Ni-substituted sites and the effect on Cu electron spin dynamics of $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$

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We report a Cu nuclear quadrupole resonance experiment on magnetic impurity Ni-substituted $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$. The distribution of Ni-substituted sites and its effect on the Cu electron spin dynamics are investigated. Two samples with the same Ni concentration $x=0.10$ and nearly the same oxygen content but different T_c's were prepared: One is an as-synthesized sample ($7-\delta=6.93$) in an oxygen gas ($T_c\approx80$ K), and the other is a quenched one ($7-\delta=6.92$) in a reduced oxygen atmosphere ($T_c\approx70$ K). The plane-site ^{63}Cu(2) nuclear spin-lattice relaxation for the quenched sample was faster than that for the as-synthesized sample, in contrast to the ^{63}Cu(1) relaxation that was faster for the as-synthesized sample. This indicates that the density of plane-site Ni(2) is higher in the quenched sample, contrary to the chain-site Ni(1) density, which is lower in the quenched sample. From the analysis in terms of the Ni-induced nuclear spin-lattice relaxation, we suggest that the primary origin of suppression of T_c is associated with the nonmagnetic depairing effect of the plane-site Ni(2).

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Magnetic impurity causes depairing in both *s*-wave and *d*-wave superconductivity. Complete suppression of the superconducting transition temperature T_c is observed for Ni-doped $La_{2-x}Sr_xCuO_4$ and $YBa_2Cu_4O_8$, somewhat more weakly than that for Zn-doped ones.[1–3] Ni impurity in the high-T_c cuprate superconductor carries a localized moment, because the uniform spin susceptibility with Curie or Curie-Weiss law is observed. The depairing effect of the potential scatterer Zn on the *d*-wave superconductivity is a natural consequence from breakdown of Anderson's theorem.[4,5] For $YBa_2Cu_3O_{7-\delta}$ with the optimized $T_c=92$ K, however, the decrease of T_c per Ni concentration is smaller than that per Zn concentration.[6–8] Figure 1(a) shows the impurity doping dependences of T_c for various high-T_c superconductors with Ni or Zn.[1,9–12] The decrease of T_c by Ni doping for $YBa_2Cu_3O_{7-\delta}$ is smallest among these compounds with Ni in Fig. 1(a). This small decrease of T_c by Ni doping for $YBa_2Cu_3O_{7-\delta}$ was attributed to a weak scattering of conducting carriers by Ni impurities (Born scatterer[13]) or to a softening of the pairing frequency itself.[9] Nevertheless, it has been suspected that only a part of the doped Ni impurities is substituted for the plane Cu(2) site and that the remaining part is for the chain Cu(1) site[14–17] (see the references in Ref. 18). The bulk T_c is considered to be determined by the amount of the in-plane Ni(2) impurity. One should note that $YBa_2Cu_3O_{7-\delta}$ has two crystallographic Cu sites, i.e., the chain Cu(1) and the plane Cu(2) sites. The recent observation of an in-plane anisotropy of optical conductivity for detwinned single crystal $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$ indicates the existence of the chain-site Ni(1).[19] A microscopic study on the selective substitution of Ni impurity is therefore of interest.

Synthesis of oxides under reduced oxygen partial pressure is frequently effective in controlling the cation solid solution, e.g., to synthesize the superconducting $La_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$,[20] or to optimize the superconducting critical current density or the irreversible magnetic field of $Nd_{1+x}Ba_{2-x}Cu_3O_{7-\delta}$.[21,22] Recently, using the reduced oxygen partial pressure technique, Adachi *et al.*, succeeded in controlling T_c of $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$ with optimal oxygen content.[18] They synthesized two series of $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$ samples having different T_c's per Ni concentration. Figure 1(b) shows the Ni doping dependence of T_c of their samples.[18] Here, we focus on two samples with the same Ni concentration $x=0.10$ [$z=0.033$ in Fig. 1(b)] and nearly the same oxygen content but different T_c. For

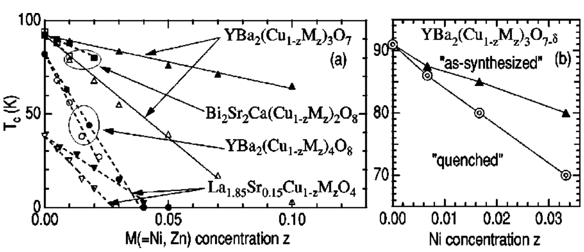

FIG. 1. (a) Impurity doping dependence of T_c as functions of the concentration z for various high-T_c superconductors with $M=$Ni or Zn. The solid (open) symbols are the Ni (Zn) doping dependence of T_c. The data are adopted from Ref. 9 for $YBa_2(Cu_{1-z}M_z)_3O_7$ (Y123, upward triangles), from Ref. 10 for $Bi_2Sr_2Ca(Cu_{1-z}M_z)_2O_8$ (Bi2212, squares), from Refs. 11 and 12 for $YBa_2(Cu_{1-z}M_z)_4O_8$ (Y124, circles), and from Ref. 1 for $La_{1.85}Sr_{0.15}Cu_{1-z}M_zO_4$ (LS214, downward triangles). The dashed and the solid lines are fitted by $T_c=T_{c0}$-m_Mz ($m_{M=\text{Ni, Zn}}$ is the fitting parameter) for the respective materials. The estimated ratio $m_{\text{Ni}}/m_{\text{Zn}}$ is about 0.26 for Y123, 0.46 for Bi2212, 0.80 for Y124, and 0.62 for LS214. The decrease of T_c by Ni doping for Y123 is smallest among these materials. (b) Ni doping dependence of T_c for "as-synthesized" or "quenched" Y123, adopted from Ref. 18. The solid curves are guide for the eyes. Note that the Ni concentration x in the text is defined by $x=3z$ in $YBa_2Cu_{3-x}M_xO_{7-\delta}$ [$7-\delta=6.92-6.95$ (Ref. 18)]. The decrease of T_c by Ni doping is larger in "quenched" Y123 than in the "as-synthesized" one.

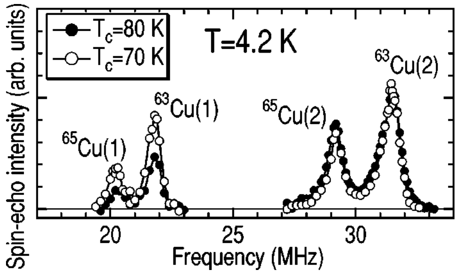

FIG. 2. Zero-field frequency spectra of the chain-site and the plane-site ^{63,65}Cu NQR for $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$ at $T=4.2$ K. The line shapes are scaled after T_2 corrections have been made.

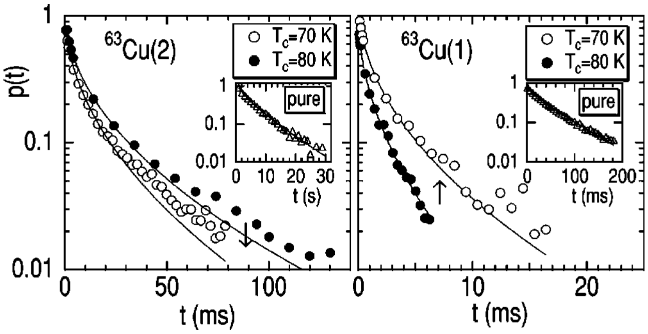

FIG. 3. The Ni-doping effect on the ^{63}Cu nuclear spin-echo recovery curves $p(t)\equiv 1-M(t)/M(\infty)$ at $T=4.2$ K. Insets show the recovery curves for the pure (Ni-free) sample at $T=4.2$ K. The solid curves are the least-squares fitting results using the theoretical function including a stretched exponential function (see the text).

convenience, let us call one sample with $T_c\approx 80$ K ($7-\delta=6.93$) an as-synthesized one, because it was synthesized in flowing oxygen gas without quenching treatment, and the other sample with $T_c\approx 70$ K ($7-\delta=6.92$) a quenched one, because it was the as-synthesized sample once again fired and quenched in a reduced oxygen atmosphere at 800 °C. The details are given in Ref. 18. Ni prefers the higher coordination of oxygen atoms.[23,7] The plane-site Cu(2) is located in the pyramid with five oxygen ions, whereas the chain-site Cu(1) is coordinated with two, three, or four nearest-neighbor oxygen ions. The two series of samples with different T_c suggest that the distribution of Ni-substituted sites over Cu(1) and Cu(2) sites is changed through synthesis under the reduced oxygen atmosphere.

In this paper, we report the measurements of Cu(1) and Cu(2) nuclear quadrupole resonance (NQR) spectra and nuclear spin-lattice relaxation curves to study microscopically the distribution of Ni-substituted sites for the above-mentioned two samples (as-synthesized and quenched ones). The observed difference in the ^{63}Cu nuclear spin-lattice relaxation at Cu(1) and Cu(2) in the two samples is supposed to be a Ni doping effect. From the Cu NQR measurements, we conclude that the heat treatment in reduced oxygen atmosphere indeed results in a redistribution of the Ni atoms in $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$, as supposed in Ref. 18.

Two samples with precisely the same mole number could not be prepared, because some parts of the samples have already been used for the characterization.[18] Hence, quantitative comparison of the relative intensity of Cu NQR spectra could not be made to estimate the relative number of the observed nuclei. The nuclear spin-lattice relaxation is independent of the sample volume and is relatively more sensitive to the impurity than the intensity of NQR spectrum. The powder samples were coated in paraffin oil. A coherent-type pulsed spectrometer was utilized for the zero-field Cu NQR measurements. The Cu NQR frequency spectra with quadrature detection were measured by integration of the spin echoes as a function of rf frequency. The Cu nuclear spin-lattice relaxation curves were measured by an inversion recovery spin-echo method, where the Cu nuclear spin-echo amplitude $M(t)$ was recorded as a function of time interval t after an inversion π pulse, in a π-t-$\pi/2$-π echo sequence, and $M(\infty)$ was also recorded in a $\pi/2$-π echo sequence (no inversion pulse) as usual.[11,12,24,25]

Figure 2 shows the Cu NQR spectra at $T=4.2$ K. The amount of the as-synthesized sample is more than that of the quenched one; nevertheless the intensity of Cu(1) NQR spectra for the as-synthesized sample was weaker than that for the quenched one. But we could not find a qualitative difference in the line profiles of Cu NQR spectra between two samples.

Figure 3 shows the ^{63}Cu nuclear spin-echo recovery curves (spin-lattice relaxation curves) $p(t)\equiv 1-M(t)/M(\infty)$ for Ni-doped samples at $T=4.2$ K. The insets of the figures show the recovery curves for pure (impurity-free) $YBa_2Cu_3O_{6.98}$ ($T_c=92$ K). Solid curves are the least-squares fitting results using the theoretical function described below. First, from comparison with the insets, all the recovery curves for both Ni-doped samples recover more quickly than those for pure $YBa_2Cu_3O_{6.98}$. Thus, Ni impurities distribute over both sites of Cu(1) and Cu(2). Second, the Cu(2) nuclear spin-echo signal recovers faster in the quenched sample than in the as-synthesized one, whereas the Cu(1) nuclear spin-echo signal recovers more slowly in the former than in the latter. The Cu(2) nuclear spin-echo signal is affected in the quenched sample more than in the as-synthesized one, whereas the Cu(1) is vice versa. Thus, it is natural to conclude that the amount of Ni(2) in the quenched sample is more than that in the as-synthesized one and that of Ni(1) is vice versa.

The ^{63}Cu nuclear spin-echo recovery curves for Ni-doped samples in Fig. 3 are nonexponential functions. For quantitative discussion, we analyzed the experimental recovery curve $p(t)$ by the exponential function times a stretched exponential function $p(t)=p(0)\exp[-wt/(T_1)_{\mathrm{host}}-\sqrt{wt/\tau_1}]$ [$p(0),(T_1)_{\mathrm{host}}$, and τ_1 are the fit parameters, and $w=3$ at Cu(2) and $w=1$ at Cu(1)], after the dilute magnetic alloy[26] or $YBa_2Cu_4O_8$ with Ni impurities.[24] The multiplicative numerical factor w is introduced to conform to the conventional expression of T_1,[27] and it is not essential in the discussion below. Here, $w=3$ is defined for the Cu(2) NQR T_1 under a uniaxial electric field gradient. The Cu(1) site is under an asymmetric electric field gradient,[28,29] so that all the x, y, and z components of the fluctuating local field contribute to the Cu(1) NQR T_1. Then, we use a simple $w=1$. $(T_1)_{\mathrm{host}}$ is the Cu nuclear spin-lattice relaxation time due to the host Cu electron spin fluctuation via a hyperfine coupling. τ_1 is the

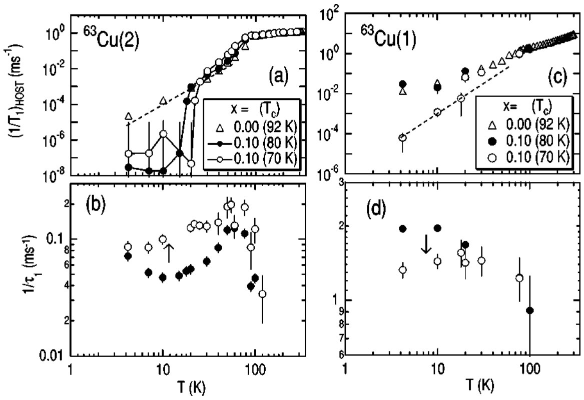

FIG. 4. Log-log plots of (a) [(c)] $^{63}(1/T_1)_{\rm host}$ and (b) [(d)] $^{63}(1/\tau_1)$ at ^{63}Cu(2) [^{63}Cu(1)] as functions of temperature for the Ni-free and for the Ni-doped samples. The dashed lines are T^3 functions. The solid curves are guide for the eyes.

impurity-induced nuclear spin-lattice relaxation time via a longitudinal direct dipole coupling or a two-dimensional Ruderman-Kittel-Kasuya-Yosida interaction.[30] We have confirmed no significant contribution from nuclear spin diffusion by measuring ^{65}Cu isotope dependence and pulse-strength H_1 dependence of the recovery curves.[31] Although for the heavily impurity-doped system[32] or spin glass system[33] where it is hard to assign separately the host and the guest contributions, a single stretched exponential function $p(t)=p(0)\exp[-(t/\tau_1)^{\alpha}]$ with a variable exponent α might be appropriate, and we believe that the present model with two time constants, $(T_1)_{\rm host}$ and τ_1, is minimal and appropriate for the $x=0.10$ samples.

Figure 4 shows the temperature dependence of the estimated (a) [(c)] $(1/T_1)_{\rm host}$ and of (b) [(d)] $1/\tau_1$ at ^{63}Cu(2) [^{63}Cu(1)]. Below T_c, the Ni-induced relaxation component (stretched exponential part) is predominant both at Cu(1) and Cu(2). In Figs. 4(b) and 4(d), the Ni-induced relaxation rate $1/\tau_1$ of Cu(2) for the quenched sample is more enhanced than that for the as-synthesized one, whereas that of Cu(1) is vice versa. In Fig. 4(b), the upturn of $1/\tau_1$ of Cu(2) below 10 K for the as-synthesized sample is consistent with the upturn of an initial relaxation rate $1/T_{1s}$ reported in Ref. 34. In Fig. 4(d), the difference in $1/\tau_1$ of Cu(1) between the two samples with Ni is larger at lower temperatures than about 30 K. It is hard to estimate precisely the small $1/\tau_1$ and its difference above about 30 K, where the signal-to-noise ratio of Cu(1) NQR is poor in the Ni-doped samples.

In Fig. 4(a), the host relaxation rate $(1/T_1)_{\rm host}$ of Cu(2) in the as-synthesized and the quenched samples decreases more steeply than that in pure $YBa_2Cu_3O_{6.98}$, as the temperature is decreased below $T=18$–20 K and below $T=25$–30 K, respectively. The dashed lines are T^3 functions characteristic of d-wave superconductivity. The steep decrease of $(1/T_1)_{\rm host}$ and the upturn of $1/\tau_1$ is also observed for $YBa_2Cu_{4-x}Ni_xO_8$ with $x=0.12$ ($T_c=15$ K).[24] It is theoretically suggested that the spin-orbit coupling between an itinerant electron and a Ni local moment induces a local superconducting state with a different order parameter (d_{xy}-wave symmetry) around Ni in the $d_{x^2-y^2}$-wave superconducting state.[35] The steep decrease of $(1/T_1)_{\rm host}$ far below T_c may indicate a Ni-induced impurity band associated with the different order parameter, more gapped on the Fermi surface than a pure $d_{x^2-y^2}$-wave gap.

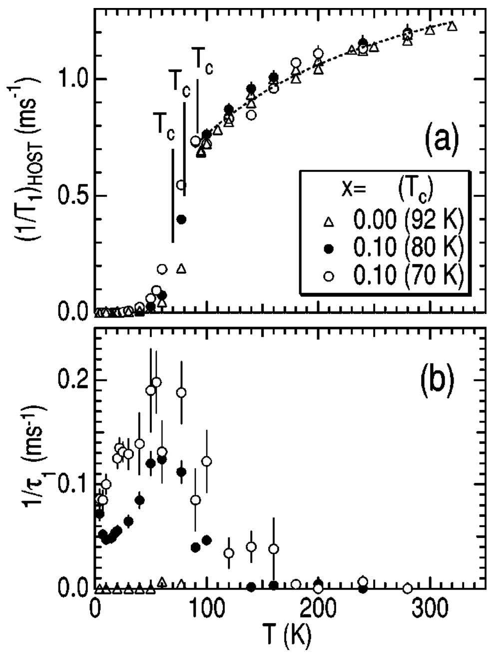

FIG. 5. The Ni-doping effect on the temperature dependence of (a) $^{63}(1/T_1)_{\rm host}$ and (b) $^{63}(1/\tau_1)$ at ^{63}Cu(2) in linear scale. The thick bars indicate the respective T_c's. The dotted curve is the least-squares fitting result by a function of $TC/(T+\Theta)$($C=1.7$ ms^{-1} and $\Theta=128$ K) above T_c.

Figure 5 shows the temperature dependence of (a) $(1/T_1)_{\rm host}$ and (b) $1/\tau_1$ at Cu(2) in linear scale to show up the above T_c data. The host $(1/T_1)_{\rm host}$ of Cu(2) above T_c is nearly independent of Ni doping, whereas $1/\tau_1$ increases with decreasing T_c. In general, $1/\tau_1$ is an increasing function of the impurity concentration.[26] Thus, the observed increase of $1/\tau_1$ indicates the systematic increase of the in-plane Ni(2) concentration $x_{\rm plane}$.

In the theoretical model of superconducting pairing mediated by antiferromagnetic spin fluctuations, the external depairing effect on T_c is written by $T_c=T_{c0}-\Delta T_c$ with $T_{c0}\sim\Gamma_0(Q)\chi_0(Q)$ [$\Gamma_0(Q)$ is the host antiferromagnetic spin-fluctuation frequency, and $\chi_0(Q)$ is the static staggered spin susceptibility[36,37]] and with $\Delta T_c\propto x_{\rm plane}$.[38] In spin-fluctuation theory,[39] one can obtain $(1/T_1)_{\rm host}\propto TC/(T+\Theta)$[$C\propto\chi_0(Q)/\Gamma_0(Q)$ and $\Theta\propto\Gamma_0(Q)$ (Ref. 40)] in the leading order. The actual fit result by this function is the dotted curve in Fig. 5(a). Thus, the quantitative temperature dependence of $(1/T_1)_{\rm host}$ tells us the characteristic spin-fluctuation parameters, $\chi_0(Q)$ and $\Gamma_0(Q)$, which may describe the pairing interactions. The nearly Ni-independent $(1/T_1)_{\rm host}$ in Fig. 5 indicates that the host spin-fluctuation spectrum is nearly invariant under Ni doping. In Ref. 9, the host $(1/T_1)_{\rm host}$ was estimated to be systematically enhanced by Ni doping, which was regarded as evidence for softening of the spin-fluctuation frequency $\Gamma_0(Q)$ and for the central origin for reducing T_c. However, our analysis indicates that the Ni-enhanced Cu(2) nuclear spin-lattice relaxation comes from the extra relaxation in the stretched exponential part. The central origin to reduce T_c is the external pair braking effect due to the increase of the in-plane Ni(2) concentration x_{plane} in ΔT_c. This is consistent with the original result for $YBa_2Cu_{4-x}Ni_xO_8$. However, it is still hard to estimate the quantitative value of x_{plane}.

Here, we analyze the temperature dependence of $1/\tau_1$ and discuss the pair breaking mechanism of Ni. For an isolated local moment system in a conventional metal, the dynamical spin susceptibility as a function of frequency $\omega/2\pi$ is expressed by $\chi(q,\omega)=\chi_L/(1-i\omega/\Gamma_L)$, where $\chi_L \propto S(S+1)/T$ is the static spin susceptibility and $\Gamma_L=\alpha T \equiv 4\pi\,(JN_F)^2 k_B T/\hbar$ (J is the coupling constant of the localized moments to the band, and N_F is the density of states at the Fermi level per spin) is the impurity fluctuation frequency due to the Korringa relaxation.[41] Then, one obtains $1/T_1$ or $1/\tau_1 \propto \Gamma_L/(\omega_N^2+\Gamma_L^2)$ ($\omega_N/2\pi$ is the nuclear resonance frequency).[42,27] Since $1/\tau_1 \propto 1/\alpha T$ at high temperatures ($T \gg \omega_N/\alpha$) and $1/\tau_1 \propto \alpha T/\omega_N^2$ at low temperatures ($T \ll \omega_N/\alpha$), then $1/\tau_1$ takes a maximum value at $T=\omega_N/\alpha$ ($\Gamma_L=\omega_N$).

In Figs. 4(b) and 4(d), $1/\tau_1$ of ^{63}Cu(2) and of ^{63}Cu(1) takes a maximum at about 60 K and 10–20 K, respectively, which can be associated with the maximum at $T=\omega_N/\alpha$. Assuming the Korringa relaxation $\Gamma_L=4\pi\,(JN_F)^2 k_B T/\hbar$ and setting $\Gamma_L=\omega_N=22$ MHz for Cu(1) at $T=10$ K and $\Gamma_L=31.5$ MHz for Cu(2) at $T=60$ K, one obtains $|JN_F| = 0.5\sqrt{\hbar\omega_N/\pi k_B T}=0.003$ for Cu(1) and $=0.0014$ for Cu(2). From a typical value of $N_F=1.5$ states/(eV spin direction), $|J|$ is 1.9 meV for Cu(1) and 0.94 meV for Cu(2). The actual maximum of $1/\tau_1$ takes place in the superconducting state. Thus, replacing the Korringa term $k_B T$ by a d-wave gapped term $k_B T(k_B T/\Delta_{max})^2$ [$2\Delta_{\text{max}}=8k_B T_c$ (Ref. 43)], one estimates $|JN_F|=0.009$ and then $|J|=5.8$ meV for Cu(2) (if taking into account the effect of the antiferromagnetic spin correlation, one gets a smaller $|J|$). The estimated magnitude of $|J|$ is smaller than the in-plane exchange interaction $|J_{\text{Ni-Ni}}| = 11$–31 meV of $La_2NiO_{4+\delta}$,[44,45] $|J_{\text{Cu-Cu}}|=150$ meV of $YBa_2Cu_3O_{6+\delta}$,[46] or a typical $4s$-$3d$ exchange interaction $|J_{sd}|\sim 0.1$ eV. Since $|\pi JN_F S|=0.03 \ll 1$, the decrease of T_c due to the magnetic scattering can be calculated within the lowest order Born approximation.[38] From $\Delta T_c = 0.25\pi^2 x_{\text{plane}} N_F J^2 S(S+1)/k_B$ with $S=1$ in a $d_{x^2-y^2}$-wave superconductor,[47] the sole occupation at Cu(2) ($x_{\text{plane}}=x/2=0.05$) in the quenched sample yields $\Delta T_c=0.08$ K at most. This value is smaller than the observed $\Delta T_c \sim 20$ K in the quenched sample by a factor ~ 250, suggesting that magnetic pair breaking is not the mechanism of suppression of T_c in $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$ (similar estimation for Zn doping is seen in Ref. 48). With this regard, we mention that the magnetic impurity Ni should also has a nonmagnetic scattering part.[47,49–51] Although this kind of spin-independent scattering part does not affect s-wave superconductivity, it can suppress d-wave superconductivity, so that we have to include this nonmagnetic part, as well as the magnetic one, in considering the depairing effect by Ni in the high-T_c cuprates.[51] Thus, one can expect that a moderately strong, nonmagnetic scattering of Ni is a promising origin of T_c suppression.

In conclusion, the Cu NQR experiment demonstrated that both Cu(1) and Cu(2) nuclear spin-lattice relaxations in $YBa_2Cu_{3-x}Ni_xO_{7-\delta}$ are affected by Ni doping, that is, the doped Ni impurities are substituted for both Cu(1) and Cu(2) sites. One of the reasons for the small decrease of T_c by Ni doping in $YBa_2Cu_3O_{7-\delta}$ is partial substitution of the Ni impurities for the chain site. In the light of the Ni-induced nuclear spin-lattice relaxation, the host Cu(2) spin fluctuation spectrum in $YBa_2Cu_3O_{7-\delta}$ with optimal oxygen content is quite robust for Ni doping.

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[51] In the $d_{x^2-y^2}$-wave superconductivity, when both the nonmagnetic and magnetic impurity scatterings by Ni impurities are taken into account within a *t*-matrix approximation, we find

$$\Delta T_c = \frac{x_{\mathrm{plane}}}{4k_B N_F}\left[1-\frac{1}{2}\left(\frac{1}{1+(\gamma_n-\gamma_m)^2}+\frac{1}{1+(\gamma_n+\gamma_m)^2}\right)\right].$$

Here, γ_n and γ_m, respectively, describe the depairing effects originating from nonmagnetic and magnetic parts of the magnetic impurities: $\gamma_n = \pi N_F u$ and $\gamma_m = \pi N_F JS$, where u is a nonmagnetic potential strength of Ni. We briefly mention that γ_n does not appear equation above in the case of the isotropic *s*-wave superconductivity because of Anderson's theorem.