

^{63}Cu and ^{89}Y NMR study of an optimally doped $\text{YBa}_2\text{Cu}_3\text{O}_{6.94}$ single crystal

T. Auler*

*Instituto de Física de São Carlos, Universidade de São Paulo, C.P. 369, 13560-970 São Carlos-SP, Brazil
and Laboratoire de Spectrométrie Physique, Université Joseph Fourier Grenoble I, B.P. 87,
38402 St.-Martin d'Hères, France*

M. Horvatić

Grenoble High Magnetic Field Laboratory, Max-Planck-Institut für Festkörperforschung and Centre National de la Recherche Scientifique, B.P. 166, 38042 Grenoble Cedex 9, France

J. A. Gillet

Laboratoire de Spectrométrie Physique, Université Joseph Fourier Grenoble I, B.P. 87, 38402 St.-Martin d'Hères, France

C. Berthier

*Laboratoire de Spectrométrie Physique, Université Joseph Fourier Grenoble I, B.P. 87, 38402 St.-Martin d'Hères, France
and Grenoble High Magnetic Field Laboratory, Max-Planck-Institut für Festkörperforschung
and Centre National de la Recherche Scientifique, B.P. 166, 38042 Grenoble Cedex 9, France*

Y. Berthier and P. Ségransan

Laboratoire de Spectrométrie Physique, Université Joseph Fourier Grenoble I, B.P. 87, 38402 St.-Martin d'Hères, France

J. Y. Henry

*Département de Recherche Fondamentale, Centre d'Etudes Nucléaires de Grenoble, B.P. 85X, 38041 Grenoble Cedex, France
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We report ^{63}Cu spin-spin and spin-lattice relaxation rates ($^{63}T_2^{-1}$ and $^{63}T_1^{-1}$) and ^{89}Y magnetic hyperfine shift (^{89}K) in the normal state of an optimally doped single-crystalline sample $\text{YBa}_2\text{Cu}_3\text{O}_{6.94}$ with $T_c = 93$ K (which corresponds to the maximal T_c for the $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ family). The comparison between ($^{63}T_1T$) $^{-1}$ and ($^{63}T_{2g}$) $^{-1}$ evidences that the spin pseudogap does *not* open above T_c at optimal doping, although the static spin susceptibility starts to decrease well above T_c , at $T_0 = 150$ K. Comparing the NMR data of this optimally doped sample with those of two other "90 K" samples previously studied by our group, an underdoped $\text{YBa}_2\text{Cu}_3\text{O}_{6.92}$ and an overdoped $\text{YBa}_2\text{Cu}_3\text{O}_7$, strongly suggests that the crossover to the spin pseudogap regime occurs precisely at the optimal doping level. A phenomenological phase diagram based on the NMR results in the normal states is proposed. We discuss to which extent our data agree with the predictions of the magnetic scaling theory. [S0163-1829(97)00641-3]

I. INTRODUCTION

The nonstandard properties of the normal state ($T > T_c$) of the copper oxide high- T_c superconductors (HTSC's) have attracted a great interest in the hope of finding the clue to the high-temperature superconductivity. In particular, inelastic neutron scattering (INS) and nuclear magnetic resonance (NMR) studies¹ have revealed the presence of antiferromagnetic (AF) fluctuations and a spin pseudogap in the low-energy excitations.² While the AF fluctuations seems to be present in all HTSC's, the spin pseudogap is rather restricted to the underdoped compounds, being their most outstanding feature because it opens above T_c . These results have given support to theories in which strong correlations play an essential role in the cuprates, like some approaches based on the t - J model³ or the magnetic scaling (MS) model,⁴ which predict the occurrence of a spin pseudogap phase in the underdoped HTSC's.

A remarkable fact is that from the experiments two distinct temperature crossovers may be identified, one associated with the spin response at $\mathbf{q} = \mathbf{Q}_{AF}$ and the other with that

at $\mathbf{q} = 0$. The crossover associated with $\mathbf{q} = 0$ is related to the decay of the static spin susceptibility $\chi_s(\mathbf{q} = 0, \omega = 0)$ below a characteristic temperature T_0 , as measured in bulk experiments or in NMR magnetic hyperfine shift probes. Whether or not the gap recently observed by photoemission experiments in $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ (Bi2212) (Ref. 5) is related to crossover at T_0 is still an open question. The characteristic temperature T_0 varies strongly with doping as evidenced by NMR results in $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ (LSCO),⁶ $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$ (Y123),^{7,8} Bi2212,⁹ and $\text{HgBa}_2\text{CuO}_{4+\delta}$.¹⁰ As regards the opening of a spin pseudogap at $\mathbf{q} = \mathbf{Q}_{AF}$, it corresponds to a transfer of spectral weight of magnetic excitations from low to higher energy, when the temperature decreases below a characteristic temperature T^* . The occurrence of this gap above T_c , has been evidenced by INS measurements in underdoped Y123.^{11,12} In parallel, the NMR data also suggested a decrease of the low-energy excitations from the precursor decay of the nuclear spin-lattice relaxation rate divided by T of the planar copper ($^{63}T_1T$) $^{-1}$ well above T_c .¹³⁻¹⁵ Notwithstanding, this does not suffice to determine the opening of a spin pseudogap; it is rather through the comparison be-

tween $(^{63}T_1T)^{-1}$ and the Gaussian component of the nuclear spin-spin relaxation rate $(^{63}T_{2g})^{-1}$ that the opening (or not) of the spin pseudogap can be confirmed.^{16–18}

The static and dynamic responses of the electronic spin as a function of the doping level in Y123 as well as other HTSC compounds has been the subject of numerous NMR studies.^{6–9,18} In previous works^{8,18} we have pointed out the spin pseudogap and the decrease with the temperature of the static spin susceptibility as the main characteristics of the underdoped regime of the Y123, while in the overdoped regime the spin pseudogap coincides with T_c and χ_s slightly increases with decreasing T . In particular, the dependence of the opening of the spin pseudogap with doping is an important issue in order to elucidate its relationship with superconductivity. Pursuing this idea, other groups have recently reported NMR studies of the evolution of T_c and the AF correlations as a function of the doping level,¹⁹ but in the absence of $^{63}T_{2g}$ data, the conclusion is uncertain. The aim of the present communication is to report NMR results which clearly show that the spin pseudogap at \mathbf{Q}_{AF} does *not* open above T_c at the optimal doping composition of an Y123 single crystal. Moreover, a comparison with the data of other ‘‘90 K’’ samples studied by our group strongly suggests that the crossover to the spin pseudogap regime at $\mathbf{q}=\mathbf{Q}_{\text{AF}}$ occurs precisely at the optimal doping level.

II. EXPERIMENT

The measurements were carried out on a ‘‘porous’’ $\text{YBa}_2\text{Cu}_3\text{O}_{6.94}$ single-crystal. Specific sample preparation²⁰ ensured the highest T_c value (of 93 K) for the Y123 family and a very sharp superconducting transition [$\Delta T_c(10\text{--}90\%)=0.15$ K in ac susceptibility with $H_{\text{ac}}=0.1$ Oe_{rms}], enabling us to identify the sample as *optimally* doped. Details of its characterization by specific heat measurements are given elsewhere,²⁰ and we also confirmed the T_c determination by the *in situ* measurement of the detuning of the NMR probe in zero magnetic field. The $^{63}T_1$ and the $^{63}T_{2g}$ were measured on the central line of the quadrupole splitted spectrum of the $^{63}\text{Cu}(2)$ with the applied magnetic field $H_0=5.7$ T parallel to the c axis. The ^{89}Y magnetic hyperfine shift (MHS) (^{89}K) with respect to the YCl_3 reference was measured with $H_0=15\text{T}(\parallel c)$ using the Carr-Purcell-Meiboom-Gill sequence to increase the signal-to-noise ratio.

III. RESULTS AND DISCUSSION

In the presentation of the results, we directly compare the data for the optimally doped sample to those obtained on two other ‘‘90 K’’ single crystals previously studied by our group:^{8,18} a slightly underdoped $\text{YBa}_{1.93}\text{Sr}_{0.07}\text{Cu}_3\text{O}_{6.92}$ [$T_c=91$ K, $\Delta T_c(10\text{--}90\%)=2.5$ K] and an overdoped $\text{YBa}_{1.92}\text{Sr}_{0.08}\text{Cu}_3\text{O}_7$ [$T_c=90$ K, $\Delta T_c(10\text{--}90\%)=1$ K], where the T_c 's were determined from the detuning of the NMR probe in zero magnetic field. These samples are small pieces of the single crystals used for INS experiments at the Leon Brillouin Laboratory.¹² Regarding the presence of Sr impurity at the site of Ba, as is discussed in a previous work,²¹ its main effect is to slightly depress T_c ($\Delta T_c \leq 1\text{--}2$ K) with respect to impurity free Y123 of the same nominal oxygen content. Therefore, we emphasize that, de-

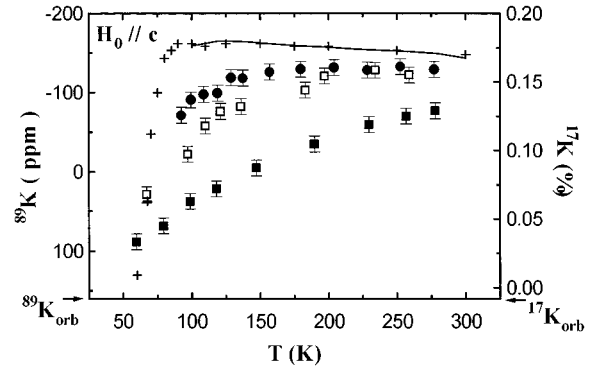


FIG. 1. T dependence of $^{89}K_c$ in $x=0.92$ (\square) and $x=0.94$ (\bullet) sample. Additional data are $^{89}K_c$ for $x=0.5$ (\blacksquare) and $x=1$ (solid line) samples (from Refs. 23 and 22, respectively), and $^{17}K_c$ ($+$) from Refs. 8 and 21. The origin of the vertical axes coincides with K_{orb} so that one is left with the spin contribution to the MHS.

spite the presence of Sr impurity, we are actually comparing three ‘‘90 K’’ samples with respect to their doping level, which is reflected on the value of T_c and, as is discussed below, through the ^{89}Y MHS.

The T dependence of $^{89}K_c$ is shown in Fig. 1 for the optimal, the $x=0.92$ and, for the sake of completeness, also for two fully oxygenated samples ($x=1$, $^{17}K_c$ from Refs. 8 and 21 and $^{89}K_c$ from Ref. 22) and for a less-oxygenated sample ($x=0.5$).²³ As far as the static spin susceptibility is concerned, it was demonstrated that the MHS of all nuclear sites [Cu(2), O(2,3), and Y] couple to a single spin degree of freedom.²⁴ The MHS is composed of a T -independent orbital part $^N K_{\alpha\alpha, \text{orb}}$ plus a T -dependent spin part $^N K_{\alpha\alpha, \text{spin}}(T) = N A_{\alpha\alpha, \text{spin}} \chi_{\alpha\alpha, \text{spin}}(T)$, where $N=63, 17$, and 89 stand for Cu(2), O(2,3), and Y sites, respectively. It is well known that the MHS is very sensitive to the doping level of the system CuO_2 planes, and this is confirmed by the data of Fig. 1: χ_s increases and becomes less T dependent with greater doping levels. This enabled us to clearly distinguish the doping level of our samples, from the underdoped $x=0.92$ sample up to the overdoped $x=1.0$ sample. In the underdoped regime, $\chi_s(T)$ decays monotonically with decreasing T , whereas in the overdoped regime it is slightly increasing. In the less-doped compounds, like $\text{YBa}_2\text{Cu}_4\text{O}_8$ (Ref. 25) and $\text{YBa}_2\text{Cu}_4\text{O}_{6.63}$ (Ref. 24), T_0 is above 300 K, but in the slightly underdoped sample ($x=0.92$) $T_0=200$ K and in the optimal sample ($x=0.94$) $T_0=150$ K. Note that at optimal doping $\chi_c(T)$ starts to decrease at $T_0=150$ K and the T dependence is intermediate between the underdoped and overdoped behavior; i.e., it is *flat* at higher temperature. In the overdoped regime, the temperature T_0 tends towards T_c , and for the more heavily doped compositions $\chi_s(T)$ is roughly temperature independent above T_c , like in ordinary metals. More precisely, $\chi_s(T)$ slightly increases with decreasing T ,^{8,21,22} a behavior which may be expected in a narrow band metal. These features of $\chi_s(T)$ are quite general in HTSC's and have been analogously observed on the NMR measurements in LSCO (Ref. 6) and $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ (Ref. 9). Curiously, in optimally doped $\text{Tl}_2\text{Ba}_2\text{CuO}_{6+y}$,²⁶ the temperature behavior of $\chi_s(T)$ is identical to our optimal doping sample, suggesting that this behavior might be characteristic of optimally doped compositions.

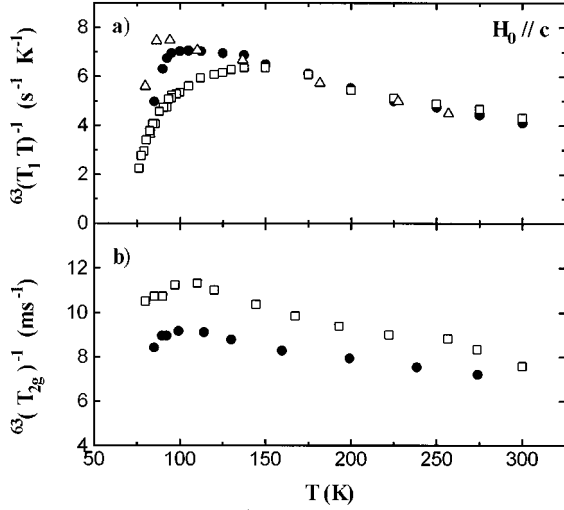


FIG. 2. T dependence of (a) $(^{63}T_1T)^{-1}$ and (b) $(^{63}T_{2g})^{-1}$: \square is for the $x=0.92$ sample, \bullet for the $x=0.94$ sample, and \triangle is for the $x=1$ sample.

Figure 2(a) shows $(^{63}T_1T)^{-1}$ as a function of T for three samples with $x=0.92, 0.94$, and 1.0 . The overall tendency of $(^{63}T_1T)^{-1}$ is well known, increasing as T decreases, but turning down differently according to the doping level of the sample.^{13,15} At higher temperatures (between 150 and 300 K), the data of all samples superpose almost perfectly. The discrepancy sets on at $T^*=140$ K where $(^{63}T_1T)^{-1}$ of the slightly underdoped sample ($x=0.92$) passes through a maximum. This behavior is typical of less-doped samples such as $\text{YBa}_2\text{Cu}_3\text{O}_{6.52}$,^{8,21} $\text{YBa}_2\text{Cu}_3\text{O}_{6.63}$,¹⁷ and $\text{YBa}_2\text{Cu}_4\text{O}_8$.^{25,27} For the optimal and overdoped samples, $(^{63}T_1T)^{-1}$ continuously increases down to T_c , saturating somewhat above T_c for the optimal composition. This flattening close to T_c has been analogously observed in optimally doped $\text{Tl}_2\text{Ba}_2\text{CuO}_{6+y}$,²⁸ but also in overdoped $\text{HgBa}_2\text{CaCu}_2\text{O}_{6+\delta}$,²⁹ and so it is not yet clear if this feature is characteristic of doping levels near the optimal composition or it may have some other origin. In the overdoped $\text{YBa}_{1.92}\text{Sr}_{0.08}\text{Cu}_3\text{O}_7$ sample, $(^{63}T_1T)^{-1}$ increases more linearly with decreasing T , turning down abruptly at T_c (i.e., the flattening before T_c is much less pronounced). The difference in the T dependence of $(^{63}T_1T)^{-1}$ as a function of doping contrast to the similarity of the behavior of $(^{63}T_{2g})^{-1}$ shown in Fig. 2(b). For both $x=0.92$ and $x=0.94$ samples, $(^{63}T_{2g})^{-1}$ grows as T decreases, passing through a large maximum around 100 K (unfortunately for the $x=1$ sample, these data are not available). The T dependence and the amplitude of $(^{63}T_{2g})^{-1}$ are larger for the less-doped sample. We point out again the remarkable similarity between the T dependence of $(^{63}T_1T)^{-1}$ and $(^{63}T_{2g})^{-1}$ of the optimally doped sample investigated here and that reported by Itoh *et al.*²⁸ for a nearly optimal $\text{Tl}_2\text{Ba}_2\text{CuO}_{6+y}$.

Now we compare $(^{63}T_1T)^{-1}$ to $(^{63}T_{2g})^{-1}$. The imaginary part of the dynamical electron spin susceptibility $\chi''(\mathbf{q}, \omega)$ is probed by $(^{63}T_1T)^{-1}$, while information on the real part $\chi'(\mathbf{q}, \omega=0)$ may be extracted from $(1/^{63}T_{2g})$.³⁰ If $\chi''(\mathbf{q}, \omega)$ and $\chi'(\mathbf{q}, \omega=0)$ are dominated by the contribution near $\mathbf{q}=\mathbf{Q}_{\text{AF}}$ and if one first neglects a possible temperature dependence of the correlation length ξ (in agreement with all

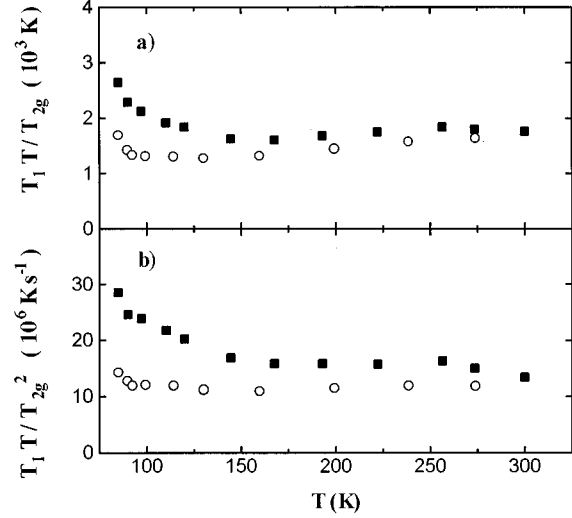


FIG. 3. T dependence of the ratios (a) $^{63}T_1T/^{63}T_{2g}$ and (b) $^{63}T_1T/(^{63}T_{2g})^2$: \blacksquare is for the $x=0.92$ sample and \circ for the $x=0.94$ sample.

available neutron data in Y123 compounds), it can be shown that $(^{63}T_1T)^{-1} \propto \chi''(\mathbf{Q}_{\text{AF}}\omega_n)/\omega_n \equiv J(\omega_n)$, where $\omega_n \approx 0$ is the nuclear Larmor frequency, while $(^{63}T_{2g})^{-1} \propto \chi'(\mathbf{Q}_{\text{AF}}) \propto \int_0^\infty J(\omega) d\omega$.¹ Clearly, a loss of spectral weight of the low-energy AF excitations will strongly affect $J(\omega_n)$ and $(^{63}T_1T)^{-1}$, but barely the total integral over $J(\omega)$ and $(^{63}T_{2g})^{-1}$. Thus the T dependence of the ratio T_1T/T_{2g} provides a powerful method to investigate whether a spin pseudogap at $\mathbf{q}=\mathbf{Q}_{\text{AF}}$ opens or not: If this ratio starts to increase at a certain temperature T^* , a spin pseudogap is opening. If one considers scaling hypothesis,⁴ then $(^{63}T_{2g})^{-1} \propto \xi^{-1} \chi'(\mathbf{Q}_{\text{AF}})$ and $(^{63}T_1T)^{-1} \propto \xi^{z-2} \chi'(\mathbf{Q}_{\text{AF}})/\Gamma_{\text{AF}}$, where z is the critical dynamic exponent and Γ_{AF} the bare characteristic energy of AF fluctuations. When $z=1$ (quantum critical regime), one expects $^{63}T_1T/^{63}T_{2g}$ to be constant within the temperature range in which the scaling hypothesis apply, then to increase as soon as the gap corresponding to the quantum disordered regime opens. When $z=2$ (nearly antiferromagnetic Fermi liquid), the temperature-independent quantity is expected to be $^{63}T_1T/(^{63}T_{2g})^2 \propto [\xi^{-2} \chi'(\mathbf{Q}_{\text{AF}})] \Gamma_{\text{AF}}$. The T dependences of the ratios $^{63}T_1T/^{63}T_{2g}$ and $^{63}T_1T/(^{63}T_{2g})^2$ are plotted in Figs. 3(a) and 3(b), respectively. In both the slightly underdoped and the optimally doped samples $^{63}T_1T/^{63}T_{2g}$ and $^{63}T_1T/(^{63}T_{2g})^2$ are weakly T dependent above a certain temperature T^* . For the underdoped sample $T^* \approx 140$ K coincides with the maximum of $(^{63}T_1T)^{-1}$, while for the optimally doped sample $T^* \approx T_c$. Although these data are not available for the overdoped sample, the $(^{63}T_1T)^{-1}$ increasing continuously from high T down to T_c , without any saturating tendency, suffices to ensure that there is no signature of the opening of the spin pseudogap above T_c . Therefore, these results strongly suggest that the crossover to the spin pseudogap regime at $\mathbf{q}=\mathbf{Q}_{\text{AF}}$ is occurring precisely at the optimal doping.

There is not yet a consensus about the microscopic origin of the pseudogap and neither about its possible relationship with the high T_c . In the MS theory,⁴ at high temperatures,

there is a crossover to a universal scaling regime at some temperature T_{cr} corresponding to a coherence length $\xi/a \approx 2$. T_{cr} is associated with T_0 and marks the onset of quantum critical (QC) scaling behavior, of the $z=1$ dynamic critical exponent, whose low-temperature end is given by T^* . At T^* the system crosses over to the quantum disordered regime with the opening of a spin gap in the spin excitation spectrum. According to the MS model,⁴ when the disorder introduced by the hole doping is too high, the scaling is no longer valid. So the overdoped samples are in a nonuniversal mean-field regime, which resembles a $z=2$ description, where $\chi'(\mathbf{Q}_{\text{AF}})\Gamma_{\text{AF}}$ is T independent and therefore a relation $(T_1 T)/(T_{2g})^2 = \text{const}$ should be obeyed in the limit of long correlation length for the AF spin fluctuations. As a matter of fact, $(T_1 T)/(T_{2g})^2 = \text{const}$ is quite well verified both for the optimally doped sample investigated here and $\text{Tl}_2\text{Ba}_2\text{CuO}_{6+y}$.²⁸ The MS model⁴ also predicts that in the QC regime, in the range of temperature between T_0 and T^* , $T_1 T (\propto \Gamma_{\text{AF}})$, $T_{2g}^{-1} (\propto \xi)$, and $\chi_s(T)$ should all be linear in T , and thus the ratio $(T_1 T)/(T_{2g})$ is constant. If these predictions seem to be fulfilled in $\text{YBa}_2\text{Cu}_4\text{O}_8$,³¹ in the ‘‘90 K’’ Y123 samples this corresponds to a rather narrow T range and the error bars remain too important to allow one to distinguish between the two regimes. The same way, even for the slightly underdoped sample, one cannot reliably decide whether $(T_1 T)/(T_{2g}) = \text{const}$ or $(T_1 T)/(T_{2g})^2 = \text{const}$ is better verified above T^* . We note also that a linear behavior of $(T_1 T)^{-1}$ and T_{2g}^{-1} extends up to the higher temperatures, far above T_0 . Within the MS theory⁴ the flattening of T_{2g}^{-1} around 110 K is attributed to the saturation of ξ . However, it must be stressed that the interpretation of $(T_1 T)^{-1}$ and T_{2g}^{-1} changes for short coherence length ξ ,^{12,32} and if the non-negligible $\mathbf{q} \approx 0$ contribution is taken into account.²³

In order to clarify the role of magnetic excitations in superconductivity, the relationship between the anomalies observed in the NMR measurements and those observed in transport measurements has been discussed. First Bucher *et al.*³³ and, more recently, Barzykin and Pines,^{4(a)} argued that T^* coincides with the temperature T_ρ below which the resistivity ceases to be linear in T . Indeed, for $\text{YBa}_2\text{Cu}_4\text{O}_8$ this seems right, but not for LSCO where the anomaly at T_ρ occurs at a temperature much higher than T^* . As Nakano *et al.*³⁴ have pointed out, in LSCO, T_ρ is closer to the maximum of the static spin susceptibility (T_0). Further strong evidence relating T_ρ to T_0 was recently brought on by resistivity measurements³⁵ revealing that the characteristic temperatures T_ρ are not affected by Zn doping in Y123. Once Zheng *et al.*³⁶ have shown that the Zn doping destroys the spin pseudogap while leaving almost unchanged $\chi_s(T)$, these results support that the anomalies in the resistivity are related to crossover temperature T_0 , and not to the spin pseudogap at $\mathbf{q} = \mathbf{Q}_{\text{AF}}$. Finally, Julien *et al.*³⁷ arrived at the same conclusion from almost perfect scaling of the in-plane resistivity (divided by T) and $^{63}K_{ab}$ in an underdoped $\text{HgBa}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\delta}$.

The key finding of this work is the coincidence of the disappearance of the spin pseudogap precisely at optimal doping, which suggests that some relation might exist between this phenomena and superconductivity. Actually, the importance of the AF correlations to the high T_c was evi-

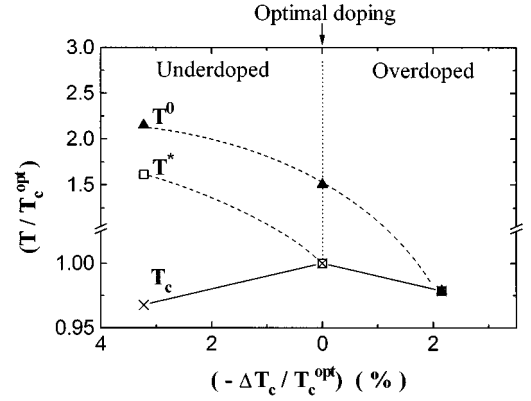


FIG. 4. Magnetic phase diagram for Y123 based on the NMR data presented here: the characteristic temperatures as a function of the doping level. T_0 and T^* are crossover temperatures defined in the text. Temperatures are expressed with respect to the maximal T_c of the optimally doped composition (T_c^{opt}), and the doping level is expressed as the relative decrease of T_c from T_c^{opt} .

denced by Zheng *et al.*,³⁶ who have shown that the substitution of Cu(2) by Zn (which depresses T_c) destroys the spin pseudogap in $\text{YBa}_2\text{Cu}_4\text{O}_8$, with a minor change in $\chi_s(T)$. This indicates, first, that the decrease of T_c produced by nonmagnetic Zn doping is related to the modification of the AF correlations and, second, that the spin dynamics at the center and at the border of the Brillouin zone are somehow decoupled.²³ In addition to this, the high pseudogap temperature and high characteristic energies of the spin fluctuations reported in mercury compounds^{37,38} may also suggest a relationship to the high T_c 's of these compounds. Of course, the experimental evidence is yet too sparse to enable any conclusion about the relationship between the spin pseudogap and superconductivity, and so further investigations are needed.

IV. SUMMARY

The main findings of this work are summarized in the magnetic phase diagram proposed in Fig. 4, where the crossover temperatures T^* and T_0 [determined from $(^{63}T_1 T)^{-1}$ and χ_s , respectively] as well as T_c are plotted as a function of the relative deviation from the optimal doping. For the first time we were able to compare the NMR results of three single crystals belonging to the ‘‘90 K plateau’’ of the Y123 compounds. We observed quite distinct behavior for $(^{63}T_1 T)^{-1}$, $(^{63}T_{2g})^{-1}$, and $^{89}K_c$ according to the doping level. When the doping level is smaller than the optimal, a spin pseudogap regime appears in the range of temperature between T^* and T_c . We could determine that the crossover to the spin pseudogap most probably occurs at the optimal doping, where T^* coincides with T_c within the experimental precision. The temperature T_0 is always superior to T^* and T_c . Upon increasing doping, T_0 decreases, with $T_0 > T^*$ at optimal doping and tending towards T_c in the overdoped regime. The properties in the narrow range $T_0 > T > T_c$ could not be clearly identified. We also speculate that a similar temperature dependence for χ_c , $(T_1 T)^{-1}$, and $(T_{2g})^{-1}$ is

found in every optimally doped cuprate. Further studies comparing these quantities as a function of the doping in the LSCO and other cuprates are desirable in order to shed more light on the problem of the relationship between the spin pseudogap and superconductivity.

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- *Present address: Universidade Federal de São Carlos, Via Washington Luís, km 235, C.P. 676, 13.565-905 São Carlos, SP, Brazil
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