( \tau_c^{-1} \) continues to decrease but \( T_1^{-1} \) decreases. Thus, \( T_{g,NMR} \), the temperature of freezing on the timescale of \(^{139}\)La NMR (\( \omega_n \approx 10^8 \text{ Hz} \)), is defined as the temperature at which the relaxation rate is at its maximum value:

\[
(\frac{T_1}{T_{1,c}})_{\text{max}} = T_{g,NMR}^{-1} = \gamma_n^2 \left( h_\perp^2 \right) \omega_n^{-1}.
\]

For our pristine \( x = 0.12 \) sample, the peak of the mean \( T_1^{-1} \) occurs at \( T_{g,NMR} = 13 \) K. The temperature at which the increase of \( T_1^{-1} \) becomes noticeable may be defined as \( T_{g,slow} = 45 \) K. Interestingly, the ratio \( T_{g,slow}/T_{g,NMR} \approx 3.5 \pm 0.5 \) is much lower for \( x = 0.12 \) than for \( x = 0.10 \) (\( T_{g,slow}/T_{g,NMR} \approx 7.4 \)) and for other values of \( x \) [10, 31].

This statement holds even if an onset \( T_{g,NMR} \) is considered as described in the next section. Note also that the ratio is believed to be magnetic field independent for fields \((H_0 \approx 14 \text{ T})\) investigated here [19].

A more quantitative approach requires an analysis of the temperature dependence of \( T_1 \). Although it is not clear on which theoretical model such an analysis should be based, two models which have been used in the context of spin-freezing in the cuprates can be used.

First, we use Eq. [3] with an activated correlation time \( \tau_c = \tau_0 \exp(E_a/k_B T) \) to fit the temperature dependence of \( T_1^{-1} \) data with \( \tau_0 \) and \( E_a \) as fitting parameters, as depicted in Fig. [6]. This fit allows to extract the effective ‘energy barrier’ \( E_a \) for the activation of a thermally driven spin freezing process. Fitting the data for \( T_{g,NMR} \leq T < T_{g,slow} \), we find the effective energy barrier \( E_a = 140 \pm 30 \) K for our pristine \( x = 0.12 \) sample. This value is to be compared with \( E_a = 84 \pm 20 \) K for the disordered \( x = 12\% \) sample and \( E_a = 13 \pm 3 \) K for \( x = 10\% \).

Another possibility is to use the renormalized classical form [28] of the 2D Heisenberg model on a square lattice, which also captures well the increase of \( T_1^{-1} \) with decreasing temperature above \( T_{g,NMR} \) (see also [14]). This form allows to extract the effective spin stiffness \( (\rho_s) \) parameter. As illustrated in Fig. [6], in the temperature range \( T_{g,NMR} < T < T_{g,slow} \), the data for both \( x = 0.12 \) samples fit well to the low temperature limit \((T \lesssim 2\pi \rho_s/2)\) of the renormalized classical form, expressed as

\[
\frac{1}{T_1} \propto \frac{e^{C/T} \cdot (T/C)^{3/2}}{C \cdot (1 + T/C)^3},
\]

Here \( C \equiv 2\pi \rho_s \) is a fitting parameter. We find a value of \( \rho_s = 25 \pm 5 \) K for the spin stiffness of our pristine \( x = 0.12 \) sample. For the disordered \( x = 0.12 \) sample, \( \rho_s = 19 \pm 4 \) K is lower. An even weaker value \((\rho_s = 2 \pm 1 \) K\) is found for the \( x = 0.10 \) sample. This correlates with the fact that \( T_{g,slow}/T_{g,NMR} \gtrsim 7.4 \) for this sample. Due to its weak spin stiffness parameter, the data for the \( x = 0.10 \) sample are fitted to the high temperature limit \((T \gtrsim 2\pi \rho_s/2)\) of the renormalized classical form [28], given by

\[
\frac{1}{T_1} \propto \left( 1 + \frac{C}{4T} \right)^{-1/2} \exp \left[ \left( 1 + \frac{C}{4T} \right) \left( \frac{C}{2T} \right)^2 \right],
\]

with \( C \equiv 2\pi \rho_s \) as a fitting parameter. Not surprisingly (given the similarities of the fitting formulas in this tem-
plotted in Fig. 5a. The dashed lines are fits to the Eq. 3 with 

FIG. 6: (Color online) Fits to the relaxation rate 

For both 

It is in fact the maximum, and not the mean, 

The distribution of 

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Since the elastic NS signal is integrated over a typical energy window of \(\sim 1\) meV and the slowing down of the fluctuations occurs on a relatively wide \(T\) range, we observe \(T_{\text{g}}^{\text{NS}} > T_{\text{g, onset}}^{\text{NMR}}\). 

VI. INHOMOGENEITY OF \(T_\theta\)

For a more adequate description of the spin freezing, it is in fact the maximum, and not the mean, \(T_1^{-1}\) value present in the system which should be considered. We define this value to be the rate at which the probability distribution is 1\% of its maximum. Based on results shown in Figs. 4 and 5, we deduce that a typical maximum value is approximately \((T_1^{-1})_{\text{max}} \approx 10^3\) s\(^{-1}\). It is apparent in Fig. 6 that the weight of the Gaussian distribution at \(T_1^{-1} = 10^3\) s\(^{-1}\) becomes sizeable at \(T_{\text{NMR, onset}}^{\text{NS}} = 20\) K. This means that spins begin to be frozen well above 13 K in some regions of the sample. Our \(T_{\text{NMR, onset}}^{\text{g}} = 20\) K agrees perfectly with the appearance of a coherent precession of the muon spin in a \(x = 0.12\) single crystal similar to ours [20].

Here, it must be recognized that \((T_1^{-1})_{\text{max}} = 10^3\) s\(^{-1}\) probably also represents a cutoff value above which the \(^{139}\)La NMR signal is unobservable (wipeout phenomenon) [12, 14, 19]. However, the consistency of the above analysis [30] suggests that our data correctly describe the spin dynamics of the system at least down to 20 K.

For the disordered sample, the freezing occurs at a lower temperature: \(T_1^{-1}\) vs. \(T\) shows a peak at \(T = 10\) K and \(T_{\text{NMR, onset}} = 15\) K. This is consistent with the qualitative analysis discussed in the previous section and with the decrease of \(T_{\text{NS}}^{\text{NS}}\) from 30 K (pristine) [3, 6, 7] to 25 K (disordered) [15], as determined by neutron scattering. Since the elastic NS signal is integrated over a typical energy window of \(\sim 1\) meV and the slowing down of the fluctuations occurs on a relatively wide \(T\) range, we observe \(T_{\text{g}}^{\text{NS}} > T_{\text{g, onset}}^{\text{NMR}}\).

VII. INHOMOGENEITY OF THE SPIN DYNAMICS

The distribution of \(T_1\) values acquires a significant width only at \(T \approx 60\) K and below. The fact that \(\sigma_{\text{log}} \approx 0\) at \(T = 80\) K and 100 K is remarkable. This means that the distribution of \(T_1^{-1}\) values seen by \(^{63}\)Cu or \(^{17}\)O NMR at these temperatures, and attributed to a nanoscale inhomogeneity of the hole concentration [11], is completely absent in the \(^{139}\)La \(T_1\) data. This contrasting situation is explained by two facts: First, the hyperfine field at \(^{139}\)La sites results from the coupling to several electronic sites in two different CuO\(_2\) planes. Thus, the hyperfine field is spatially more homogeneous than the electronic density. Second, the relatively weak amplitude of this (transferred) hyperfine field produces much weaker differences in the \(T_1\) values for \(^{139}\)La than for \(^{63}\)Cu or \(^{17}\)O. Still, the \(^{139}\)T\(_1\) inhomogeneity, which shows up at lower temperature, appears to be correlated to inhomogeneities in the static local magnetization, as revealed by the similar temperature dependence of both the distribution width \(\sigma_{\text{log}}\) and the static \(^{139}\)La linewidth shown in Fig. 3 and Fig. 2 respectively. However, for the reasons given above, no direct correlation between the inhomogeneity of \(^{139}\)T\(_1\) and the resonance frequency across the \(^{139}\)La line could be established.

Since \(^{17}\)O and \(^{63, 65}\)Cu NMR signals undergo a significant wipeout below \(\sim 50\) K [19, 32], it is difficult to know whether the inhomogeneity probed in \(^{139}\)La \(T_1\) measurements below \(\sim 60\) K is distinct from the one probed at higher temperatures and attributed to spatial variation of the hole concentration. At any given temperature below \(\sim 20\) K, the difference in \(^{139}T_1^{-1}\) values between \(x = 0.12\) and \(x = 0.10\) samples exceeds an order of magnitude. However, even if the amplitude of the nanoscale doping inhomogeneity does not produce a significant effects on the \(^{139}\)La NMR at \(\sim 100\) K, the possibility that it produces the large \(^{139}T_1\) distribution at low \(T\) cannot be discarded. On the other hand, it is possible that magnetic heterogeneity (ubiquitous in glassy systems) develops prior to the glass transition in La\(_{2-x}\)Sr\(_x\)CuO\(_4\), in addition to a nanoscale electronic inhomogeneity. In support to this is the fact that spin freezing and substantial magnetic inhomogeneity are reported in YBa\(_2\)Cu\(_3\)O\(_{6+x}\) [2], while a significant nanoscale variation of the hole concentration is apparently absent in this system [33].

As evident in Fig. 5, \(T_1\) values are typically dis-
tributed over more than one order of magnitude at low $T$, that is $\sigma_{\log}$ reaches a value of $\sim 1.5$ at $T = T_{g,NMR}$. This width does not depend strongly on the number of holes since similar values of $\sigma_{\log}$ are observed for $x = 0.10$ and $x = 0.06$ (not shown) at $T \sim T_{g,NMR}$.

Strikingly, the same value of $\sigma_{\log} \simeq 1.5$ is found at $T = T_{g,NMR}$ in both pristine and disordered samples for $x = 0.12$ (Fig. 5). Thus, the distribution width appears to be insensitive to an $\sim 1\%$ of in-plane disorder as well. One could argue that this result can be explained by the fact that La$_{1.88}$Sr$_{0.12}$CuO$_4$ is already a significantly disordered material in its pristine version. However, this argument does not hold since a typical 1 % of non-magnetic impurities or vacancies is highly detrimental to $T_g$ and it clearly affects other magnetic properties as well.

Finally, we will comment on alternative explanations of the stretched-exponential behavior of the NMR relaxation. It was suggested that the inhomogeneous magnetic state is related to extended charge density waves with imaginary order parameter (id-CDW) [34]. Within this model, the observed inhomogeneity in the NMR and $\mu$SR quantities would originate from sliding motions of orbital currents coexisting with the $d$-wave superconducting state. It is known that any order parameter of $d$-wave symmetry should be highly sensitive to impurities. This is in contrast to our data, showing that the inhomogeneity of $^{139}T_1$ is insensitive to 1% of atomic disorder. Therefore, the id-CDW scenario is unlikely to account for our observations.

Furthermore, a power-law time dependence of the spin-spin correlation function, instead of a distribution, is also debated in canonical spin-glasses as an explanation for the stretched-exponential NMR or $\mu$SR relaxation [35]. Anyhow, we did not consider this possibility as the presence of magnetic heterogeneity in La$_{2−x}$Sr$_x$CuO$_4$ is established by various experimental facts, such as the partial wipeout of the NMR signal or the $T$-dependent broadening of the NMR linewidth.

VIII. CONCLUSION

We have presented an NMR investigation of La$_{1.88}$Sr$_{0.12}$CuO$_4$, a prototypical material for studying spin-glass and stripe-ordering instabilities in the superconducting regime. Our analysis can be viewed as a parametrization of the stretched-exponential spin-lattice relaxation of $^{139}$La nuclei. We observe that this phenomenon is not affected by a $\sim 1\%$ level of disorder. This result might thus support proposals [15] of heterogeneous dynamics, or more generally of glassiness, which is not due to quenched disorder. However, how much of the magnetic heterogeneity/glassiness of La$_{2−x}$Sr$_x$CuO$_4$ is attributable to nanoscale variation of the hole doping remains unclear. It would also be interesting to investigate whether our results for the special $x = 0.12$ material still hold in samples with lower Sr concentration, including the non superconducting cluster spin-glass region of the phase diagram. The glassy nature of magnetism in superconducting cuprates clearly calls for further theoretical and experimental consideration, especially given the recent observation of an electronic glass by scanning tunneling microscopy [36].

IX. ACKNOWLEDGMENTS

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