

**Substantial oxygen-isotope effect in heavily overdoped  
 $\text{Y}_{1-x}\text{Ca}_x\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ : Quantitative agreement with a  
phonon-mediated model**

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**Abstract**

We have studied the oxygen-isotope effect on the superconducting transition temperature  $T_c$  in overdoped  $\text{Y}_{1-x}\text{Ca}_x\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  with  $x = 0.10, 0.20,$  and  $0.25$ . The oxygen-isotope exponent  $\alpha_O$  is found to be small ( $\sim 0.02$ ) for  $x = 0.10$  but substantial ( $\sim 0.1$ ) for  $x = 0.20$  and  $0.25$ . We show that  $\alpha_O$  jumps up to a nearly constant value of about  $0.1$  above a critical doping level of about  $0.19$ . It is remarkable that this critical doping level coincides with a doping level above which the normal-state pseudogap diminishes. The sizable oxygen-isotope exponent above the critical doping level is in quantitative agreement with a phonon-mediated model based on strong coupling to multiple-phonon modes.

The importance of phonons in the pairing mechanism can usually be checked by the dependence of  $T_c$  on the isotope-mass  $M$  (isotope effect). For most conventional phonon-mediated superconductors, the isotope exponent ( $\alpha = -d \ln T_c / d \ln M$ ) is close to 0.5, as expected from the BCS theory. For high- $T_c$  cuprate superconductors, the isotope effects have been investigated by several groups with consistent results [1–7]. There are small oxygen- and copper-isotope exponents in optimally-doped cuprates, but large exponents (even larger than 0.5) in underdoped cuprates [8]. Due to the high  $T_c$  values and the earlier observation of a small oxygen-isotope exponent ( $\alpha_O \simeq 0.02$ ) in an optimally-doped cuprate superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_{7-y}$  (YBCO) [1–3], most researchers believe that electron-phonon coupling cannot be the origin of 90 K superconductivity in YBCO. On the other hand, there exist large  $\alpha_O$  values and substantial oxygen-isotope effects on the in-plane penetration depth in underdoped cuprates [8–10]. The large oxygen-isotope effects on both  $T_c$  and the in-plane penetration depth in underdoped cuprates can be consistently explained if one considers that  $T_c$  in underdoped cuprates is essentially proportional to  $n_s/m_{ab}^*$  (where  $n_s$  is the supercarrier density and  $m_{ab}^*$  is the in-plane effective supercarrier mass) [11] and that  $m_{ab}^*$  depends on the isotope mass due to a polaronic effect [12]. Other possible effects such as impurity scattering [13, 14] and/or suppression of the density of states due to the normal state pseudogap [13] might also lead to the observed large isotope effects on both  $T_c$  and  $n_s/m_{ab}^*$ . However, these effects [13] cannot simultaneously lead to the observed small  $\alpha_O$  and large oxygen-isotope effect on  $n_s/m_{ab}^*$  in optimally-doped YBCO [15–17]. Several independent experiments [8, 10, 17–19] have also ruled out the oxygen-mass dependence of the carrier concentration [14, 20]. Therefore, the observed large oxygen-isotope effect on  $n_s/m_{ab}^*$  in optimally and underdoped cuprates is only consistent with the existence of polaronic supercarriers.

Now a question arises: What happens to  $\alpha_O$  when the polaronic carriers disappear? Electronic specific-heat data indicate that there exists a critical doping level  $p_{cr}$  of about 0.19 in YBCO and Ca substituted YBCO, above which the normal-state pseudogap disappears [21]. For  $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$  (LSCO), the normal-state pseudogap state is still present up to a doping level of 0.24 (Ref. [21]), implying that  $p_{cr} > 0.24$  in this system. Although the origin of the normal-state pseudogap is still debated, one plausible explanation is that there coexist Fermi-liquid-like carriers and polaronic/bipolaronic oxygen holes and that the pseudogap is related to the bipolaron binding energy [22]. If this scenario is relevant, both the pseudogap and polaronic/bipolaronic oxygen-holes diminish above  $p_{cr}$  so that the superconducting

transition is similar to that in conventional superconductors. If the superconducting pairing is mainly mediated by phonons, the total isotope effect on  $T_c$  should become large above this critical doping level. Therefore, the recovery of the large conventional isotope exponent above this critical doping level would unambiguously expose electron-phonon coupling as an important pairing mechanism.

Here we report studies of the oxygen-isotope effect on the superconducting transition temperature  $T_c$  in overdoped  $Y_{1-x}Ca_xBa_2Cu_3O_{7-\delta}$  with  $x = 0.10, 0.20,$  and  $0.25$ . We find that the oxygen-isotope exponent  $\alpha_O$  is small ( $\sim 0.024$ ) for  $x = 0.10$  and becomes substantial ( $\sim 0.1$ ) for  $x = 0.20$  and  $0.25$ . We show that  $\alpha_O$  jumps up to a nearly constant value of about 0.1 above a critical doping level of about 0.19. It is remarkable that this critical doping level coincides with a doping level above which the normal-state pseudogap diminishes. The sizable oxygen-isotope exponent above the critical doping level is in quantitative agreement with a phonon-mediated model based on strong coupling to multiple-phonon modes.

Samples of  $Y_{1-x}Ca_xBa_2Cu_3O_{7-\delta}$  were prepared by a conventional solid-state reaction using  $CaCO_3$  (99.99%),  $BaCO_3$  (99.997%),  $CuO$  (99.995 %), and  $Y_2O_3$  (99.999 %). The powders were mixed, ground thoroughly, and fired in air at 950 °C for  $\sim 24$  hours with one intermediate grinding. The samples were ground again, pressed into pellets, and sintered in air at 920 °C for  $\sim 20$  hours. To obtain samples with small grains and sufficient porosity for the isotope experiments, we reground the samples thoroughly, pressed them into pellets, and annealed them in air at 800 °C for 20 hours.

Three pairs of the samples with different Ca concentrations were wrapped in gold foil and subjected to  $^{16}O$  and  $^{18}O$  isotope diffusion. The diffusion was carried out for about 48 hours at 750 °C and in an oxygen partial pressure of about 0.8 bar with one intermediate refilling of the isotope gases. The cooling rate from 750 °C to 670 °C is about 1.7 °C/minute, and the average cooling rate from 670 °C to 350 °C is about 10 °C/minute. The oxygen isotope-enrichment was determined from the weight changes of both  $^{16}O$  and  $^{18}O$  samples. The  $^{18}O$  samples were thus found to have  $\sim 96\%$   $^{18}O$  and  $\sim 4\%$   $^{16}O$ .

Magnetization was measured with a Quantum Design sample vibrating magnetometer (VSM). The Meissner effect was measured in a magnetic field of 1 Oe or 10 Oe after the samples had been cooled from the normal state in the field. Data were taken continuously during cooling and warming with a same rate. For most measurements, the cooling and warming rates are 1 K per minute. Such slow cooling and warming rates ensure a very small thermal lag, which is about 0.5 K. Increasing the rate to 3 K per minute increases

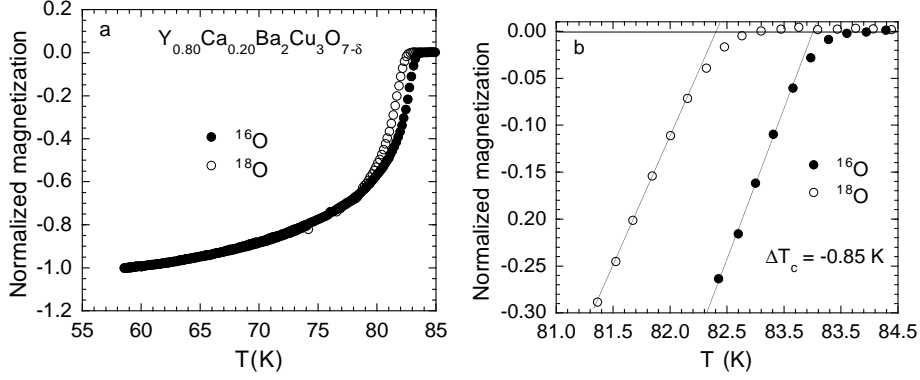


FIG. 1: a) Temperature dependencies of the normalized magnetizations for the  $^{16}\text{O}$  and  $^{18}\text{O}$  samples of  $\text{Y}_{0.80}\text{Ca}_{0.20}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ . b) The expanded view near the transition region. The oxygen-isotope shift is  $0.85 \pm 0.04$  K.

the thermal lag to about 1.5 K, but the isotope shift is nearly independent of the rate. The data in Fig. 1 and Fig. 2 below are corrected for these thermal lags.

Figure 1a show temperature dependencies of the normalized magnetizations for the  $^{16}\text{O}$  and  $^{18}\text{O}$  samples of  $\text{Y}_{0.80}\text{Ca}_{0.20}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ . It is apparent that the magnetizations of the two isotope samples show a parallel shift. In order to see the isotope shift more clearly, we show in Fig. 1b the expanded view near the transition region. We clearly see that the  $T_c$  of the  $^{18}\text{O}$  sample is significantly lower than that of the  $^{16}\text{O}$  sample. If the transition temperature is determined from the linear portion of the magnetization data extended to the base line as indicated in the figure, we find that the isotope shift is 0.85 K. The oxygen-isotope exponent  $\alpha_O = -d \ln T_c / d \ln M_O$  is calculated to be 0.092, which is larger than the Sn isotope exponent (0.08) in the conventional phonon-mediated superconductor  $\text{Nb}_3\text{Sn}$ . In contrast, the isotope shift in  $\text{Y}_{0.90}\text{Ca}_{0.10}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  is significantly smaller, as seen in Fig. 2. The oxygen-isotope exponent is calculated to be 0.024. The magnitude of the isotope exponent for  $\text{Y}_{0.90}\text{Ca}_{0.10}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  is very close to that (0.027) for optimally doped  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  (Ref. [15]).

In Figure 3, we plot  $\alpha_O$  and  $T_c$  as a function of Ca content  $x$ . The data for  $x = 0$  are taken from Ref. [15]. It is striking that the isotope exponent remains constant and small for  $x$  between 0 and 0.10, and jumps to large values (about 0.1) for  $x = 0.20$  and 0.25. We will show that this unusual doping dependence is associated with the disappearance of the normal-state pseudogap above a critical doping level of about 0.19.

The oxygen content of our samples can be estimated from the final cooling rate we use to

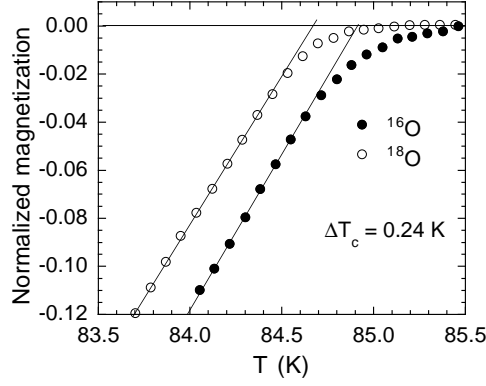


FIG. 2: a) Temperature dependencies of the normalized magnetizations for the  $^{16}\text{O}$  and  $^{18}\text{O}$  samples of  $\text{Y}_{0.90}\text{Ca}_{0.10}\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ . The oxygen-isotope shift is  $0.24 \pm 0.04$  K.

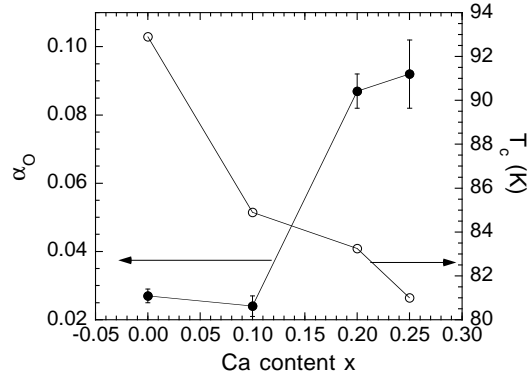


FIG. 3: The oxygen-isotope exponent  $\alpha_{\text{O}}$  and  $T_c$  as a function of Ca content  $x$  in  $\text{Y}_{1-x}\text{Ca}_x\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$ . The data for  $x = 0$  are taken from Ref. [15].

prepare the samples. Studies of the effect of the cooling rate on the oxygen content in ceramic samples of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  indicate that the oxygen content at a cooling rate of  $10^\circ\text{C}/\text{minute}$  has no difference from that at a cooling rate of  $1^\circ\text{C}/\text{minute}$  (Ref. [23]). This implies that a thermodynamic equilibrium is reached at the cooling rate of  $10^\circ\text{C}/\text{minute}$  even in this quite dense ceramic sample. Therefore, we expect that a thermodynamic equilibrium is also reached at the cooling rate of  $10^\circ\text{C}/\text{minute}$  in our porous samples. It is also established [24] that the  $\delta$  value is about 0.07-0.08 for ceramic samples cooled in 1 bar oxygen partial pressure and at a cooling rate of about  $1^\circ\text{C}/\text{minute}$ . Then the  $\delta$  value of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  should be about 0.1 when it is cooled in 0.8 bar oxygen partial pressure and at the cooling rate of  $10^\circ\text{C}/\text{minute}$ . Experiments show [25] that Ca substitution does not change the average Cu valence so that  $\delta = \delta_0 + x/2$ , where  $\delta_0$  is the concentration of the oxygen vacancies for  $x = 0$ . The bond-valence sum calculations (see Fig. 4 of Ref. [26]) indicate that the oxygen

vacancies do not equally reduce the hole doping levels of  $\text{CuO}_2$  planes and  $\text{CuO}$  chains while  $\text{Ca}$  substitution contributes holes mainly to  $\text{CuO}_2$  planes. With  $\delta_0 = 0.1$  estimated from our preparation conditions, we find the  $\delta$  values of our  $\text{Ca}$  substituted samples to be 0.15, 0.20, and 0.225 for  $x = 0.10$ , 0.20, and 0.25, respectively.

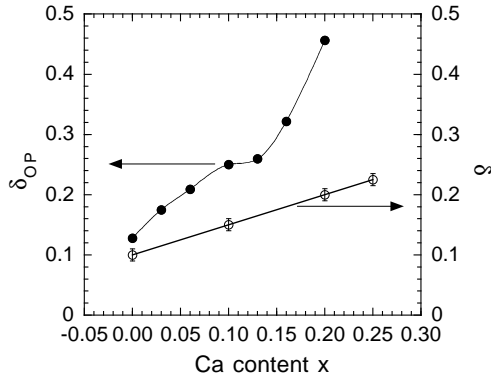


FIG. 4: The concentration  $\delta$  of oxygen vacancies in  $\text{Y}_{1-x}\text{Ca}_x\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  as a function of  $x$  together with the  $\delta_{OP}$  values corresponding to the optimal  $T_c$ 's with  $p = 0.16$ . The  $\delta_{OP}$  data are from Ref. [26].

Figure 4 shows the  $\delta$  values of our  $\text{Ca}$  substituted samples as a function of  $x$  together with the  $\delta_{OP}$  values corresponding to the optimal  $T_c$ 's with  $p = 0.16$ . It is apparent that the difference between  $\delta_{OP}$  and  $\delta$  becomes more pronounced above  $x = 0.13$ . We can estimate the hole doping level  $p$  of  $\text{CuO}_2$  planes from the bond-valence sum calculations which show that  $dp/d\delta = -0.20$  for  $\delta < 0.5$ . Using  $dp/d\delta = -0.20$  and the difference  $\delta_{OP} - \delta$  (see Fig. 4), we calculate the  $p$  values to be 0.180 and 0.210 for  $x = 0.10$  and 0.20, respectively. These  $p$  values may be slightly overestimated due to a possible precipitation of  $\text{Ca}$  out of solid during annealing in the temperature region of 750-800 °C. Furthermore, since a change in the  $\delta$  value of 0.01 leads to a change of  $p$  of 0.002, a possible uncertainty in the  $\delta$  value of 0.04 gives rise to an uncertainty in the  $p$  value of 0.008, which is rather small.

In Fig. 5, we plot the oxygen-isotope exponent as a function of  $p$  for  $\text{Y}_{1-x}\text{Ca}_x\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  ( $\text{Ca-YBCO}$ ) and  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$  ( $\text{BSCCO}$ ). It is remarkable that the  $\alpha_O$  value jumps up to a nearly constant value of about 0.1 above a critical doping level, which is about 0.19 for the  $\text{Ca-YBCO}$  system and about 0.20 for the  $\text{BSCCO}$  system. This critical doping level for the  $\text{Ca-YBCO}$  system coincides with the doping level above which the normal-state pseudogap diminishes. This implies that the substantial oxygen-isotope exponent above the critical doping level is associated with the disappearance of the normal-state pseudogap.

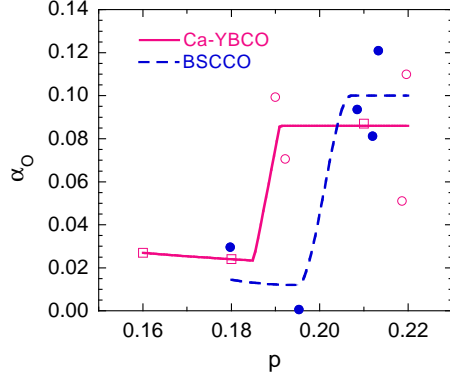


FIG. 5: The oxygen-isotope exponent as a function of the hole doping level  $p$  for  $Y_{1-x}Ca_xBa_2Cu_3O_{7-\delta}$  (Ca-YBCO) and  $Bi_2Sr_2CaCu_2O_{8+y}$  (BSCCO). Solid circles are for BSCCO (Ref. [27]), open squares (this work and Ref. [15]) and open circles (Ref. [27]) are for Ca-YBCO. Solid and dashed lines are guided to eyes. From these lines, it is apparent that the  $\alpha_O$  value jumps up to a nearly constant value of about 0.1 above a critical doping level, which is about 0.19 for the Ca-YBCO system and about 0.20 for the BSCCO system.

Since the existence of the normal-state pseudogap below  $p_{cr}$  makes superconductivity unconventional, the disappearance of the normal-state pseudogap above  $p_{cr}$  should restore a conventional superconducting behavior. The recovery of the conventional superconducting behavior suggests that the total isotope exponent should be close to 0.5 if electron-phonon coupling mainly contributes to the electron pairing. The fact that the observed oxygen-isotope exponent above  $p_{cr}$  is substantial (about 0.1), but still significantly below 0.5, implies that other non-oxygen related phonon modes and/or high-energy electronic bosonic modes also contribute to the electron pairing. Ashauer *et al.* [28] and Rammar [29] calculated various measurable quantities based on strong coupling Eliashberg equations and several hypothesized electron-phonon spectral functions. In the simplest case where the electron-phonon spectral function  $\alpha^2(\omega)F(\omega)$  is proportional to the phonon density of states  $F(\omega)$  measured by neutron inelastic scattering, Rammar [29] calculated the oxygen-isotope exponent to be 0.08-0.10 when the electron-phonon coupling constant  $\lambda_{e-ph}$  is in the range of 1.4 to 6.5. The oxygen-isotope exponent becomes smaller than 0.05 when the coupling constant is less than 0.4. A negative  $\mu_{eff}^*$  value is required to obtain 91 K superconductivity for smaller  $\lambda_{e-ph}$ . For example,  $\mu_{eff}^* = -0.15$  when  $\lambda_{e-ph} = 1.4$ . Within the combined phonon and nonphonon mechanism, a negative  $\mu_{eff}^* = \mu^* - \lambda_{e-el}$  (Ref. [30]) is possible only when the coupling constant  $\lambda_{e-el}$  of high-energy electronic bosonic modes is larger than the Coulomb

pseudopotential  $\mu^*$ . A negative  $\mu_{eff}^*$  also requires that the high-energy electronic bosonic modes are not related to spin fluctuations which normally lead to a repulsive interaction. The observed oxygen-isotope exponents above  $p_{cr}$  (Fig. 5) are in the range of 0.08-0.10, which are in quantitative agreement with the calculated values (0.08-0.10) when  $\lambda_{e-ph} \geq 1.4$  (Ref. [29]). This simple scenario of strong multiple-phonon coupling is also consistent with tunneling and angle-resolved photoemission spectra, which show multiple-phonon coupling features that precisely match with the peak features in the phonon density of states [31, 32].

The tunneling spectrum [33] of optimally doped  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$  indicates that the electron-phonon spectral function  $\alpha^2(\omega)F(\omega)$  is approximately proportional to the phonon density of states  $F(\omega)$  measured by neutron inelastic scattering. This justifies the simple assumption made by Rammar [29]. In fact, this simple relation between the electron-phonon spectral function and the phonon density of states holds in most phonon-mediated superconductors [30]. Moreover, the electron-phonon coupling constant  $\lambda_{e-ph}$  is found [33] to be about 3 in this optimally doped  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+y}$ , which would lead to an  $\alpha_O$  value of about 0.10 if there were no polaronic/bipolaronic effect. The substantial oxygen-isotope effect on the supercarrier mass and the negligible oxygen-isotope on  $T_c$  in optimally doped cuprates can be well explained by a polaronic effect [16]. Above the critical doping level, the polaronic effect may be negligibly small so that the oxygen-isotope exponent  $\alpha_O$  becomes sizable, consistent with the conventional phonon-mediated pairing mechanism.

Since this tunneling spectrum may mainly probe the anti-nodal states that have the maximum coupling constant, the average coupling constant over the entire Fermi surface might be significantly smaller due to a very anisotropic gap symmetry [32, 34, 35]. This highly anisotropic gap arises from the strong long-range electron-phonon coupling which is poorly screened due to the layered structures. Since the electrical resistivity may mainly probe the states near the diagonal direction where the electron-phonon coupling constant is small, the strong multiple-phonon coupling model is not in contradiction with electrical transport data, which have been explained in terms of weak electron-phonon coupling [36].

In summary, we have studied the oxygen-isotope effect on the superconducting transition temperature  $T_c$  in overdoped  $\text{Y}_{1-x}\text{Ca}_x\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta}$  with  $x = 0.10, 0.20,$  and  $0.25$ . We find that the oxygen-isotope exponent  $\alpha_O$  is small ( $\sim 0.02$ ) for  $x = 0.10$  and becomes substantial ( $\sim 0.1$ ) for  $x = 0.20$  and  $0.25$ . We show that  $\alpha_O$  jumps up to a nearly constant value of about 0.1 above a critical doping level of about 0.19. It is remarkable that this critical doping level coincides with a doping level above which the normal-state pseudogap diminishes. The

sizable oxygen-isotope exponent above the critical doping level is in quantitative agreement with a phonon-mediated model based on strong coupling to multiple-phonon modes. The present work thus provides strong evidence for an important role of electron-phonon coupling in high-temperature superconductivity.

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