

## Site-selective oxygen isotope effect on the magnetic-field penetration depth in underdoped $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$

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We report site-selective oxygen isotope ( $^{16}O/^{18}O$ ) effect (OIE) measurements on the in-plane penetration depth  $\lambda_{ab}$  in underdoped  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$ , using the muon-spin rotation technique. A pronounced OIE on the transition temperature  $T_c$  as well as on  $\lambda_{ab}^{-2}(0)$  was observed, which mainly arises from the oxygen sites within the superconducting  $CuO_2$  planes (100% within error bar). The values of the corresponding relative isotope shifts were found to be  $\Delta T_c/T_c = -3.7(4)\%$  and  $\Delta \lambda_{ab}^{-2}(0)/\lambda_{ab}^{-2}(0) = -6.2(1.0)\%$ . Our results imply that in this compound the phonon modes involving the movement of planar oxygen are dominantly coupled to the electrons.

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More than 15 years after the discovery of the cuprate high- $T_c$  superconductors (HTSC), their pairing mechanism is still not understood. For a long time, a role of phonons in this mechanism was dismissed, despite evidence of their relevance, for example, by isotope experiments (see, e.g., Refs. 1–6). One of the reasons for this was the prevailing belief that phonons simply could not account for such high transition temperatures  $T_c$ . The recent discovery of  $MgB_2$ ,<sup>7</sup> where phonons are clearly the pairing glue leading to an unexpectedly high  $T_c \approx 40$  K, clearly falsified this belief. Many other widely cited reasons as to why phonons ought to play no role in HTSC also do not stand to scrutiny.<sup>8,9</sup> Recent work by inelastic neutron scattering (INS)<sup>10,11</sup> and angle resolved photoemission spectroscopy (ARPES)<sup>12,13</sup> gave good indications of a strong electron-phonon coupling in various HTSC, invigorating the debate about the relevance of phonons.<sup>14,15</sup>

For conventional superconductors (including doped fullerenes<sup>16</sup> and  $MgB_2$ <sup>17</sup>), key experimental evidence for a phonon mediated pairing was provided by measurements of the isotope effect (IE) on  $T_c$ . In complex materials, such as the HTSC,  $T_c$  may depend on many parameters in a non-trivial way. Important complimentary information can be obtained from the IE on the magnetic penetration depth  $\lambda$ . Previous studies<sup>3–6</sup> showed substantial oxygen isotope ( $^{16}O/^{18}O$ ) effect (OIE) on the in-plane penetration depth  $\lambda_{ab}$ , which indicates an unusual (e.g., nonadiabatic) coupling of the electrons to phonon modes involving the movement of the isotope substituted atoms. It is important to identify the relevant phonon modes responsible for this effect. Keen insight in this respect can be obtained from studying the site-selective oxygen isotope effect (SOIE), as we will detail below. While two reliable reports on the SOIE on  $T_c$  exist,<sup>2,18</sup> the SOIE on  $\lambda_{ab}$  was previously deduced only in a very indirect way from magnetization measurements in the vicinity of  $T_c$ .<sup>18</sup>

In this communication, we report on the SOIE on  $T_c$  and on  $\lambda_{ab}$  in underdoped  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$ ,<sup>19</sup> measured di-

rectly and accurately by using the transverse field muon-spin rotation (TF- $\mu$ SR) technique.<sup>20</sup> The result clearly indicates a strong coupling of the electronic subsystem to phonon modes involving movements of the oxygen atoms in the  $CuO_2$  plane, while suggesting that modes involving apical and chain oxygen are less strongly coupled to the electrons. This agrees with the conclusions observed from the combination of INS (Refs. 10 and 11) and ARPES (Refs. 12 and 13) results, which were obtained in a completely different way and in other HTSC.

Polycrystalline samples of  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$  were prepared by standard solid-state reaction.<sup>21</sup> In order to prepare oxygen site-selective samples a two-step exchange process was applied.<sup>2,21</sup> In the first step [500 °C, 35 h at 1.2 bars in  $^{16}O_2$  ( $^{18}O_2$ ) gas] two pairs of completely oxygen substituted samples ( $^{16}O_{pac}$  and  $^{18}O_{pac}$ ) were prepared [here indexes  $p$ ,  $a$ , and  $c$  mean planar (within  $CuO_2$  planes), apical and chain oxygen, respectively]. In the second step (330 °C, 150 h, 1.2 bar) two site-selective oxygen samples ( $^{16}O_p^{18}O_{ac}$  and  $^{18}O_p^{16}O_{ac}$ ) were prepared via annealing one  $^{16}O_{pac}$  sample in a  $^{18}O_2$  atmosphere and one  $^{18}O_{pac}$  sample in  $^{16}O_2$  gas. The other two samples (one  $^{16}O_{pac}$  and one  $^{18}O_{pac}$ ) were simultaneously annealed in the same atmosphere as before. For the totally exchanged sample ( $^{18}O_{pac}$ ) the  $^{18}O$  content, estimated from the change of the sample weight after annealing, was found to be 90(2)%. The oxygen-tracer diffusion calculation<sup>21</sup> for the material used in this work (average measured grain size is 16  $\mu$ m) shows that after annealing at 330 °C, during 150 h more than 90% of the apex + chain sites and only about 10–15% of the plane sites should be exchanged. Keeping in mind that there are 4 planar and 3 apical and chain oxygen sites in the unit cell of  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_7$ , for the above-mentioned annealing procedure, one would expect about 45% of an average oxygen substitution. The experimental estimate (from the change of the sample weight) gives 41(2)% for both site-selective samples, in good agreement with the theoretical calculation.

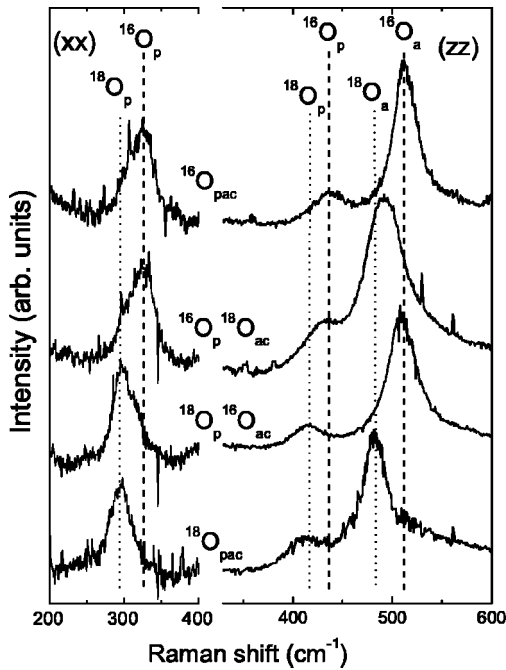


FIG. 1. Room-temperature Raman spectra of the  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$  samples. For  $xx$  polarization the line corresponds to the out-of-phase motion of the planar oxygen atoms ( $^{16}O$ ,  $325\text{ cm}^{-1}$ ;  $^{18}O$ ,  $294\text{ cm}^{-1}$ ). For  $zz$  polarization the two lines correspond to the in-phase motion of the  $CuO_2$  plane oxygen ( $^{16}O$ ,  $436\text{ cm}^{-1}$ ;  $^{18}O$ ,  $415\text{ cm}^{-1}$ ) and to the bond-stretching mode of the apical oxygen ( $^{16}O$ ,  $512\text{ cm}^{-1}$ ;  $^{18}O$ ,  $482\text{ cm}^{-1}$ ).

The site selectivity of the oxygen exchange was then checked by Raman spectroscopy. The 488-nm and 514.5-nm lines of an Ar laser were used for excitation. Figure 1 shows Raman spectra of all samples with  $zz$  and  $xx$  polarizations. In the  $zz$  polarization for the  $^{16}O_{pac}$  sample we observe two lines, one corresponding to the in-phase motion along the  $c$  axis of the planar oxygen atoms ( $436\text{ cm}^{-1}$ ) and one corresponding to the bond-stretching mode of apical oxygen, which in this compound is located at  $512\text{ cm}^{-1}$ .<sup>22</sup> In the  $xx$  polarization a line at  $325\text{ cm}^{-1}$  is seen, corresponding to the out-of-phase vibrations of the planar oxygen atoms.<sup>22</sup> In the  $^{18}O_{pac}$  sample, the Raman lines are all shifted to lower frequencies with amounts agreeing well with the ones obtained in Ref. 2, indicating a nearly complete exchange of  $^{16}O$  with  $^{18}O$ . In the site-selective sample  $^{16}O_p^{18}O_{ac}$ , only the position of the apical oxygen line is shifted to the lower frequency, whereas the lines corresponding to the plane oxygen stay the same [apart from a small shift of one Raman line ( $433\text{ cm}^{-1}$  instead of  $436\text{ cm}^{-1}$ ), probably due to a small unintentional partial substitution by  $^{18}O$ ]. Note that the apical line ( $492\text{ cm}^{-1}$ ) is also slightly shifted from the expected  $482\text{ cm}^{-1}$ , indicating that oxygen exchange for the apical and chain oxygen is slightly incomplete. In the  $^{18}O_p^{16}O_{ac}$  sample only two planar lines are shifted, while the apical line stays the same. The chain oxygen atom in yttrium barium copper oxide (YBCO) with oxygen content close to seven is not Raman active. However, based on the fact that diffusion in YBCO occurs via the chain oxygen (see, e.g., Ref. 21) one can conclude that the chain oxygen is exchanged completely.

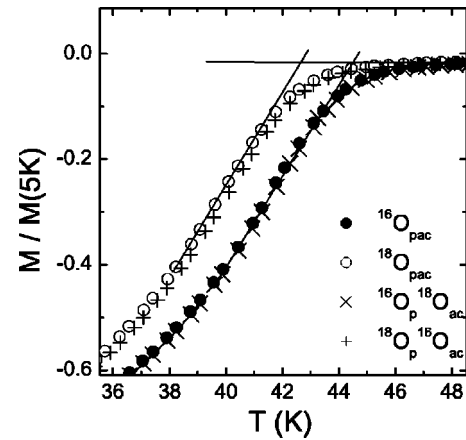


FIG. 2. Section near  $T_c$  of the normalized (to the value at 5 K) magnetization curves (1 mT, FC) of the site-selective  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$  samples.

Therefore, the Raman results confirm the almost complete site-selective oxygen substitution in all samples.

To determine the site-selective oxygen isotope shift on  $T_c$ , low field [1 mT, field cooled (FC)] superconducting quantum interference device (SQUID) magnetization  $M$  measurements were performed. Figure 2 shows the  $M(T)$  dependencies in the vicinity of  $T_c$  for all four samples. It can be seen that the  $^{16}O_{pac}$  sample has a higher  $T_c$  than the  $^{18}O_{pac}$  one with nearly the same transition width. The isotope shift  $\Delta T_c = -1.66(17)\text{ K}$  for completely substituted samples ( $^{16}O_{pac}$  and  $^{18}O_{pac}$ ) is in agreement with previous results<sup>5,23</sup> ( $T_c$  was extracted from the intercept of the linear extrapolation as shown in Fig. 2). The data points of the site-selective  $^{16}O_p^{18}O_{ac}$  sample coincide within error with those of the  $^{16}O_{pac}$  sample, whereas the  $^{18}O_p^{16}O_{ac}$  data points coincide with the  $^{18}O_{pac}$  ones, indicating that the OIE on  $T_c$  is related to the oxygen within the  $CuO_2$  planes. The results of the SOIE on  $T_c$  are summarized in Table I. The OIE exponent on  $T_c$  was determined as  $\alpha_O = -d\ln T_c / d\ln M_O$  ( $M_O$  is the mass of the oxygen isotope).

In order to determine the SOIE on  $\lambda_{ab}$  we performed TF- $\mu$ SR measurements at the Paul Scherrer Institute (PSI), Switzerland, using the  $\pi$ M3 beam line. The samples were cooled from far above  $T_c$  in a magnetic field of 200 mT. In a powder sample the magnetic penetration depth  $\lambda$  can be extracted from the muon-spin depolarization rate  $\sigma(T) \propto 1/\lambda^2(T)$ , which probes the second moment  $\langle \Delta B^2 \rangle^{1/2}$  of the probability distribution of the local magnetic-field function  $p(B)$  in the mixed state.<sup>20</sup> For highly anisotropic layered superconductors (like the cuprate superconductors)  $\lambda$  is mainly determined by the in-plane penetration depth  $\lambda_{ab}$ :  $\sigma(T) \propto 1/\lambda_{ab}^2(T)$ .<sup>20</sup> The depolarization rate  $\sigma$  was extracted from the  $\mu$ SR time spectra using a Gaussian relaxation function  $R(t) = \exp[-\sigma^2 t^2/2]$ . As discussed in Ref. 5, the measured total depolarization rate  $\sigma$  is determined by three terms: a superconducting ( $\sigma_{sc}$ ), an antiferromagnetic ( $\sigma_{afm}$ ), and a small nuclear magnetic dipole ( $\sigma_{nm}$ ) contribution. The antiferromagnetic contribution  $\sigma_{afm}$  is present *only* at low temperatures. Therefore data points below 10 K were excluded in the analysis. The dipole contribution  $\sigma_{nm}$  is practically tempera-

TABLE I. Summary of the SOIE on  $T_c$  and  $\lambda_{ab}^{-2}(0)$  for  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$  extracted from the experimental data (see text for an explanation). The values of  $\Delta T_c/T_c$ ,  $\alpha_O$ ,  $\Delta\lambda_{ab}^{-2}(0)/\lambda_{ab}^{-2}(0)$ , and  $\beta_O$  are not corrected for the incomplete oxygen-isotope exchange. The values of  $\alpha_O^c$  and  $\beta_O^c$  are corrected for the 100% oxygen exchange within  $CuO_2$  planes (see text).

Sample	$T_c$	$\Delta T_c$	$\Delta T_c/T_c$	$\alpha_O$	$\alpha_O^c$	$\sigma_{sc}(0)$	$\Delta\sigma_{sc}(0)$	$\Delta\lambda_{ab}^{-2}(0)/\lambda_{ab}^{-2}(0)$	$\beta_O$	$\beta_O^c$
	(K)	(K)	(%)			( $\mu s^{-1}$ )	( $\mu s^{-1}$ )	(%)		
$^{16}O_{pac}$	44.62(12)					1.090(7)				
$^{16}O_p^{18}O_{ac}$	44.66(10)	0.04(16)	0.09(36)	-0.007(29)	-0.048(30)	1.070(9)	-0.020(11)	-1.8(1.0)	0.14(8)	0.07(8)
$^{18}O_{pac}$	42.96(12)	-1.66(17)	-3.72(40)	0.298(32)	0.331(36)	1.022(9)	-0.068(11)	-6.2(1.0)	0.50(8)	0.56(9)
$^{18}O_p^{16}O_{ac}$	43.17(11)	-1.45(16)	-3.25(36)	0.260(29)	0.297(33)	1.018(7)	-0.072(10)	-6.6(0.9)	0.53(7)	0.61(8)

ture independent below 100 K, and since  $^{16}O$  and  $^{18}O$  oxygen have no nuclear magnetic moments it is also not sensitive to the oxygen substitution. Thus,  $\sigma_{sc}$  was determined by subtracting  $\sigma_{nm}$  measured above  $T_c$  from  $\sigma$  according to  $\sigma_{sc} = \sqrt{\sigma^2 - \sigma_{nm}^2}$ . Figure 3 shows the temperature dependence of  $\sigma_{sc}$  for the  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$  site-selective samples. It is evident that a remarkable oxygen isotope shift of  $T_c$  as well as of  $\sigma_{sc}$  is present. More importantly, the data points of the site-selective  $^{16}O_p^{18}O_{ac}$  ( $^{18}O_p^{16}O_{ac}$ ) samples coincide with those of the  $^{16}O_{pac}$  ( $^{18}O_{pac}$ ) samples. In order to put into evidence these results, the curve fitting the temperature dependence of  $\sigma_{sc}(T)$  for  $^{16}O_{pac}$  (shown below) was subtracted from the experimental data [Fig. 3(b)]. It can be seen that the experimental points for the two pairs of samples mentioned above coincide within the error bars (nearly within the error bars in the case of  $^{16}O_{pac}$  and  $^{16}O_p^{18}O_{ac}$ ). The data in Fig. 3 were fitted to the power law

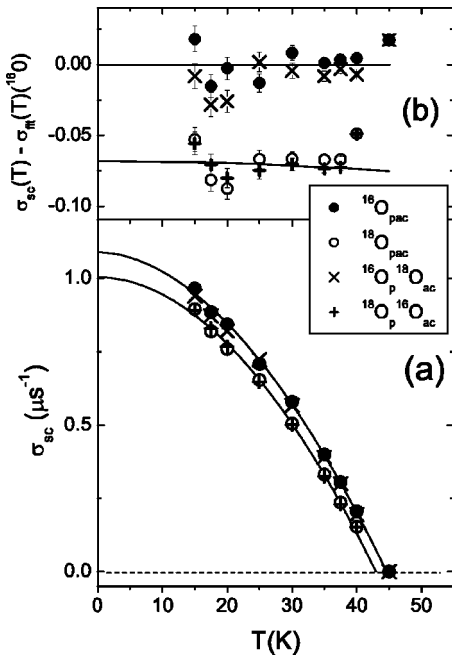


FIG. 3. (a) Temperature dependence of the depolarization rate  $\sigma_{sc}$  in site-selective  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$  samples (200 mT, FC). Data points below 10 K are not shown (see text for an explanation). The solid lines correspond to fits to the power law  $\sigma_{sc}(T)/\sigma_{sc}(0) = 1 - (T/T_c)^n$  for the  $^{16}O_{pac}$  and  $^{18}O_{pac}$  samples. (b) Same as in (a), but after subtracting the fitted curve for the  $^{16}O_{pac}$  sample.

$\sigma_{sc}(T)/\sigma_{sc}(0) = 1 - (T/T_c)^n$  with  $\sigma_{sc}(0)$  as a free parameter.<sup>20</sup> The value of  $T_c$  was taken from the magnetization measurements (see Table I),  $n$  was fitted as a common parameter for all four data sets and was found to be  $n = 1.87(4)$ , which is typical for underdoped YBCO.<sup>20</sup> Two points should be mentioned. (i) The exponent  $n$  is isotope independent<sup>4,5</sup> therefore fits with the same  $n$  for all data sets allow us to increase the accuracy in the determination of  $\sigma_{sc}(0)$ . (ii) Fits of experimental data with the power law satisfy the  $\chi^2$  criterion, indicating that this function describes our data well. The values of  $\sigma_{sc}(0)$  obtained from the fits are listed in Table I and are in agreement with previous results.<sup>24,5</sup> From the values of  $\sigma_{sc}(0)$  the relative isotope shift of the in-plane penetration depth  $\Delta\lambda_{ab}^{-2}(0)/\lambda_{ab}^{-2}(0) = [\sigma_{sc}(X)(0) - \sigma_{sc}(^{16}O_{pac})(0)]/\sigma_{sc}(^{16}O_{pac})(0)$  and the OIE exponent  $\beta_O = -d \ln \lambda_{ab}^{-2}(0)/d \ln M_O$  were determined (see Table I). Here  $X$  denotes  $^{16}O_p^{18}O_{ac}$ ,  $^{18}O_p^{16}O_{ac}$ , and  $^{18}O_{pac}$ . Our results (summarized in Table I) are in good agreement with previous estimates of SOIE from magnetization experiments<sup>18</sup> and our previous  $\mu$ SR non-site-selective OIE results.<sup>5</sup>

In Fig. 4, we plot the OIE exponent  $\beta_O$  [ $-\Delta\lambda_{ab}^{-2}(0)/\lambda_{ab}^{-2}(0)$ ] vs the OIE exponent  $\alpha_O$

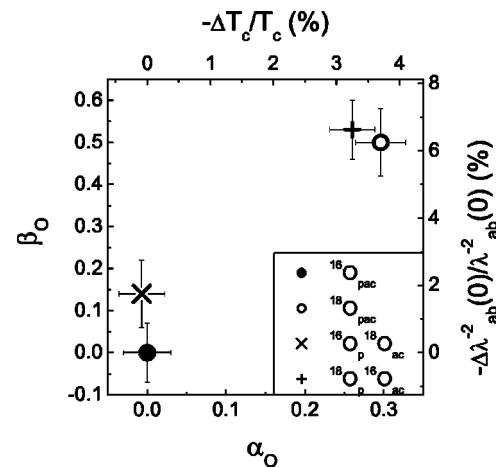


FIG. 4. Plot of the OIE exponent  $\beta_O$  ( $-\Delta\lambda_{ab}^{-2}(0)/\lambda_{ab}^{-2}(0)$ ) vs the OIE exponent  $\alpha_O$  ( $-\Delta T_c/T_c$ ) for site-selective  $Y_{0.6}Pr_{0.4}Ba_2Cu_3O_{7-\delta}$  samples. The error bars of the “trivial”  $^{16}O_{pac}$  point (zero isotope shift by definition) indicate the intrinsic uncertainty of the experimental procedures on the estimation of  $T_c$  and  $\lambda_{ab}$ .

( $-\Delta T_c/T_c$ ), in analogy with Ref. 5. Substantial OIE on  $T_c$  and  $\lambda_{ab}^{-2}(0)$  are observed for the completely  $^{18}\text{O}$  substituted ( $^{18}\text{O}_{\text{pac}}$ ) and the site selective  $^{18}\text{O}_{\text{p}}^{16}\text{O}_{\text{ac}}$  samples, which are the same within error bars. The other site-selective sample ( $^{16}\text{O}_{\text{p}}^{18}\text{O}_{\text{ac}}$ ) shows zero (within error bar) OIE on  $T_c$  and a very small, but possibly nonzero, OIE on  $\lambda_{ab}^{-2}(0)$ . This effect very likely arises from the small fraction of  $^{18}\text{O}$  that substituted  $^{16}\text{O}$  oxygen within the  $\text{CuO}_2$  planes, as seen in the Raman data (see Fig. 1). However, within the error bars, the OIE on  $\lambda_{ab}^{-2}(0)$  can still be considered the same for the  $^{16}\text{O}_{\text{pac}}$  and  $^{16}\text{O}_{\text{p}}^{18}\text{O}_{\text{ac}}$  samples. Based on the fact that nearly the whole OIE on  $T_c$  and  $\lambda_{ab}(0)$  arises from the oxygen within  $\text{CuO}_2$  planes, and using the plane oxygen content ( $\approx 87.5\%$ ) obtained from the tracer-diffusion calculation<sup>21</sup> we may perform correction of the OIE exponents  $\alpha_{\text{O}}$  and  $\beta_{\text{O}}$  for the 100% site-selective oxygen substitution. The corrected values of  $\alpha_{\text{O}}^c$  and  $\beta_{\text{O}}^c$  are listed in Table I (the OIE exponents for  $^{18}\text{O}_{\text{pac}}$  sample are corrected for 90%  $^{18}\text{O}$  content estimated from the mass change).

In summary, we performed  $\mu\text{SR}$  measurements of the site-selective oxygen isotope effect on the in-plane magnetic-field penetration depth in underdoped polycrystalline

$\text{Y}_{0.6}\text{Pr}_{0.4}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  samples. It was found that the whole or nearly the whole effect on the transition temperature  $T_c$  and on the in-plane magnetic penetration depth  $\lambda_{ab}^{-2}$  (about 100% within error bars) comes from the oxygen *within the superconducting  $\text{CuO}_2$  planes* and not from the apical and chain oxygen. Noting that the lattice parameters remain essentially unaffected by the isotope substitution,<sup>25,26</sup> our results show the existence of a strong coupling of the electronic subsystem to phonon modes involving movements of the oxygen atoms in the  $\text{CuO}_2$  plane, while suggesting that modes involving apical and chain oxygen are less strongly coupled to the electrons. This is in agreement with recent inelastic neutron scattering<sup>10,11</sup> and photoemission<sup>12,13</sup> studies, indicating a strong interaction between charge carriers and Cu-O bond-stretching-type of phonons.

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<sup>19</sup>The  $\text{Y}_{1-x}\text{Pr}_x\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  was chosen for the following reasons. (i) This is the only compound where the site-selective oxygen exchange was successfully performed. (ii) The OIE on  $T_c$  and  $\lambda_{ab}(0)$  increases with decreasing doping (increasing  $x$ ). (iii) The oxygen substitution affects only the phonon spectra, but not the crystal and most probably not the electronic structures.

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