Electronic effect of doped oxygen atoms in Bi2201 superconductors determined by scanning tunneling microscopy

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Oxygen dopants are essential for tuning the electronic properties of the cuprate superconductors Bi₂Sr₂Caₙ₋₁CuₙO₂₊₄⁺δ. Here, we study an optimally doped Bi₂Sr₂−ₓLaₓCuO₆⁺δ and an overdoped Bi₂−ₓPbₓSr₂CuO₆⁺δ by scanning tunneling microscopy and spectroscopy (STM/STS). Based on the characteristic features of local STS, three forms of oxygen dopants are identified: interstitial oxygen atoms on the SrO layers, oxygen vacancies on the SrO layers, and interstitial oxygen atoms on the BiO layers. In both samples, the first form dominates the number of oxygen dopants. From the extracted spatial distribution of the oxygen dopants, we calculate the dopant concentrations and estimate the average hole carrier density. The magnitudes of the electronic pseudogap state in both samples are inhomogeneously distributed in space. The statistical analysis on the spatial distributions of the oxygen dopants and the pseudogap magnitude demonstrates that the doped oxygen atoms on the SrO layers tend to suppress the nearby pseudogap magnitude.

cuprate superconductor, scanning tunneling spectroscopy, doping, hole concentration, pseudogap

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1 Introduction

Copper oxide high-temperature superconductors are synthesized by doping the parent Mott insulator [1, 2]. The extra oxygen stoichiometry (δ) of Bi₂Sr₂Caₙ₋₁CuₙO₂₊4⁺δ (BSCCO) superconductors is empirically adjusted by controlled annealing in oxygen atmosphere. The disordered interstitial O atoms introduce hole carriers to the CuO₂ plane. These holes play a primary role in tuning electronic orders, such as superconducting and pseudogap (PG) states. In the phase diagram, the superconducting state exists within a dome-shaped regime, while the critical temperature of PG state monotonically decreases with increasing hole doping [3, 4]. At a fixed low temperature, the monotonically decrease of PG magnitude is observed with the increase of hole doping via adding O dopants [4].

The quantitative oxygen stoichiometry is generally difficult to be predetermined in annealed BSCCO samples. Scanning tunneling microscope (STM) is a microscopic tool of probing samples at subatomic resolution [5]. The in-situ measurement of differential conductance spectra is used to explore the superconducting and PG states at a given spatial location [5]. A few abnormal spectral features have been observed and attributed to the occupation of O dopants with

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different forms [6-8]. After an initial discovery of the interstitial O atoms on the BiO layers in Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ (Bi2212) samples [6], two other forms of O dopants, the interstitial O atoms and O vacancies, were identified on the SrO layers [7, 8]. The spatial distribution of each form of O dopant can be subsequently determined by a characteristic differential conductance map. On one hand, the quantification of the density of O dopants allows an estimation of the hole carrier density to be compared with results from other methods. On the other hand, the precise locations of the O dopants helps a numerical analysis to extract a “local” relation between the O dopants and the inhomogeneous PG state. In Bi2212, different relations were observed as the sample doping was increased from underdoping to optimal doping [7].

In addition to Bi2212, Bi$_2$Sr$_2$CuO$_{6+\delta}$ (Bi2201) samples belong to the other type of layered BSCCO materials. Owing to their crystal structural difference, the electronic properties of these two types of BSCCO samples are quantitatively different. For example, the optimal superconducting critical temperature $T_c$ of Bi2201 ($\approx 32$ K) is much lower than that of Bi2212 ($\approx 90$ K) [9, 10]. The PG state in Bi2201 extends to the overdoped regime [11], while the termination point of the PG state in Bi2212 is inconclusive yet [4, 12, 13]. Following the similar technical procedure of probing the O dopants, the studies of Bi2201 can thus verify both the reliability of the experimental methodology and the applicability of the previous Bi2212 results to the whole BSCCO family.

In this paper, we apply STM to study the O dopants in an optimally doped and an overdoped Bi2201 sample. The spatially dependent differential conductance spectra are collected, accompanying the topographic measurements over a clean area. The voltage is applied over a broad range to capture the abnormal spectral features of the O dopants. The conductance maps at characteristic voltages are used to obtain the spatial distributions of the three types of O dopants. For the interstitial O atoms on the SrO layers, which dominate the number of O dopants, we discuss its correlation with the Pb dopants in overdoped Pb-Bi2201. The influence of the O dopants on electronic properties is subsequently investigated. The hole carrier density is estimated from the O dopant concentration and compared with the Luttinger count of the measured Fermi surface (FS). Finally, in a statistical analysis on the spatial distributions of O dopants and PG magnitudes, we show that the interstitial O atoms on the SrO layers generally suppress the nearby PG magnitude.

### 2 Experimental method

In practice, single crystals of doped Bi2201 [14, 15] are produced by partial substitution of Sr by La, or Bi by Pb. In this paper, we select a Bi$_2$(Sr$_{1-x}$La$_x$)$_2$CuO$_{6+\delta}$ (La-Bi2201 with $x = 0.37$ and $T_c \approx 32$ K) and a Bi$_2$(Sr$_{0.75}$Pb$_{0.25}$)$_2$CuO$_{6+\delta}$ (Pb-Bi2201 with $y = 0.25$ and $T_c \approx 13$ K) to represent the optimally doped and overdoped Bi2201 samples, respectively. The high-quality single crystals of these two samples were grown using the traveling-solvent floating-zone method [16, 17]. Typical samples were cut from the as-grown ingots and annealed with specific temperature and atmosphere conditions [16, 17].

In our ultrahigh vacuum (UHV) STM system [18], single crystal samples were in-situ cleaved at liquid nitrogen temperature and inserted into the STM head for low-temperature measurements at $T = 4.5$ K. The STM topography was taken at a set of the sample bias $V_b$ and setpoint current $I_s$. The local differential conductance ($dI/dV$) spectra, as a function of voltage $V$, were acquired by a standard lock-in technique with a modulation frequency of $f = 983.4$ Hz. The scanning tips were etched electrochemically from tungsten wires, and treated by electron-beam sputtering and field emission cleaning on an Au (111) crystal sample. The representative data of the La-Bi2201 and Pb-Bi2201 samples are presented below.

### 3 Results

Figure 1(a) and (b) display the topographies taken on the exposed BiO layers of La-Bi2201 (30 nm $\times$ 30 nm) and Pb-Bi2201 (25 nm $\times$ 25 nm), both containing a square lattice of Bi atoms. An additional supermodulation structure is

![Figure 1](image-url)
observed in La-Bi2201, while such a supermodulatation is completely suppressed in Pb-Bi2201 due to the elimination of a periodic potential of strain [14, 19]. A part of Bi atoms are substituted by Pb atoms in Pb-Bi2201, represented by brighter spots in the lattice. In the same field of view (FOV) as the two topographies, we take measurements of the differential conductance (dI/dV) spectrum over a broad voltage range of \(-1.3 \, V \leq V \leq 1.0 \, V\). The typical spectra of La-Bi2201 and Pb-Bi2201 are presented in Figure 1(c) and (d), respectively. In the results of La-Bi2201 (Figure 1(c)), dI/dV suddenly decreases with increasing voltage \(V \approx -1.1 \, V \) in the red spectrum, suddenly increases with increasing voltage \(V > 0.8 \, V\) in the blue spectrum, and exhibits a resonance peak around \(V \approx -0.9 \, V\) in the orange spectrum. These three characteristic spectra correspond to atomic defects of different forms. For comparison, the spectrum at a normal position on the BiO layer is also shown in black line. The dI/dV spectra of Pb-Bi2201 (see Figure 1(d)) exhibit similar features, but the resonance peak of the orange spectrum is weaker in Pb-Bi2201 than in La-Bi2201. At the vicinity of \(V \approx -0.9 \, V\), the dI/dV signal of the orange line is consistently larger than the values of the other three characteristic lines, so that we can still reliably identify this defect feature. In previous studies of Bi2212 samples, the three defect features were attributed to three different forms of O dopants [7, 8]. Here we make the same assignment: the rapid increase of dI/dV below \(V < -1.1 \, V\) for an interstitial O atom on the SrO layer, the resonance peak around \(V \approx -0.9 \, V\) for an interstitial O atom on the exposed BiO layer, and the rapid increase above \(V > 0.8 \, V\) for a missing O atom at its lattice site of the SrO layer. For conciseness, we designate the first two defect forms as O\(_i\)(Sr) and O\(_i\)(Bi) respectively, and name the last form O vacancy.

The differential conductance maps of La-Bi2201 and Pb-Bi2201 at \(V = -1.3 \, V\) are displayed in Figure 3(a) and (b), respectively. In both conductance maps, a large number of atomic-scale bright spots appear on a dark background, and each bright spot represents an O\(_i\)(Sr) defect. As a comparison, O\(_i\)(Bi) defects and O vacancies are rarely observed in their characteristic conductance maps [see Figure 2(b), (c), (e), (f)]. In our two Bi2201 samples, the O\(_i\)(Sr) defects dominate over the other two forms of O dopants, which is different from the case in Bi2212 [7]. As the doping increases from underdoped to optimally doped in Bi2212, the O vacancies are gradually filled and its density approaches zero. The present paper investigates optimally doped La-Bi2201 and overdoped Pb-Bi2201, so the densities of O vacancy for both samples are expected to be negligible. On the other hand, the density of O\(_i\)(Bi) defects is quite large in underdoped and optimally doped Bi2212. The negligible density of O\(_i\)(Bi) in both of our samples is possibly material dependent [20].

To further clarify the spatial locations of O\(_i\)(Sr) defects in Pb-Bi2201, we simultaneously measured the topography and differential conductance map \((V = -1.3 \, V\) in an alternative \(5 \, \text{nm} \times 5 \, \text{nm}\) area. As shown in Figure 3(a) and (b), the new FOV is smaller than that in Figure 1(b) and (d), but the spatial resolution of the measurement is improved to \(\approx 0.5 \, \text{Å}\). The centers of the bright spots (marked by white dots) in the conductance map are precisely located on the measured topography of the BiO layer. With respect to the primitive cell of the Bi-O lattice shown in Figure 3(a), each relative position

![Figure 2](image-url)  
**Figure 2** (Color online) Differential conductance maps characterizing the O dopants. For La-Bi2201 (FOV in Figure 1(a)), the voltages are (a) \(V = -1.3 \, V\), (b) \(V = -0.9 \, V\), and (c) \(V = 1 \, V\). For Pb-Bi2201 [FOV in Figure 1(b)], the voltages are (d) \(V = -1.3 \, V\), (e) \(V = -0.9 \, V\), and (f) \(V = 1 \, V\).

![Figure 3](image-url)  
**Figure 3** (Color online) (a) A topography of Pb-Bi2201 (5 nm \(\times 5 \, \text{nm}\)) acquired at \(V_b = 100 \, \text{mV}\) and \(I_s = 50 \, \text{pA}\). The Pb atoms substituting for the Bi atoms are marked by blue dots. (b) The dI/dV map at \(V = -1.3 \, V\) acquired in the same FOV as (a). The O\(_i\)(Sr) defects are marked by white dots. The Pb atoms and O\(_i\)(Sr) defects do not appear simultaneously within the blue and red circles in (a) and (b). (c) Scatter plot of the relative spatial locations of the O\(_i\)(Sr) defects in the BiO lattice.
of the O$_1$(Sr) defect is recorded in a scatter plot as shown in Figure 3(c). Most of these defects are confined to the Bi sites rather than the O sites. Since the STM measurement only determines the ($x, y$) coordinates, the O$_1$(Sr) defects are expected to be located vertically below the Bi sites on the lower SrO layer. On the other hand, the substituting Pb atoms generally occupy the Bi sites of the exposed BiO layer and can be identified by isolated bright spots (marked by blue dots) in the topography [21]. The comparison between the Pb atoms in the topography and the O$_1$(Sr) defects in the conductance map shows a mild correlation, implying that the two defects are spatially independent. For example, only one feature (either Pb atoms or O$_1$(Sr) defects) appears within the two blue and two red circles in Figure 3(a) and (b).

In the family of BSCCO materials, the interstitial O atoms directly introduce hole carriers whose concentration is a fundamental parameter of superconductivity. As the number of interstitial O atoms is quantified in the conductance maps, the hole doping level $p$ can be estimated from the density of the interstitial O atoms, assuming that each O atom provides two holes. In our La-Bi2201 and Pb-Bi2201 samples, this crude estimation yields $p \approx 0.11$ and $p \approx 0.26$, respectively. However, we notice that the estimated hole carrier concentration in optimally doped La-Bi2201 is smaller than the value of $p = 0.16$ used in the empirical Presland [22] and Ando [23] formulas. To further explore this difference, we apply an alternative method of estimating the hole doping based on FS measurements [11, 13]. In the Luttinger theorem [24], the hole doping level $p$ is given by

$$p = 2 \frac{A_{FS}}{A_{BZ}} - 1,$$

where $A_{FS}$ is the area of the hole pocket centered at $(\pi, \pi)$ and $A_{BZ}$ is the area of the square-shaped first Brillouin zone (BZ). In the STM experiment, the FS can be constructed from quasiparticle interference (QPI) patterns in the $dI/dV$ maps of $g(r, V)$ [11, 13, 25-27]. In practice, the ratio map of $Z(r, V) = g(r, V)/g(r, -V)$ and its Fourier transformed map of $Z(q, V) = FT[Z(r, V)]$ are applied, which enhances the QPI signal and cancels the setpoint effect [27]. In the $Z(q, V)$-map, the maximum QPI intensities arise from elastic scattering between high density of states (DOS) regions in the momentum $k$-space. Figure 4(a) and (b) display the two integrated ratio maps of $Z(q) = \sum_V Z(q, V)$ for optimally doped La-Bi2201 (FOV of Figure 1(a)) and overdoped Pb-Bi2201 (FOV of Figure 1(b)), respectively. The integration is over the voltage range of $5 \text{ mV} < V < 20 \text{ mV}$ so that QPI patterns around the Fermi level can be efficiently collected [11, 13]. As shown in Figure 4(a) and (b), each trace of the scattering $q$-wavevectors forms a closed path centered at $(\pi, \pi)$. Following the octet model, the scattering $q$-wavevector is twice the normal-state Fermi momentum $k_F$, i.e., $k_F = q/2$ [11, 13]. In the momentum $k$-space, the scattered data of $k_F$ acquired from the QPI measurement are plotted in Figure 4(c) and (d). To draw the entire FS, we introduce a tight-binding model [28], which expresses the energy dispersion curves as:

$$
\epsilon(q) = \epsilon_0 + 2t_0 \left[ \cos(k_xa_0) + \cos(k_ya_0) \right]
+ 4t_1 \cos(k_xa_0) \cos(k_ya_0)
+ 2t_2 \left[ \cos(2k_xa_0) + \cos(2k_ya_0) \right]
+ 4t_3 \left[ \cos(2k_xa_0) \cos(k_ya_0) + \cos(k_ya_0) \cos(2k_ya_0) \right].
$$

(2)

Following the approach in ref. [11], we fix four parameters, $t_0 = -0.22$ eV, $t_1 = 0.034315$ eV, $t_2 = -0.035977$ eV and $t_3 = 0.0071637$ eV, and allow the reference energy $\epsilon_0$ to be varied. The FS is extracted self-consistently by fitting the solution of $\epsilon(q_F) = 0$ with the QPI-determined Fermi momentums. Figure 4(c) and (d) present the best fitting results of $k_F$ with $\epsilon_0 = 0.211$ eV for La-Bi2201 and $\epsilon_0 = 0.246$ eV for Pb-Bi2201. Subsequently, we calculate the area $A_{FS}$ of the hole pocket and estimate the hole carrier concentration by eq. (1), obtaining $p \approx 0.21$ for La-Bi2201 and $p \approx 0.28$ for Pb-Bi2201. For overdoped Pb-Bi2201, the values of hole doping estimated from the density of O dopants and the Luttinger count are close to each other. For optimally doped La-Bi2201, the two estimations are however different, one below the number of 0.16 in the Presland and Ando formulas and

![Figure 4](Color online) Integrated Fourier transformed ratio $Z(q)$-maps of (a) La-Bi2201 (FOV in Figure 1(a)) and (b) Pb-Bi2201 (FOV in Figure 1(b)). Open red circles mark the scattering momentums $q$ of strong QPI signals. In the first Brillouin zones of the $k$-space of (c) La-Bi2201 and (d) Pb-Bi2201, the Fermi momentums $k_F$ extracted from the QPI $q$-momentums are also shown in open red circles. The solid lines in (c) and (d) are the numerical fitting results of the entire FS following a tight-binding model in eq. (2).
the other above. In fact, the same disparity between the two estimation methods can be found from the data of Bi2212 (we make the same calculations with the Bi2212 data extracted from refs. [7] and [24] and obtain the same behavior). Therefore, quantifying the hole doping is a nontrivial problem and the oversimplified assumptions in these two methods requires theoretical refinement [29].

By virtue of its subatomic resolution, the STM technique can reveal the microscopic relation between the O dopants and the electronic orders such as the PG states. At each spatial location, STM measures an individual $dI/dV$ spectrum, from which a “local” PG magnitude $\Delta(r)$ can be extracted. The spatial distribution of the PG magnitudes can then be depicted in a two-dimensional map. Panels (a) and (b) of Figure 5 display the PG maps of La-Bi2201 (FOV of Figure 1(a)) and Pb-Bi2201 (FOV of Figure 1(b)), respectively. Both PG maps exhibit strong inhomogeneity [11,30-32], with spontaneously formed nanoscale domains of similar PG magnitudes. The average PG magnitudes are $\bar{\Delta} = 31.4$ meV in optimally doped La-Bi2201 and $\bar{\Delta} = 25.0$ meV in overdoped Pb-Bi2201, indicating that the average PG magnitude decreases with the increasing doping. Since the $O_2$(Sr) defects are dominant in both two Bi2201 samples, we can collect unambiguous information by excluding the effects of the other two forms of O dopants. In Figure 5(a) and (b), the $O_2$(Sr) defects (marked by white dots) are superimposed on the PG maps. In La-Bi2201 (see Figure 5(a)), the local PG magnitudes near the $O_3$(Sr) defects are generally smaller than the average value $\bar{\Delta}$, indicating that doped O atoms tend to suppress the nearby PG state. In Pb-Bi2201 (see Figure 5(b)), this tendency is less transparent. However, we can still find that the $O_3$(Sr) defects in the red regimes (small PG magnitude) are more than those in the blue regimes (large PG magnitude), which is consistent with the behavior in La-Bi2201.

For a quantitative measurement, we introduce the distance function $D(r)$, which is defined as the distance between the spatial position $r$ and the nearest $O_2$(Sr) defect [7]. Accordingly, we derive a two-dimensional distance map of $D(r)$ (see Figure 5(c) and (d)). For a given FOV, the normalized cross correlation between the PG map and the defect distribution is given by

$$C(R) = \frac{\int d^2r [D(r) - \bar{D}] [\Delta(r + R) - \bar{\Delta}]}{\sqrt{\int d^2r [D(r) - \bar{D}]^2} \int d^2r [\Delta(r) - \bar{\Delta}]^2},$$

where $\bar{D}$ is the average distance from the nearest $O_3$(Sr) defect [7]. Eq. (3) quantifies a dependence of the distance map reversely on the PG map with a spatial displacement $R$. To further exclude the spatial randomness, an integration over the angular coordinate is performed to give the cross correlation $C(R)$ as a function of the spatial distance $R = |R|$. The results of $C(R)$ for La-Bi2201 (FOV in Figure 1(a)) and Pb-Bi2201 (FOV in Figure 1(b)) are plotted in Figure 6(a) and (b), respectively. In both cases, the zero distance covariances $C(R=0)$ appear around $-0.2$ and a negative cross correlation prevails within a spatial range of nanometers.

Based on its definition in eq. (3), a negative value of $C(R=0)$ indicates the local PG magnitude is more likely to decrease when approaching $O_3$(Sr) defects. On the opposite side of the coin, the $O_3$(Sr) defects tend to be attached to the

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Figure 5 (Color online) (a), (b) Maps of the PG magnitude of La-Bi2201 (FOV in Figure 1(a)) and Pb-Bi2201 (FOV in Figure 1(d)). The locations of the $O_3$(Sr) defects determined from Figure 2(a) and (d) are marked by white dots. (c), (d) Maps of the distance from the nearest $O_3$(Sr) defect for La-Bi2201 and Pb-Bi2201.

Figure 6 (Color online) (a), (b) Cross correlation functions between the PG and distance maps for La-Bi2201 and Pb-Bi2201. (c), (d) Histograms of $O_3$(Sr) density vs. PG magnitude for La-Bi2201 and Pb-Bi2201.
regime of small PG magnitude. To verify these observations, we divide the PG magnitudes of La-Bi2201 and Pb-Bi2201 into intervals $\Delta_j \leq \Delta < \Delta_{j+1}$ and count the number of $O_i(Sr)$ defects in each $j$-th bin. The density of the $O_i(Sr)$ defects, $\rho_j = \rho(\Delta_j \leq \Delta < \Delta_{j+1})$, is then calculated by dividing the defect number over the area with the selected range of PG magnitude. The resulting $\rho_j$ histograms for La-Bi2201 and Pb-Bi2201 are plotted in Figure 6(c) and (d), respectively. In both samples, the $\rho_j$ is a nearly monotonically decreasing function of PG magnitude, confirming the tendency of doped $O_i(Sr)$ atoms to suppress the nearby PG magnitude.

A previous STM study on Bi2212 samples explored the influence of O dopants on the PG state [6, 7]. In the underdoped regime, the PG magnitude was found to be enhanced by the neighboring O dopants of all three forms. In detail, the zero distance covariance $C(R = 0)$ of the O vacancies is almost twice that of the $O_i(Sr)$ defects, while this number becomes very small for the $O_i(Bi)$ defects. Therefore, the PG state is considered to be mainly tuned by the O vacancies. An interesting question to be addressed is whether the other two types of O dopants really enhance the PG magnitude, because their influence might be hidden due to the strong interplay with the O vacancies. As the $O_i(Sr)$ defects are closer to the CuO$_2$ layers than the $O_i(Bi)$ defects, they should more strongly influence the PG state [33]. In the optimally doped Bi2212 sample with a negligible density of O vacancies, the previous experiment showed a negative correlation between the $O_i(Sr)$ defects and the PG magnitude, although the $O_i(Bi)$ defects are still positively correlated with PG magnitude. Our STM result in optimally doped La-Bi2201 and overdoped Pb-Bi2201 further confirms this negative correlation, which is consistent with the global trend that the average PG magnitude decreases with the increase of hole concentration.

4 Summary

In this paper, we apply the STM technique to investigate the O dopants in an optimally doped La-Bi2201 and an overdoped Pb-Bi2201 samples. The characteristic features in the differential conductance spectrum enable us to distinguish three different forms of O dopants: interstitial O defects on the SrO layer ($O_i(Sr)$), interstitial O defects on the BiO layer ($O_i(Bi)$), and O vacancies on the SrO layer. The spatial distributions of these three forms of O dopants are determined by the conductance maps at different characteristic voltages. In both La-Bi2201 and Pb-Bi2201 samples, the number of $O_i(Sr)$ defects is dominant, as compared to those of the other two forms. Accordingly, we explored the microscopic properties of $O_i(Sr)$ defects from three perspectives: (1) In Pb-Bi2201, the precise spatial identification shows that $O_i(Sr)$ defects are mainly located vertically beneath the Bi sites and a mild correlation exist between $O_i(Sr)$ and Pb defects. (2) The hole carrier density is estimated from the measured density of $O_i(Sr)$ defects. The estimation is consistent with the Luttinger count extracted from the STM-measured FS in overdoped Pb-Bi2201 but not in optimally doped La-Bi2201. The large difference between the two estimations in the optimally doped sample requires a future theoretical explanation. (3) We determine a negative dependence of the PG magnitude on the appearance of $O_i(Sr)$ defects. A map is constructed to measure the distance from the nearest $O_i(Sr)$ defect, and a negative correlation is found between the defect distribution and the PG map. The $O_i(Sr)$ defects tend to suppress the nearby PG magnitude, which is confirmed in the histograms of the densities of $O_i(Sr)$ defects. The systematic researches of both Bi2212 and Bi2201 samples can eventually reveal the microscopic mechanism of electronic orders in the family of BSCCO superconductors.

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