Study of the onset of superconductivity in underdoped La$_{2-x}$Sr$_x$CuO$_4$

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Abstract. The origin of the appearance of superconductivity in the high-transition-temperature (high-\(T_c\)) copper oxides has remained a subject of active inquiry. In order to address this issue, we chose La$_{2-x}$Sr$_x$CuO$_4$ as a model system and succeeded in fine controlling the hole concentration around the composition where \(T_c\) appears. \(T_c\) did not emerge smoothly at a critical hole concentration, \(x_c\). \(T_c\) and the absolute value of the Meissner signal below \(T_c\) increased progressively with Sr content \(x > x_c\). Measurements of magnetization versus temperature under hydrostatic pressure (\(P\)) are also reported for La$_{2-x}$Sr$_x$CuO$_4$, which has no charge reservoir. A \(dT_c/dP > 0\) found in 0.05 < \(x\) < 0.22 peaks out at \(x \approx 0.07\). Comparison between these results and predictions from existing models for the high-\(T_c\) superconductivity has been made.

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1. Introduction

The high-$T_c$ superconductivity found in the copper oxides in 1986 [1] exhibits three key factors: (a) it occurs in an electron or hole doped Mott insulator, (b) the basic structural unit to support superconductive pairs is the perovskite CuO$_2$ layer and (c) aside from La(O$_{1-x}$F$_x$)$_2$FeAs [2], the copper oxides remain unique to give rise to high-$T_c$ superconductivity.

Although the observation of superconductivity under pressure in the ladder-structure compound Sr$_{0.4}$Ca$_{12.6}$Cu$_{24}$O$_{41.84}$ softens the requirement (b) a little bit [3], it holds for most cuprates in which superconductivity occurs at ambient pressure. The evolution of $T_c$ versus hole density $p$ of the cuprate superconducting systems shows universally a parabolic curve. In the underdoped region, Uemura et al [4] have found a linear relationship between $T_c$ and the superfluid density $n_s(0)$, which is applicable in several cuprate systems. As $n_s(0)$ approaches zero, however, Broun et al [5] have shown recently that the linear relationship between $T_c$ and $n_s(0)$ no longer holds at $T \leq 10$ K in a single-crystal film of YBa$_2$Cu$_3$O$_{6+y}$. In their work, the hole density $p$ in the superconducting CuO$_2$ layers was adjusted continuously by a fine tuning of the total oxidation state of the charge reservoir--CuO$_x$ layers and the CuO$_2$ layers. Although this procedure can vary $p$ in the CuO$_2$ layers with fine steps, the absolute value of $p$ in the CuO$_2$ layers was not determined independently. Under the assumption of $n_s(0) \sim p$, Broun et al [5] derived the power law $T_c \sim (p - p_c)^{0.5}$ in the critical region, which was claimed to be consistent with the scaling behavior of critical phenomena in accordance with three-dimensional (3D)-XY fluctuations near critical doping [6]. To determine the relationship of $T_c$ versus $p$ is as important as the determination of $T_c$ versus $n_s(0)$ for revealing the pairing mechanism in cuprate superconductors. The precise relationship of $T_c$ versus $p$ in the critical region, however, remains largely unknown as far as we know. This situation is partially due to a lack of precise control and determination of $p$ in the superconductively active structural units. The prototype system La$_{2-x}$Sr$_x$CuO$_4$ has the K$_2$NiF$_4$ structure with single CuO$_2$ sheets separated by rock-salt layers consisting of two LaO sheets [7]. The rock-salt layer is not a charge reservoir, which makes the fraction of holes per Cu atom in the CuO$_2$ sheets unambiguously given by the Sr doping $x$ provided oxygen stoichiometry is maintained. The ability to control precisely the hole concentration in the superconductive CuO$_2$ sheets makes this system particularly useful for a study of the physics of the high-$T_c$ superconductive phenomenon in the copper oxides.

A previous work [8] has few compositions covering the underdoped region and has shown an apparently abrupt onset of superconductivity at $x \approx 0.05$ in the La$_{2-x}$Sr$_x$CuO$_4$ system. In this paper, we concentrate on determining the variation of $T_c$ with hole concentration $x = p$ in the neighborhood of the onset of $T_c$. On the other hand, hole doping makes the CuO$_2$ planes flatter...
by releasing the compressive stress in this intergrowth structure, which increases the bandwidth through the Cu–O–Cu bonds. Sr doping in La$_{2-x}$Sr$_x$CuO$_4$ increases both hole concentration and the bandwidth. It has not been well addressed whether the bandwidth $W$ or hole doping $p$ is critical to introduce superconductivity in the underdoped region. Since there is no charge reservoir in La$_{2-x}$Sr$_x$CuO$_4$, the pressure effect here is primarily to increase the bandwidth. We have measured the magnetic susceptibility under pressure of the samples on both sides of $p_c$ in order to clarify this issue.

2. Experimental

Polycrystalline La$_{2-x}$Sr$_x$CuO$_4$ ($0.050 \leq x \leq 0.060$ with $\delta x = 0.001$, $x = 0.070$, 0.090 and 0.110) samples were synthesized by solid-state reaction. One of the component materials for the synthesis, La$_2$O$_3$, absorbs water while it is weighed soon after the material has been heat treated at 980 °C and then cooled down to room temperature, which makes it impossible to have the precise amount of La$_2$O$_3$ in order to change $x$ in steps $\Delta x = 0.001$. We have chosen to use directly off-the-shelf products of La$_2$O$_3$ from Cerac, which is stable and consists of La(OH)$_3$ and La$_2$(CO$_3$)$_3$. The precise amount of La$_2$O$_3$ in this product was determined by thermogravimetric analysis, which shows a 6.5% weight loss when it converts to La$_2$O$_3$ at 980 °C. Starting materials of La$_2$O$_3$, SrCO$_3$ and CuO were carefully well mixed and calcined at 850 °C for 24 h in air. After regrinding, the powders were sintered at 975 °C, then at 1000 °C for 48 h in air. Finally, the powders were pressed into pellets and sintered at 1020–1050 °C for 24 h with slow cooling down to room temperature. The major error in the nominal composition is from the weighing error of the component SrCO$_3$, which contributes an error bar $\delta x \approx 0.0009$. Thermoelectric-power (TEP) measurements on our samples, which give a better resolution to distinguish samples with $\Delta x = 0.001$, indeed show a continuous change of the carrier density as $x$ increases. The series of compounds were confirmed by powder x-ray diffraction (XRD) to be single phase with the K$_2$NiF$_4$ structure; typical XRD of selected compositions are shown in figure 1. Magnetization measurements were performed in a superconducting quantum interference device (SQUID) magnetometer (Quantum Design) between 1.7 and 50 K. A pressure up to $P \approx 1.0$ GPa was applied in a Be–Cu piston–cylinder high-pressure cell designed for the SQUID magnetometer. Silicone oil was used as the pressure medium. The actual pressure at low temperatures was determined by measuring the superconducting transition temperature $T_c$ of a piece of Pb or Sn. Since both transition temperatures of Pb and Sn fall into the temperature range where the La$_{2-x}$Sr$_x$CuO$_4$ in this study exhibits superconducting transitions, we have used the calibration curve of the cell pressure versus turning angle of the pushing screw in our Be–Cu piston–cylinder in order to estimate the cell pressure. For the resistivity measurement under pressure, we have placed a small piece of Pb wire as the pressure manometer in a Teflon cell filled with Daphne oil 7373 as the pressure medium. The hybrid Ni–Cr–Al/Be–Cu cell [9] was mounted on an Oxford $^3$He refrigerator. TEP was measured with home-built equipment [10] between 80 and 320 K. The oxygen contents in all samples studied here were chemically determined by iodometric titration.

3. Results

The lattice parameters at room temperature are plotted as a function of Sr content in figure 2; those for compositions $x = 0.05$ and 0.06 are in good agreement with a previous report [8]. The lattice parameters of all compositions between $x = 0.05$ and 0.06 are in line with those
Figure 1. X-ray powder diffraction patterns for three compositions of La$_{2-x}$Sr$_x$CuO$_4$.

Figure 2. Sr-content dependence of the $a$-, $b$- and $c$-axis lengths. The solid lines in the figure are guides to the eyes.

of the two end members. In the underdoped range, Sr doping increases the $c$-axis length and induces an orthorhombic to tetragonal transition at $x \approx 0.1$ at room temperature. However, the change of lattice parameters for compositions within $\Delta x = 0.001$ is too small to tell whether the actual hole doping changes proportionally to the nominal $x$. We have verified this by TEP measurements. The oxygen contents in the samples were kept at four within the best resolution of iodometric titration.
Figure 3. Sr-content dependence of the TEP at 300 K. The solid line in the figure is a guide to the eyes. The inset shows the temperature dependence of TEP of three compounds with representative compositions.

TEP measurements have proven to be a useful probe of the character of the charge carriers in solids. In general, the TEP contains three contributions:

\[ \alpha(T) = \alpha_s + \alpha_t(T) + \delta\alpha(T), \]

where \( \alpha_s \) is the statistical term that is dominant in the case of small-polaron charge carriers, \( \alpha_t(T) \) is a transport term that varies with the curvature of the charge-carrier energy dispersion curve \( \varepsilon(k) \) at the Fermi energy and \( \delta\alpha(T) \) is a low-temperature enhancement for an itinerant-electron phase. The TEP of the cuprate superconductors has been reported to be dominated by the statistical term, \( \alpha_s \), in the underdoped compositions with \( p \leq 0.1 \) hole carriers per Cu atom of a CuO2 sheet and it is extremely sensitive to the carrier concentration \( p \) [10]. The inset of figure 3 shows the temperature dependence of TEP of three representative compositions; they are typical for the underdoped compositional range in La\(_{2-x}\)Sr\(_x\)CuO\(_4\). The temperature dependence of TEP was almost the same for 0.050 \( \leq x \leq 0.060 \) in La\(_{2-x}\)Sr\(_x\)CuO\(_4\) between 80 and 320 K. The magnitude of the TEP near room temperature as shown in figure 3 changes dramatically with increasing \( p \); the relative change \( \delta\alpha/\alpha \) is as large as 50% for \( \Delta x = 0.01 \). The smooth change of \( \alpha \) indicates that the charge-carrier concentration increases monotonically on crossing the non-superconducting/superconducting boundary around \( x \approx 0.05 \) as \( x \) increases. Given the oxygen stoichiometry verified by iodometric titration, the carrier concentration \( p \) in the CuO2 sheets is well controlled by the nominal hole doping \( x \) of Sr, i.e. \( p = x \). Moreover, the smooth change of \( \alpha \) as \( x \) increases makes the scenario of a static phase segregation unlikely.
Figure 4. Schematic phase diagram for La$_{2-x}$Sr$_x$CuO$_4$. We investigated the superconducting transition temperature $T_c$ for $0.050 \leq x \leq 0.060$, indicated by the circle.

The curve of $T_c$ versus $x$ from the literature, as seen in figure 4, shows an abrupt jump of $T_c$ at around $x = 0.055$. We focused in this study on how superconductivity evolves around the critical doping. The magnetic susceptibility, in dimensionless units, of figure 5(a) shows a high Meissner signal in the $x = 0.06$ sample, which decreases progressively as $x$ decreases. Moreover, the superconducting shielding effect, which is manifest as a splitting between the zero field cooled (ZFC) curve and field cooled (FC) curve at low temperatures, also falls with decreasing $x$. In order to monitor precisely the onset temperature of the Meissner signal for each composition, we use a zoom-in plot in figure 5(b), which shows a very small scale near the zero of figure 5(a). In this plot, however, the unit of the vertical axis is the absolute magnitude of the magnetic moment in emu and all the samples measured have the same mass; therefore, we can discuss a true magnetic moment and the sensitivity limit of a commercial SQUID magnetometer. The pickup coil in a SQUID magnetometer consists of three serially connected coils. A small magnet traveling through these coils creates the response signal of a dipole curve. A uniform sample holder with a dimension larger than the distance between the two outside coils does not contribute any signal. Since the scale of $10^{-6}$ emu is close to the limit of our SQUID magnetometer, a signal falling into this scale is noisy. A measurement in this scale is also subject to any contamination such as a few grains of dust. The signal from a capsule mounted inside a straw is comparable to that from the samples near $x = 0.055$. Therefore, we had to use copper wire as the sample holder. We first selected copper wires that do not show a clear dipole signal within the sensitivity limit of our SQUID magnetometer. However, the copper wire may be contaminated after even a single measurement. We have rechecked all copper wires used in order to make sure they show no signal outside the shaded area in figure 5(b). The program fitting a dipole curve of the SQUID response cannot distinguish whether the signal is from the copper wire or from the sample when the Meissner signal falls inside the shaded area of figure 5(b). The computer may switch automatically from tracking the signal from a sample to that from magnetic impurities in the copper wire. A typical example is an abrupt change of the signal inside the shaded area.
Figure 5. (a) Temperature dependence of the magnetic susceptibility with dimensionless units for underdoped La$_{2-x}$Sr$_x$CuO$_4$ and (b) a zoom-in plot of (a) in the scale 10$^{-6}$ emu for samples with the same mass. The closed and open symbols represent the data measured under the ZFC and FC conditions, respectively. Inset: $T_c$ versus $x$ obtained by using a SQUID magnetometer down to 1.7 K and an Oxford $^3$He refrigerator down to 0.4 K; the size of error bars of $T_c$ is smaller than the symbol. The arrows in the inset represent the absence of superconductivity and the temperature range below 0.4 K is described by gray color.

of $M(T)$ for the $x = 0.058$ sample in figure 5(b). Therefore, we define $T_c$ as the temperature indicated by an arrow for the $x = 0.057$ sample in figure 5(b), where a clear diamagnetic signal from a sample is picked up by the SQUID magnetometer. The curve of $T_c$ versus $x$ obtained is plotted as an inset in figure 5(a). We noticed that the Meissner signal of the $x = 0.056$ sample is anomalously lower, so we excluded this point in the curve fitting. A straight line offers the best fitting for all data points. The cut-off definition of $T_c$ is reliable since all $M(T)$ curves can be overlapped on each other except for $x = 0.056$ by shifting horizontally. It is also clear from figure 5(b) that the $x = 0.053$ sample does not exhibit any Meissner signal to the limit of our SQUID magnetometer. We have further ruled out superconductivity in this sample by a resistivity measurement down to 0.4 K in a $^3$He refrigerator, as shown in figure 6. It is interesting that this sample shows an insulator–metal transition at 3.7 K under 2.4 GPa. For the rest of the samples with $x \leq 0.052$, $T_c$ was not observed down to 1.7 K. We are confident that the solid
Figure 6. Temperature dependence of the resistance of the $x = 0.053$ sample under hydrostatic pressure. Inset: a zoom-in plot of $R(T)$ under 2.4 GPa at $T < 6$ K.

Figure 7. Temperature dependence of the magnetization of La$_{2-x}$Sr$_x$CuO$_4$ with $x = 0.06$ under several pressures. The closed and open symbols represent the data measured under the ZFC and FC conditions, respectively.

line in the inset of figure 5(a) reveals detailed information on how superconductivity evolves on crossing the critical doping.

Since the Be–Cu cell also contributes to the total SQUID response, we have to select samples of $x \geq 0.058$, which have a stronger Meissner signal than that from the cell, for the magnetic measurement under pressure. Figure 7 shows the temperature dependence of the
Figure 8. (a) Sr-content dependence of $dT_c/dP$. The open triangles denote the data reported by Zhou and Goodenough [11]. The solid and dashed lines in the figure are guides to the eyes. The table in the figure gives $dT_c/dP$. $dT_c/dP$ depends on $x$ in region I, and shows almost the same value in region II. In contrast to regions I and II, $dT_c/dP$ exhibits no finite value in region III where the Cu–O–Cu bond angle is 180°. (b) Schematic phase diagram for La$_{2-x}$Sr$_x$CuO$_4$.

magnetization of the $x = 0.060$ sample under several pressures. It is clear from this figure that $T_c$ is shifted to higher temperatures with increasing pressure. The value of $dT_c/dP$ was estimated from $T_c$ measured under four different pressures; a linear relationship between $T_c$ and $P$ was found at least up to $\approx 1.0$ GPa. The absolute value of the magnetization below $T_c$ also increases with pressure. However, the shielding effect seems not to change significantly under pressure. Figure 8 shows $dT_c/dP$ for five compositions studied in this work together with data of other
compositions reported earlier [11] in order to map out the pressure coefficient covering all the superconducting regions of La$_{2-x}$Sr$_x$CuO$_4$.

The variation of $T_c$ induced by pressure is expressed by the following equation:

$$\frac{dT_c}{dP} = \frac{dT_c^i}{dP} + (\frac{dT_c}{dp})(\frac{dp}{dP}),$$

where $p$ is the hole concentration and $dT_c^i/dP$ represents all contributions not influenced by a change of hole concentration with pressure. The variation of $T_c$ under pressure in La$_{2-x}$Sr$_x$CuO$_4$ should be explained by only the first term $dT_c^i/dP$ with no contribution from the second one because Hall coefficients have been reported to be independent of pressure [12], which rules out a pressure-induced carrier-density change as an origin of the $T_c$ enhancement. This picture is consistent with the crystal structure of La$_{2-x}$Sr$_x$CuO$_4$, which has no charge-reservoir layer. For a given hole concentration, $T_c$ increases with the Cu–O–Cu bond angle [11,13]. As shown in this work, the same mechanism to increase $T_c$ under pressure appears to work for compositions near the critical doping range.

4. Discussion

Although the evolution curve between $T_c$ and $p$ holds generally for all high-$T_c$ cuprate systems, it is surprising that the critical $p_c$ in the two systems YBa$_2$Cu$_3$O$_{6+y}$ and La$_{2-x}$Sr$_x$CuO$_4$ is identical. The recently published precise determination of $T_c$ and $n_s(0)$ of a YBa$_2$Cu$_3$O$_{6+y}$ crystal is fundamentally interesting [5]. Without a precise determination of the relationship $T_c$ versus $p$, however, that work remains incomplete for a comprehensive understanding of superconductivity near critical doping. A linear fit for $T_c$ versus $x$ as $x_c$ is approached in figure 5 disproves the speculation that a power law $T_c \sim (p - p_c)^{0.5}$ should hold in this situation. One may argue that the exact relationship of $T_c$ versus $p$ depends on how $T_c$ is defined in a measurement of $\chi(T)$ or $\rho(T)$ or $\lambda(T)$, where $\chi$, $\rho$ and $\lambda$ are magnetic susceptibility, electrical resistivity and penetration depth, respectively. Whether a different definition of $T_c$ can give a power law $T_c \sim (p - p_c)^{0.5}$ remains open. However, our original data in figure 5 are available for analysis.

In the La$_2$CuO$_{4+\delta}$ superconductors, the interstitial oxygen is mobile to below room temperature, which allows for a classic phase segregation below 300 K into an oxygen-rich superconductive phase and an oxygen-poor antiferromagnetic phase [14]. In the La$_{2-x}$Sr$_x$CuO$_4$ system, no ion is mobile below room temperature. In this system, the first-order character of the transition from localized to itinerant electronic states is manifest by local cooperative atomic displacements that create ‘correlation bags’ to give rise to large polarons containing 5–6 Cu center in the underdoped region $0 < x < 0.1$ [15]. A question remains whether, in addition, there is a phase segregation between an antiferromagnetic phase with single-hole polarons and the superconducting phase. In the two-phase model of figure 9(a), the superconductive phase is a thermodynamically stable phase for $p > p_c$. A phase separation into a less-doped phase and the superconductive phase would occur for compositions about $p_c$. The volume fraction of a granular or filamentary superconducting phase would increase with $p$ while $T_c$ remained constant in the two-phase region $p_c < p < p_{c1}$; for $p > p_{c1}$, $T_c$ would start to change as a function of $p$. The curve of $T_c$ versus $p$, including the anomalous point of $x = 0.056$ in figure 5 should not be used to support this model since the Meissner signal of the $x = 0.056$ sample is even weaker than that of the $x = 0.055$ sample. The linear fit of all points except for $x = 0.056$ clearly rules out a two-phase model. On the other hand, the observed $T_c$ versus $p$ in figure 5 does not support a single-phase model in which $T_c$ emerges smoothly from zero as in figure 9(b).
Our observation shows that the doped phase is uniform and superconductive pairs are formed only where the polaron density reaches a threshold $p_c$ as illustrated in figure 9(c).

Hole-doped cuprate superconductors normally show a $dT_c/dP > 0$; in contrast, a $dT_c/dP < 0$ is found for most Bardeen–Cooper–Schrieffer (BCS) superconductors. The curve of $T_c$ versus $p$ in a cuprate system covers three regions, i.e. underdoped (I), optimal doped (II) and overdoped (III) regions. How the superconductive phase responds to hydrostatic pressure in these three regions reveals important information for understanding the high coefficient $dT_c/dP$ and perhaps the high-$T_c$ superconductivity itself. Unfortunately, a literature search indicates that experiments in this regard are incomplete. The high-pressure measurements for the La$_{2-x}$Sr$_x$CuO$_4$ system have been made on only a few compositions, and the results are controversial between different groups before 1992 [16]. Xiong et al [17] have reported $dT_c/dP$ for 13 compositions covering $0.07 \leq x \leq 0.20$. They found a spike near $x = 0.10$ in the curve of $dT_c/dP$ versus $x$. The dramatic change of $dT_c/dP$ near $x = 0.10$ has been attributed to a pressure-induced Fermi-surface topology change. Although we are not able to repeat the same feature that Xiong et al reported, perhaps due to the few compositions we have near $x = 0.10$, the overall magnitude of $dT_c/dP$ for compositions $0.07 \leq x \leq 0.20$ is comparable between our results in figure 8 and that from Xiong et al. An important feature in our results is that $dT_c/dP$ drops abruptly to zero at the orthorhombic to tetragonal phase boundary, which was missed.

**Figure 9.** Schematic plot of $T_c$ versus $p$: (a) $T_c \sim (p - p_{c1}) + \Delta$, (b) $T_c \sim (p - p_c)$, and (c) $T_c \sim (p - p_c) + \Delta$. 
in the report from Xiong et al. Provided that pressure reduces the basal-plane buckling [16] and eventually transforms the orthorhombic phase to the tetragonal phase, our results pointed out that the high-$T_c$ superconductivity prefers flat CuO$_2$ sheets and pressure increases $T_c$ in the orthorhombic phase by reducing the buckling of the CuO$_2$ sheets. It is clear from figure 8 that $dT_c/dP$ is generally higher in the underdoped region than in the optimally doped region, which indicates an additional mechanism in region I to enhance $dT_c/dP$ on top of that through reducing the buckling. We notice that $T_c$ is proportional to superfluid density at 0 K, $n_s(0)$, i.e. Uemura relation in the underdoped region. Pressure may increase $T_c$ by enhancing the efficiency of converting polarons into superconductive pairs. This mechanism no longer exists in the optimally doped region II where the polaron model fails.

It is also interesting to compare these high-pressure results with those of the Fe-based superconductors found recently. Instead of a Mott insulator, the parent compound of the Fe-based superconductor is metallic and shows a spin-density wave (SDW) at 150–200 K [18]. Superconductivity is induced by either doping in charge carriers or applying high pressure. Taking CaFe$_2$As$_2$, for example, the SDW state is suppressed and superconductivity sets in at a pressure as low as 0.5 GPa [19, 20]. In sharp contrast, no report shows that pressure could metallize the parent compounds of the high-$T_c$ cuprate superconductors to induce superconductivity. Moreover, as shown in this work, pressure cannot induce superconductivity in the $x = 0.053$ sample, which has a smaller carrier density than a superconductive sample by only $\Delta x = 0.001$. This comparison suggests that the Mott physics plays an important role in the high-$T_c$ cuprate superconductors. The onset of superconductivity in copper-oxide superconductors depends on the number of charge carriers in the CuO$_2$ sheets and not on the bandwidth. In the Fe-based superconductors, however, superconductivity is induced by changing Fermi-surface topology.

5. Conclusions

Systematic magnetization measurements at ambient pressure and under high pressure on samples that were prepared carefully to cover the critical doping for the onset of the high-$T_c$ superconductivity indicate (i) $T_c$ emerges abruptly at a $p_c$, (ii) a linear relationship of $T_c$ versus $p$ in the range near the critical doping, (iii) a maximum $dT_c/dP$ is obtained in the underdoped region where Uemura’s relation holds and (iv) a critical doping instead of a bandwidth is needed to introduce superconductivity in the cuprates. These observations support that superconductivity emerges within a single phase at a critical hole concentration.

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