First Scanning Tunnelling Spectroscopy on Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10+\delta}$ single crystals

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Abstract

We report the first low temperature scanning tunnelling microscopy and spectroscopy study of high quality Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10+\delta}$ crystals. We present atomic resolution and show spectroscopic data acquired on two different samples. In one case, for $T_c = 109$ K and a transition width of only 1 K, we obtained an extremely homogeneous sample with $\Delta p = 60$ meV over at least 50 nm. In the other case, the respective parameters were $T_c = 111$ K and $\Delta T_c = 1.7$ K and yielded a slightly less homogeneous sample with $\Delta p = 45$ meV. We evidence strong similarities with Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ and discuss the doping level of our samples.

Key words: HTS, Bi2223, spectroscopy, tunnelling, homogeneity
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1 Introduction

The trilayer cuprate Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10+\delta}$ (Bi2223) attracted a strong interest due to its high critical temperature $T_c^{\text{max}} = 111$ K and its potential for applications. However, the difficulty in synthesizing large sized single-phase crystals significantly impeded the study of fundamental properties. Very recently the effort in developing new crystal growth processes was rewarded by a successful production of homogeneous high quality Bi2223 single crystals [1], thus opening the door to scanning tunnelling spectroscopy (STS) investigations. The determination of the intrinsic superconducting properties of this trilayer compound is of crucial importance for the determination of the generic features and behaviors of Bismuth based cuprates and more generally in the quest of the understanding of high-$T_c$ superconductivity.
In this paper, we present the first low temperature STS study of high quality Bi2223 single crystals. We investigate the spatial dependence of the local density of states (LDOS) of two different samples and discuss parallels with the parent double layer compound Bi$_2$Sr$_2$CaCu$_2$O$_{8+\delta}$ (Bi2212).

2 Sample characterization

We studied two Bi2223 single crystals grown by the travelling solvent floating zone method [2], which were post-annealed under different conditions. Sample $A$ was treated at 500 °C, 20 bar O$_2$ during 50 hours and yielded $T_{\text{onset}} = 109$ K and an extremely sharp transition width $\Delta T = 1$ K as measured by SQUID magnetometry. For sample $B$ the conditions were 500 °C, 100 bar O$_2$ during 50 hours, giving $T_{\text{onset}} = 111$ K and a larger transition width $\Delta T = 1.7$ K, indicating less homogeneity. The measurements presented here were obtained using a home built scanning tunnelling microscope which operates under ultrahigh vacuum and low temperatures [3]. The vacuum tunnel junctions were made between the in-situ cleaved Bi2223 (001) surface and an electrochemically etched iridium tip mounted perpendicular to the sample surface. All spectra where obtained at 1.8 K and zero magnetic field with tunnelling resistances ranging between 0.5 and 1 GΩ.

3 Results and discussion

In Fig. 1a we present a topographic scan showing atomic resolution. We further observe a 26 Å supermodulation along the $b$-axis which is also seen in Bi2201 [4] and Bi2212 [5] where it is attributed to extra oxygen in the BiO planes [6]. In Fig. 1b we show a typical IV-curve and its corresponding differential conductance. The IV-characteristic clearly shows a metallic-like background over a large energy range and the existence of a clear $d$-wave gap at the Fermi level. The differential tunnelling conductance presents well-developed coherence peaks at $\pm \Delta_p \simeq 56$ meV and clear dip-hump structures which appear asymmetrically on both sides of the Fermi level. These features are consistent with Bi2212 [5,7], where the broad hump observed at negative bias has been related to a van Hove singularity [8].

The power of STS lies in the ability to investigate the spatial dependence of the LDOS. Recently, much attention has been paid to the question whether the electronic structure of high-$T_c$ cuprates is intrinsically inhomogeneous at the nanometer scale [9] or if long range homogeneity of the spectral signature can be obtained. In fact, for overdoped Bi2212 it has been demonstrated that an appropriate crystal preparation yields homogeneous samples [10]. A necessary,
Fig. 1. (a) Topographic image of in-situ cleaved Bi2223 obtained under UHV at 1.8K ($I=0.8\text{nA}$, $V_s=0.6\text{V}$). The 13x13nm$^2$ inset shows the 26 Å supermodulation. (b) Typical IV-curve and corresponding $dI/dV$ spectrum acquired on sample B at 1.8K ($R_t=1\text{GΩ}$).

![Topographic image and IV-curve](image)

Fig. 2. Trace of 201 spectra along 50nm line on sample A at 1.8K ($I=0.6\text{nA}$, $V_s=0.3\text{V}$).

However not exclusive, criterium for sample homogeneity is a sharp superconducting transition width $\Delta T/T_c < 1\%$ in susceptibility measurements.

In the Figs. 2 and 3 we compare traces of equidistant spectra acquired along a line on the surfaces of sample A and B respectively. As anticipated by the sharp transition width $\Delta T/T_c = 0.9\%$, sample A shows an extremely high homogeneity, as demonstrated by the gap distribution in Fig. 4: the standard deviation $\sigma$ is of only 3 meV for a mean gap $\bar{\Delta}_p = 60$ meV. In contrast, sample B, with $\Delta T/T_c = 1.5\%$, shows a much broader distribution $\sigma = 7$ meV and $\bar{\Delta}_p = 45$ meV. This behavior is consistent with the empirical criterium mentioned above and demonstrates that homogeneity can be obtained on Bi2223 over a range of at least 50 nm. Note the spatial reproducibility of
Fig. 3. Trace of 201 spectra along 100nm line on sample B at 1.8K (I=0.6nA, $V_s=0.6V$).

Fig. 4. Gap distributions of sample A and B.

the background conductance outside the peak-dip-hump structure for both traces, which is characteristic for a stable vacuum tunnelling junction.

The doping relation $T_c(p)$ of Bi2223 has not been established yet. However, various observations point out that $T_c(p)$ could deviate from the generic dome shaped relation by Presland et al. [12]. Indeed, the doping dependence has been studied by other methods suggesting a large plateau with $T_c = T_{c,\text{max}}$ in the overdoped phase [13,14]. Furthermore, it has been found that Bi2223 is far less sensitive to oxygen doping than Bi2212 since upon post-annealing only small variations of $T_c$ are achieved, even with high oxygen pressure treatments [13]. With that respect it is striking that for a variation of only 2% of $T_c$ we observe
Fig. 5. Comparison of the LDOS doping dependence between Bi2212 (Ref. [11]) and Bi2223. All spectra have been normalized to the background conductance at $V_s=300\text{meV}$ and shifted vertically for clarity. $\alpha$ is the slope of the background fit at $V_s=-200\text{meV}$. The Bi2223 panel shows the average spectra of the Figs. 2 (bottom) and 3 (top).

Two observations allow us to tentatively assign sample A and B as being underdoped and optimally doped, respectively. Firstly, the gap magnitude is larger in sample A than in B. Assuming that the gap falls monotonically with increasing doping, as established for Bi2212 [11,15] and suggested for Bi2223 by $c$-axis conductance experiments [14], this indicates that sample A has a lower hole content than B. Secondly, for sample B we have $T_c = T_{c,max}$, which is characteristic for optimally or possibly overdoped samples [13].

This conclusion is corroborated by a careful comparison of the background conductance in Bi2212 and Bi2223 spectra. Various tunneling experiments on Bi2212 revealed that the background conductance outside the gap is asym-
metric and varies with doping [11,7], the strongest effect occurring below the Fermi level. The background slope $\alpha$ taken reasonably far from the Fermi level and the dip-hump structure ($V_s \simeq -200$ meV) is positive for overdoped samples, about zero for optimal doping and negative for the underdoped case, as illustrated in the top panel of Fig. 5. In Bi2212, this observation has been attributed to a broad van Hove singularity at negative bias that shifts away from the Fermi level as $p$ decreases [8]. In the lower panel of Fig. 5 we show the average spectra of the traces on sample $A$ and $B$. Focusing on the background slope at negative bias shows a striking similarity between underdoped Bi2212 and sample $A$, and respectively between optimally doped Bi2212 and sample $B$.

We now turn to the analysis of the LDOS variations observed along the trace of sample $B$. Fig. 6 displays the average spectra corresponding to the energy intervals of the gap distribution shown in Fig. 4. The systematic decrease of the coherence peak intensity with increasing gap magnitude and the corresponding dip-hump shift, are strikingly similar to what is observed in Bi2212 when reducing the doping [16,17]. Furthermore, for each spectrum we again determined the slope $\alpha$ at -200 meV which appears to depend linearly on the gap magnitude (see Fig. 6 inset). In addition the sign change of the slope occurs at what is believed to be optimal doping. With the tentative background versus doping relation discussed above, it is therefore possible to interpret the spatially varying LDOS in less homogeneous samples as being related to a local variation of doping, as reported in Bi2212 [18].
4 Conclusion

We showed the first STS study on Bi2223 single crystals. Atomic resolution as well as the characteristic supermodulation have been observed. One sample with a transition width of only $\Delta T/T_c = 0.9\%$ exhibits an extremely homogeneous LDOS. The LDOS features, i.e. the $d$-wave spectral shape, the strong coherence peaks and the existence of a dip-hump structure, as well as the various LDOS on the surface of a less homogeneous sample, show striking similarity with Bi2212. Using the doping dependence of the background conductance and of the gap of Bi2212 as a template, we made a qualitative estimate of the doping level of our samples. We further evidenced a linear dependence and sign change of the slope of the background as a function of the gap magnitude.

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