Gap like structure in a nonsuperconducting layered oxycarbonate

\[ \text{Bi}_{2+x}\text{Sr}_{1-x}\text{Cu}_2\text{CO}_3\text{O}_{8+\delta} \] single crystal

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The magnetic field and temperature dependence of the in-plane tunneling conductance \( dI/dV(V) \) in high-quality nonsuperconducting (down to 10 mK) layered oxycarbonate Bi\(_{2+x}\)Sr\(_{1-x}\)Cu\(_2\)CO\(_3\)O\(_{8+\delta}\) single crystals has been investigated using break junctions. Combining measurements of the in-plane magnetoresistivity \( \rho_{ab}(T, H) \) and the magnetotunneling, we present evidence for the existence of a small "pseudogap" in a nonsuperconducting cuprate, without local incoherent pairs or any correlation phenomena associated with superconductivity. We are unable to distinguish if such a "pseudogap" is totally unrelated to superconductivity or if its existence is a necessary condition for the subsequent occurrence of superconductivity with increasing carrier density in the sample.

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I. INTRODUCTION

It is currently well established that in the normal state of underdoped high-\( T_c \) superconductors (HTS) there is a pseudogap state, in which the density of states is depleted upon decreasing temperature below a characteristic temperature \( T^* \gg T_c \). Many experiments (for a review, see e.g. Ref. [1]) have investigated this problem, however, the data and their interpretations are still controversial. This is the all-important question of high temperature superconductivity since it connects normal state correlations (referred to the pseudogap) to the origin of the high \( T_c \). At present, there are two markedly different, but nevertheless plausible, explanations of the pseudogap in the normal state. One is associated with the formation of local incoherent pairs above \( T_c \), that is, the pseudogap is a precursor to superconductivity (see e.g. Refs. [2 and 3]), and in the other the pseudogap competes with superconductivity (see e.g. Ref. [4]). Whether the pseudogap state is the precursor to superconductivity or a state that competes with superconductivity has been a matter of long-standing debates.

Very recently Dubroka et al.\(^ 5 \) based on studies of the infrared c-axis conductivity of \( R\text{Ba}_2\text{Cu}_3\text{O}_{7-\delta} \) (\( R \equiv \text{Y}, \text{Gd}, \text{Eu} \)) have suggested that the anomalous normal-state properties of underdoped cuprate superconductors are determined by two distinct correlation phenomena. One of them is due to the competing pseudogap below temperature \( T^* \gg T_c \), whereas the other exhibits a signature of the precursor superconducting state at \( T_c < T < T^{\text{ons}} \). In this state a growth of the spectral weight of the condensate was observed. The magnetic field had a similar effect on the coherence of an electronic state at \( T_c < T < T^{\text{ons}} \) as it has at \( T < T_c \). These results are consistent with other work\(^ 6 \) which also demonstrated that in \( \text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{8+\delta} \) (Bi2212) and \( \text{Bi}_{2+x}\text{Sr}_{2-x}\text{Cu}_0\text{O}_{6+\delta} \) (Bi2201) in the pseudogap region two different states coexist; one is due to pair formation and persists to an intermediate temperature \( T_{\text{pair}} < T^* \) and the second - the "proper" pseudogap - characterized by a loss of the spectral weight and anomalies in the transport properties that extends up to \( T^* \).

It is important to note that Dubroka et al.\(^ 5 \) also surmised that \( T^{\text{ons}} \) remains finite even in heavily underdoped samples where \( T_c = 0 \). This suggestion is in reasonable agreement with our results for the underdoped Bi2201 cuprates reported in Ref. [7]. By measuring angular dependencies of the in-plane and out-of-plane magnetoresistivity at temperatures from 1 K down to 30 mK, we have obtained evidence for the presence of vortex like excitations in a nonsuperconducting cuprate in the pseudogap region (in a conventional superconductor the transition out of the superconducting state is caused by the proliferation of vortices, which destroy the long-range phase coherence).

On the other hand, as pointed out in Ref.[8], HTS are characterized by a relatively small phase stiffness and poor screening, both of which imply a significantly larger role for phase fluctuations. As a consequence, in these materials, the transition to the superconducting state may not display a typical mean-field behavior, and phase fluctuations may have a significant influence on the low-temperature properties. In particular, the onset of long-range phase order controls the value of \( T_c \). In such a scenario, the long-range coherence is lost at \( T_c \) due to fluctuations of the phase of the superconducting order parameter.

Okada et al.\(^ 9 \) have studied the onset temperature of superconducting fluctuations \( T^{\text{ons}} \) of \( \text{Bi}_2\text{Sr}_{2-x}\text{R}_x\text{CuO}_y \) (\( R \equiv \text{La and Eu} \)) by measuring the Nernst effect. They have suggested that the three distinct temperatures, \( T^{\text{ons}}, T^*, \) and \( T_c \), are the consequence of a coexisting competing pseudogap state and incoherent superconductivity, which has been observed below \( T^{\text{ons}} \). The latter is qualitatively different from the pseudogap phenomenon that is characterized by \( T^* \). The experimentally obtained phase diagram indicates that the pseudogap state sup-
presses $T_c$ and enhances superconducting fluctuations. Recently Grbić et al.\textsuperscript{10} have determined the temperature range of superconducting fluctuations above $T_c$ in YBa$_2$Cu$_3$O$_{7−δ}$. Their study shows that the temperature range of the superconducting fluctuations may be as large as 23 K in a deeply underdoped sample. This relatively wide temperature range for the superconducting fluctuations is qualitatively consistent with the theoretical predictions for phase fluctuations in underdoped samples.\textsuperscript{9}

On the basis of present knowledge, it is still not well understood how the phenomena characterized by these three temperatures $T_c$, $T_{onset}$ ($T_{onset}$) and $T^*$ are related to each other and there is currently no consensus concerning the role of the superconducting fluctuations in the temperature region $T_{onset} > T_c$. Hence, the origin of the pseudogap remains unclear but must be understood before HTS problem can be solved. It is however, experimentally well established that the temperature and magnetic field dependence of the resistivity in HTS are essentially determined by the normal-state pseudogap.\textsuperscript{11–14} In this paper, we attempt to find the pseudogap in a nonsuperconducting cuprate without superconducting fluctuations or local incoherent pairs and to study the effect of the magnetic field and temperature on this gap using the tunneling method. This work is motivated by our previous investigation\textsuperscript{7} of vortex like excitations in a non superconducting Bi2201 single crystals. Measurements were performed on layered oxycarbonate Bi$_{2+δ}$Sr$_{4−x}$Cu$_2$CO$_3$O$_{8+δ}$ single crystals.

II. EXPERIMENT

Ceramic oxycarbonate samples with $T_c$ of 30 K were synthesized by intergrowing low-temperature superconductor Bi2201 and nonsuperconducting compound Sr$_4$Cu$_2$CO$_3$.\textsuperscript{15} Single crystals of this oxycarbonate were obtained for the first time in Ref.[16] The Bi$_{2+δ}$Sr$_{4−x}$Cu$_2$CO$_3$O$_{8+δ}$ crystals used for present study were grown in gas cavities in a KCl solution-melt free growth method. The results of a complex analysis of the elemental composition, structure, and superconducting properties have been detailed in a recent publication.\textsuperscript{17} Crystals were mirror-smooth faceted plates with the dimensions 1.5 mm $\times$ (0.4 – 0.8) mm $\times$ (4 – 6) μm with the most developed $ab$ planes. The actual cationic composition of the used samples was measured at 50-70 different points on the crystal and the scatter in the data was less than 2%. The X-ray diffraction analysis confirmed the presence of a single phase in our single crystals. The half-width of the X-ray rocking curves for the crystals was less than 0.1° and does not exceed the instrumental resolution.\textsuperscript{17} These data clearly demonstrate the high structural quality and high homogeneity of the samples on a microscopic scale. We were able to make high quality single-phase superconducting and nonsuperconducting oxycarbonate single crystals by varying the Bi content and consequently the doping level.\textsuperscript{13} Here we have used the fact that the substitution of trivalent Bi for divalent Sr in the Bi compounds reduces the hole concentration in the CuO planes.$^{18}$

The $T_c$ value of the crystals formed by our free growth method can be as high as 36 K.\textsuperscript{17} A microprobe analysis of the investigated samples showed that the nonsuperconducting oxycarbonate single crystals grow at an excess of Bi and have the composition, normalized according to the formula disregarding the CO$_3$ group, Bi/Sr/Cu/O = 2:4:2:8, 2.45 : 3.56 : 1.38 : 8.61 with the lattice parameter $c = 39.289$ Å.

A four-probe contact configuration, with symmetrical positions of the low-resistance contacts on both $ab$-surfaces of the samples was used for the measurements of $R_{ab}$ resistance and the tunneling studies. The temperature and magnetic field dependence of the resistance $R_{ab}(T, H)$ was measured using a lock-in amplifier. For the low temperature magnetotransport measurements, the crystal was placed directly inside the mixing chamber of a top-loading dilution fridge. For the in-plane transport current $J$, a configuration with $H \perp J$ was used.\textsuperscript{7}

The sample for the tunneling measurements is fixed on a flexible substrate. In liquid helium, using a differential screw mechanism, the flexible substrate is bent so that the single crystal breaks along a previously made incision, resulting in a symmetric tunnel break junction Bi$_{2+δ}$Sr$_{4−x}$Cu$_2$CO$_3$O$_{8+δ}$-insulator-Bi$_{2+δ}$Sr$_{4−x}$Cu$_2$CO$_3$O$_{8+δ}$. The details of our break junction setup are described elsewhere.$^{19,20}$ Mechanically re-turning the break junction repeatedly, we were able to fabricate a large number of tunnel junctions at different places along the initial break of the crystal where tunneling occurs in the $ab$-plane. A RuO$_2$ thermometer was used to measure the local temperature of the sample and the temperature was continuously recorded during each measurement.

III. RESULTS AND DISCUSSION

A. In-plane resistivity $ρ_{ab}$

Figure 1 (main panel) displays the temperature dependence of the in-plane $ρ_{ab}$ resistivity for the oxycarbonate single crystal, with a logarithmic scale for the horizontal axis in order to emphasize the low temperature behavior. The data points show the resistivity data at $H = 10$ T and $H = 16$ T (open and closed circles, respectively) with the magnetic field parallel to the $c$-axis. This figure clearly demonstrates that at zero magnetic field, the sample shows an insulating behavior and remains in its normal state down to 10 mK. At ultra low temperatures, $T = 10 – 70$ mK, $ρ_{ab}$ shows a downward deviation (saturation) from the insulating behavior. A similar deviation from the $\log(1/T)$ dependence of the in-plane resistance of Bi2201 in normal state at ultra low temperatures has been studied in detail in Ref.[13].

The inset in Fig. 1 plots the transverse in-plane magneto-
FIG. 1. (color online) Temperature dependence of the in-plane $\rho_{ab}$ resistivity for the oxycarbonate single crystal, with a logarithmic scale for the temperature axis. The data points show the resistivity data for various values of the magnetic field applied parallel to the $c$-axis. The inset plots the transverse in-plane magnetoresistance $\rho_{ab}(H)$ for the same single crystal at various temperatures from 10 mK to 0.5 K with the magnetic field parallel to the $c$-axis. (Sample No. 1)

toresistance $\rho_{ab}(H)$ for the same single crystal at various temperatures from 10 mK to 0.5 K with the magnetic field parallel to the $c$-axis. At ultra low temperatures, the magnetic field dependence shows a small ($\simeq 17\%$ at 10 mK and 16 T) negative magnetoresistance and becomes positive with increasing temperature above 0.3 K (near $\simeq 13\%$ at 0.5 K and 16 T). The observed magnetoresistance behavior of the oxycarbonate single crystal is in complete agreement with that of the negative transverse in-plane magnetoresistance reported in investigations of nonsuperconducting Bi2201 single crystals.\textsuperscript{21} The authors explained these results by localization and the gradual suppression of localization effects by the magnetic field.\textsuperscript{22} Since our results are in agreement with these experiments, it is reasonable to assume that they have the same physical origin.

For comparison in Fig. 2, we show the same data for $\rho_{ab}$ versus the temperature (main panel) and magnetic field (inset) as in Fig. 1, but for our oxycarbonate single crystal No. 2 with less excess Bi. As can be seen from this figure, the sample at zero magnetic field also shows an insulating behavior with a saturation at ultra low temperatures. However, in contrast to the sample No. 1, the magnetic-field dependence of $\rho_{ab}$ of the sample No. 2 shows only a positive magnetoresistance, $\simeq 60\%$ at 15 mK and 16 T, (see inset Fig. 2) in the temperature region 15 mK - 0.5 K. As noted above, in previous studies of Bi2201 single crystals in magnetic field,\textsuperscript{7} we have shown that the large positive in-plane magnetoresistance of the nonsuperconducting cuprates in the insulating state is associated with the presence of vortex like excitations. Similar vortex like excitations have been observed in superconducting cuprates above the zero-field $T_c$ in magnetic fields by the detection of a Nernst signal (see e.g. Ref. [23]). It is not difficult to see that the sample No. 2 is unsuitable for clarification of the major issue of high temperature superconductivity; whether the pseudogap state is the precursor to superconductivity. This is because the temperature $T_{ons}$ ($T_{onset}$ or $T_{pair}$), below which the incoherent superconductivity state exists (precursor of the superconducting state in which fluctuations of the phase of the superconducting order parameter or the vortex like excitations can take place), can remain finite even in heavily underdoped samples where $T_c = 0$.\textsuperscript{5} All these phenomena prevent an investigation of the proper pseudogap since we cannot rule out their influence.

The sample No. 1, on the other hand, shows the insulating behavior without any correlation phenomena associated with superconductivity (Fig. 1). At $T > 0.3$ K, the magnetic field has practically no effect on $\rho_{ab}$. 

FIG. 2. (color online) Temperature dependence of the in-plane $\rho_{ab}$ resistivity for the oxycarbonate single crystal, with a logarithmic scale for the temperature axis. The data points show the resistivity data measured at various magnetic fields applied parallel to the $c$-axis. In the inset the transverse in-plane magnetoresistance $\rho_{ab}(H)$ is plotted for the same single crystal at various temperatures from 15 mK to 0.5 K with the magnetic field parallel to the $c$-axis. (Sample No. 2)
FIG. 3. (color online) (a) Tunneling conductance $dI/dV$ as a function of the bias voltage $V$ for a tunnel break junction at $T = 1.8$ K measured in zero magnetic field (solid line). The dashed line is a smoothed curve obtained by averaging the experimental $dI/dV(V)$ curve (see text). The inset shows the zero-field tunneling conductance at $T = 1.8$ K over a larger voltage range. (b) Tunneling conductance normalized by a smoothed curve (dashed line in (a)). (Sample No. 1)

B. Gaplike structure

Figure 3(a) (main panel) displays the tunneling conductances $dI/dV$ as a function of the bias voltage $V$ for a tunnel break junction fabricated on Bi$_{2+\delta}$Sr$_{4-x}$Cu$_2$CO$_3$O$_{8+\delta}$ single crystal (sample No. 1) at $T = 1.8$ K measured in zero magnetic field (solid line). The inset in Fig. 3 shows the zero-field tunneling conductance at $T = 1.8$ K at higher voltage. It can be seen that the conductance is a parabola as required for a normal-state tunnel structure.\(^{24}\) Note that all the $dI/dV(V)$ investigated here correspond to $T$-independent resistances at large bias as expected for junctions with a good tunnel barrier without current leakage. As can be seen in Fig. 3, $dI/dV(V)$ curves have the shape typical for SIS tunnel break junctions fabricated on HTS single crystals at $T > T_c$. They have a weak (the conductance does not go to zero) but nevertheless well-marked gap like structure.

Since the tunnel conductance in Fig. 3(a) increases sharply with increasing voltage, we show in Fig. 3(b) the normalized tunneling conductance where the gap like structure is better seen. The measured $dI/dV(V)$ curve has been normalized by a smoothed (averaged) curve (dashed line in Fig. 3(a)), which was deduced by broadening the experimental curve (solid line) in order to smooth the gap-related structure. The method used to obtain the normalized tunneling conductance is described in Ref.[25]. The energy gap, defined as half of the peak-to-peak separation of the two main maxima in the $dI/dV(V)$ curves in Fig. 3(b), is 8 meV at 1.8 K.

The tunneling spectra presented in Fig. 3(b) reveal a very large zero-bias conductance with only near 1.5% conductance variation of the gap structures. We have not observed anything similar in the tunneling experiments

FIG. 4. (color online) (a) Tunneling conductances $dI/dV(V)$ for the same tunnel break junction at $T = 1.8$ K for various magnetic fields applied along the c-axis of the crystal. For clarity, the curves have been shifted vertically relative the bottom one. In magnetic field the gap like structure does not change either in shape or in the voltage position (dashed vertical line). (b) Tunneling conductances $dI/dV(V)$ for the same tunnel break junction in zero magnetic field at various temperatures. The curves have been shifted with respect to the upper one. (Sample No. 1)
with Bi2212 single crystals in the superconducting state. In spite of numerous attempts, we were unable to obtain $dI/dV(V)$ curves with a sharp gap structure and a small zero-bias conductance as found for the superconducting break junctions on the Bi2212.\footnote{Kanigel et al.,\textsuperscript{28} Chien et al.\textsuperscript{29}}

Figure 4(a) displays the tunneling conductances $dI/dV(V)$ for the same tunnel break junction at $T = 1.8$ K for various magnetic fields applied along the c-axis of the crystal. For clarity, the curves have been shifted vertically relatively the lowest curve. It is significant that there are no changes in the $dI/dV(V)$ curves with magnetic field, notably the gap like structure does not change either in shape or in the voltage position (dashed vertical line).

Figure 4(b) shows the tunneling conductances $dI/dV(V)$ for the same tunnel break junction in zero magnetic field at various temperatures. The curves have been shifted vertically with respect to the upper one. As temperature increases the gap like structure broadens and diminishes in amplitude rapidly with a little shift toward higher voltages. Similar behavior of the tunneling spectra has been observed in studies of HTS at temperatures above $T_c$ by using a scanning tunneling microscope (see e.g. Ref. \textsuperscript{[26]}) and mesas, (see e.g. Ref. \textsuperscript{[27]}) providing evidence for the evolution of a superconducting gap in a pseudogap in the normal state.

In our case it is simply not possible to relate the observed gap like structure in our $dI/dV$ spectra to the superconducting gap: Firstly, because any “tails” of superconductivity in sample No. 1 are lacking (Fig. 1). Secondly, the voltage position of the gap peak in $dI/dV(V)$ remained unchanged in the applied magnetic fields (Fig. 4(a)), whereas, the superconducting gap peak simply must shift to lower voltages with increasing field.\footnote{Kurosawa et al.,\textsuperscript{32} Chien et al.\textsuperscript{30}} Thirdly, the superconducting gap peak must shift to lower voltages with increasing temperature,\footnote{Kurosawa et al.,\textsuperscript{33} Chien et al.\textsuperscript{30}} which emphatically is not what is observed in Fig. 4(b). Thus it seems reasonable to assume that the gap like structure observed here in the $dI/dV$ spectra is associated with a some “pseudogap” in the electronic density of states, which exists in our nonsuperconducting cuprate. In this case such a gap is either unrelated to superconductivity at all or the existence of such a gap is a necessary condition for the subsequent occurrence of superconductivity with increasing carrier density in the sample.

Recent ARPES studies (see, e.g. Lee et al.,\textsuperscript{28} Kanigel et al.,\textsuperscript{29} Chien et al.\textsuperscript{30}) of Bi2212 showed that the pseudogap as well as the superconducting gap are anisotropic. Above $T_c$, there is a gapless Fermi arc near the nodal region of momentum space and away from this Fermi arc region, the well-known pseudogap gradually takes over and reaches its maximum at the antinodal region. In our break junctions fabricated on single crystals of the layered cuprate superconductors, the tunneling occurs along the $ab$ plane. Since the in-plane energy gaps are highly anisotropic, the tunneling conductance spectra can be quite different depending on the tunneling direction relative to the crystalline axes, and do not always show maximum value of gap corresponding the bulk density of states (see e.g. Kashiwaya et al.,\textsuperscript{31} Tanaka et al.\textsuperscript{32}).

According to these articles, in the case of $ab$-plane tunneling, the main effect of varying the tunneling direction in $d$-wave superconductors resides in the appearance of a zero-bias conductance peak for certain tunneling directions. Needless to say, we could not reconstruct the break junction in such a way as to investigate all points of a Fermi surface. Nevertheless, a number of the tunnel break junctions exhibited $dI/dV(V)$ curves with a zero-bias conductance peak. Figure 5 displays the zero-field tunneling conductances $dI/dV(V)$ for tunnel break junctions which appear to be along two distinctly different directions of the $ab$-plane for the same single crystal at $T = 1.8$ K. Curve 1 has the gap like structure as in Fig. 3 and Fig. 4, but with a gap value equal to 15 meV, whereas, curve 2 has a zero-bias conductance peak. The large value of the peak relative to the gap structure in the curve 1 is probably associated with different barrier heights.\textsuperscript{31}

In our opinion, it is not surprising that the magnitude of the “pseudogap” observed here is much lower than the typical values which are usually identified as the pseudogap. Similarly small values of the pseudogap have been reported in underdoped Bi(La)2201,\footnote{Kurosawa et al.,\textsuperscript{33} Chien et al.\textsuperscript{30}} and in electron doped cuprates PrCeCuO\textsubscript{4}.\footnote{Kurosawa et al.,\textsuperscript{33} Chien et al.\textsuperscript{30}} Furthermore, Kurosawa et al.\textsuperscript{33} based on studies of the underdoped Bi(La)2201 using scanning tunneling microscopy/spectroscopy showed that on the antinodal parts of the Fermi surface in momentum space there are two different types of gap. One is the usual large pseudogap, whose gap width is much larger than that of a $d$-wave pairing gap, and the other is the small pseudogap, whose gap width is comparable to that of simple $d$-wave gap. This small pseudogap can open inside the
large pseudogap at a lower temperature $T_c$.\textsuperscript{33}

It is also important to note that the temperature and magnetic field dependence of the resistance and tunneling measurements were performed on several nonsuperconducting single crystals similar to sample No. 1 and the extracted magnitudes of the energy gap were in close agreement. However, obtaining a complete data set on a given single crystal is difficult since tunnel break junctions are often unstable with time and changing temperature and magnetic field. We show here measurements only for the single crystal for which we have a complete data set (temperature and magnetic field dependence of the resistance and tunneling characteristic). However, we stress similar results have been obtained on a number of other single crystals.

\section{IV. CONCLUSION}

We have studied the magnetic field and temperature dependence of the in-plane tunneling conductance in high-quality non superconducting (down to 10 mK) layered oxycarbonate $\text{Bi}_{2x+z}\text{Sr}_{4-x}\text{Cu}_2\text{CO}_3\text{O}_{8+\delta}$ single crystals. Combining measurements of the in-plane magnetoresistivity $\rho_{ab}(T, H)$ and the magnetotunneling, we present evidence for the existence of a small "pseudogap" in the nonsuperconducting cuprate without local incoherent pairs or any correlation phenomena associated with superconductivity. This suggests that the observed "pseudogap" in HTS cuprates is either unrelated to superconductivity at all, or that the existence of this gap may be a necessary condition for the occurrence of superconductivity with increasing carrier density in the sample.

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