Article

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Reference

KUGLER, Martin, *et al.* A ^{3}He refrigerated scanning tunneling microscope in high magnetic fields and ultrahigh vacuum. *Review of Scientific Instruments*, 2000, vol. 71, no. 3, p. 1475-1478

DOI : 10.1063/1.1150483

A ^{3}He refrigerated scanning tunneling microscope in high magnetic fields and ultrahigh vacuum

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(Received 28 July 1999; accepted for publication 9 November 1999)

We present a scanning tunneling microscope (STM) designed to operate between 275 mK and room temperature, in magnetic fields up to 14 T and in ultrahigh vacuum (UHV). The system features a compact STM connected to an UHV compatible ^{3}He refrigerator fitting into a bottom loading cryostat with a superconducting magnet. In this configuration, the cryostat is sitting on top of the UHV chamber, resulting in a very short distance between the STM access and the experimental position. It further enables proper thermal anchoring of the entire STM setup, allowing millikelvin temperatures to be reached in true UHV conditions. We achieve a hold time of about 40 h at 275 mK and a turnaround time of 10 h between room and base temperature. We demonstrate atomic resolution and present tunneling spectra obtained at 275 mK on the high-T_c superconductors $Bi_2Sr_2CaCu_2O_{8+\delta}$ and $YBa_2Cu_3O_{7-\delta}$. *© 2000 American Institute of Physics.*
[S0034-6748(00)02503-X]

I. INTRODUCTION

Scanning tunneling microscopy has become a widely used technique since its development in 1982 by Binning and Rohrer.[1] Over the years, there has been a large effort in instrumentation design, in particular towards scanning tunneling microscopes (STMs) operating at low temperatures in ultrahigh vacuum (UHV).[2] In addition to its fantastic ability to image surfaces with atomic-scale resolution, the STM has proven to be a unique tool for spatially resolved tunneling spectroscopy. In recent years, the interest in locally probed electron spectroscopy has been steadily growing, especially in the field of superconductivity. The direct access to the local density of states (LDOS)[3] with very high energy and spatial resolution, gives the possibility to investigate crucial characteristics of the superconducting state like the quasiparticle gap, the pairing symmetry, the low-energy excitations in the vortex cores,[4–6] as well as eventual pseudogap signatures present in the normal state.[7,8] A further remarkable demonstration of scanning tunneling spectroscopy (STS) is the successful real space imaging of the vortex cores and the vortex lattice by mapping the spatial variation of the LDOS.[4–6,9–11]

Up to now, only few STM/STS experiments have been performed below 1 K,[4,12,13] demonstrating the promising scientific insight that is within reach. Nevertheless, achieving scanning tunneling spectroscopy at millikelvin temperatures, in high magnetic fields and in UHV, still remains a challenge. The effort is worthwhile, as the very low temperatures will give the opportunity to study unconventional low-T_c superconductors like strontium–ruthenates[14] and heavy fermions or magnetic superconductors[15] like chevrel phases and borocarbides. Together with high magnetic fields it will furthermore give a chance to investigate magnetic field induced superconductivity (MFIS).[16] Also for high-T_c superconductors it is crucial to be able to make investigations both at high fields and low temperatures for a better understanding of their anomalous non-BCS behavior. Finally, true UHV conditions are essential if accurate spectroscopy on materials with highly sensitive surfaces is aimed.

We succeeded in developing such a low temperature STM, which features a large temperature range reaching down to sub-Kelvin temperatures, high magnetic fields, and UHV. In this article, we will mainly focus on the cryogenic setup and show familiar results obtained on well-known compounds with the goal to demonstrate the stability of the instrument at very low temperatures in UHV. The STM head has been described in details elsewhere.[17,18]

II. SYSTEM DESIGN

Good thermal anchoring to reach millikelvin temperatures while keeping the vibration level low, is a very important condition for any low temperature STM application in UHV. Additional constraint arises from the need of *in situ* tip and sample exchange as well as from the limited space available within the bore of a conventional superconducting magnet. Central features of our design are a compact STM,[17,18] an UHV compatible single-shot ^{3}He refrigerator, and a bottom loading cryostat. The STM is attached to the ^{3}He pot by a spring to provide vibration isolation. Flexible copper wires bridge the spring to achieve a sufficient thermal contact.

The bottom loading configuration, although implying a more complex cryostat design compared with conventional

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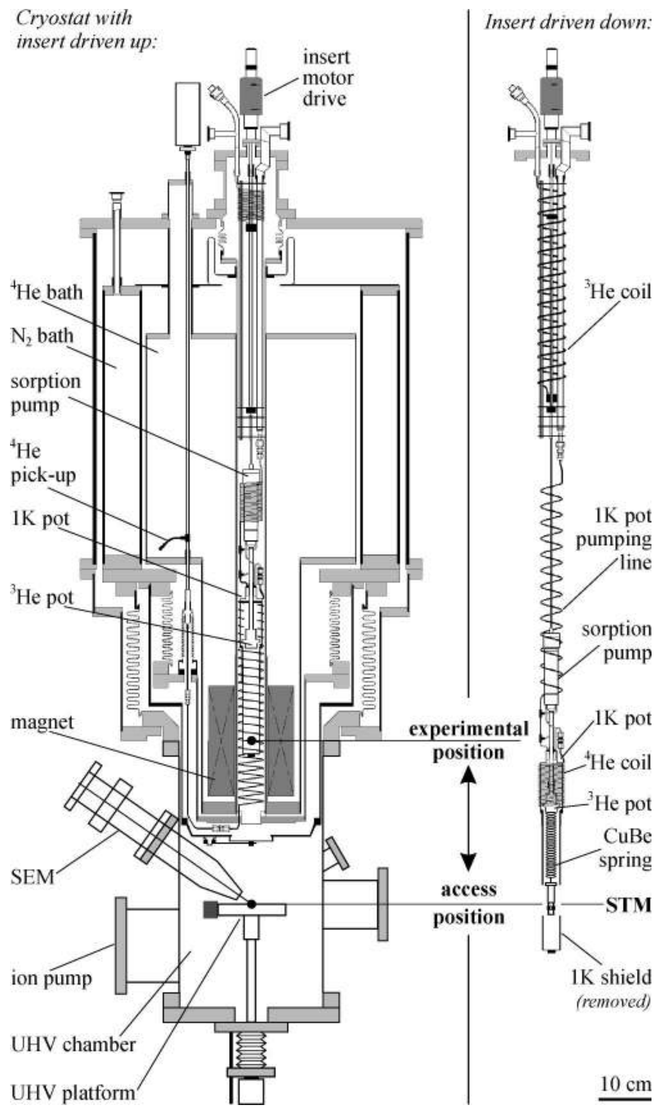

FIG. 1. Complete system setup. On the left-hand side, the cryostat is shown with the inset driven up for STM measurements at low temperatures and high magnetic fields. On the right-hand side, the inset is driven down, to remove the bottom of the 1 K shield and to access the STM for *in situ* tip and sample exchange.

top loading systems, offers numerous advantages. It substantially reduces the distance between the experimental and the STM access positions (see Fig. 1 for their exact location), resulting in a very compact construction. A special coiled capillary design[20] supplying liquid ^{4}He to the ^{3}He refrigerator, allows the complete ^{3}He insert together with the STM, to travel between both positions. This leads to a much lower reference temperature compared to the one obtained by pressing the STM against a cold finger in UHV, and it provides ideal thermal anchoring for all electrical and mechanical connections between the top of the cryostat and the STM. Whether the STM is in the in-field experimental position at low temperatures or in the access position for *in situ* tip and sample exchange, it always stays in the same UHV environment and is never disconnected from the control electronics, thus keeping the tunnel junction under permanent control. Finally, while the STM warms up to room temperature when accessing tip and sample, the ^{3}He insert stays below 40 K. This avoids warming up the sorption pump to room temperature, thus maintaining the cryogenic vacuum and considerably shortening the cooling time of the microscope.

To reduce vibrational noise, we adopted essentially a two stage vibration isolation: (i) the entire system stands on a building independent basement and the pumping lines are fed through concrete blocks, thus eliminating most vibrations originating from the building and the roughing pumps, (ii) the STM is decoupled from the cryogenic assembly by the spring (≈ 2 Hz resonance frequency) which connects it to the ^{3}He pot. At present, damping is only provided by the wires running between the STM and the ^{3}He pot.

III. CRYOGENICS

The cryostat and the single-shot ^{3}He refrigerator have been designed with the distinctive aims of being bottom loading, with a fast cooldown and a long hold time at base temperature.[19] As shown in Fig. 1, the refrigerator is housed in an UHV tube (clear bore $\phi = 56$ mm) which is open at the bottom. To allow the travel of the ^{3}He insert, a patented design[20] of two capillary coils has been developed. One coil is dedicated to run the 1 K pot and the second connects the sorption pump to an external ^{3}He storage vessel. The 1 K pot is directly supplied with liquid ^{4}He by a capillary tube (^{4}He coil) which is connected to the main bath at the cryostat bottom. The same tube then operates as the 1 K pot pumping line and is further used to cool the sorption pump. Thus a single flow of ^{4}He enables the complete insert to run. The flow is regulated by a needle valve at the ^{4}He pickup. Pumping the 1 K pot through long coiled capillary tubing still allows a 1 K pot reference to achieve a temperature of about 1.6 K with a 63 m^3/h pump.

To obtain the best cryogenic performances of the system, all electrical and mechanical connections from the top are thoroughly thermalized at the 1 K pot. The heat load is further reduced by enclosing both, the ^{3}He pot and the STM, within a radiation shield connected to the 1 K pot (1 K shield). To achieve proper cooling and a rapid thermal equilibrium on the STM, it is connected to the ^{3}He pot by a CuBe spring and by very fine copper braids which provide additional heat conductance to the sample holder. To optimize the thermalization of the instrument itself, we used throughout highly thermal conductive materials.[21]

The single-shot refrigerator can run at a base temperature of 275 mK for about 40 h with a total of 4.5 l ^{3}He gas and a residual heat leak to the ^{3}He pot of about 25 μW. After *in situ* tip and sample exchange at room temperature, the STM is cooled back to 275 mK within 10 h, 90% of the time being necessary to reach 20 K.

At present, no active temperature regulation is installed on the STM itself. The ^{3}He pot temperature acts as the STM reference temperature and can be regulated between 275 mK and room temperature. Monitoring the STM and ^{3}He pot temperatures showed, that below 20 K the thermal equilibrium is typically achieved within a minute. Above 20 K a delay in the order of an hour is necessary step due to the increasing heat capacity of the metallic STM parts. Installing

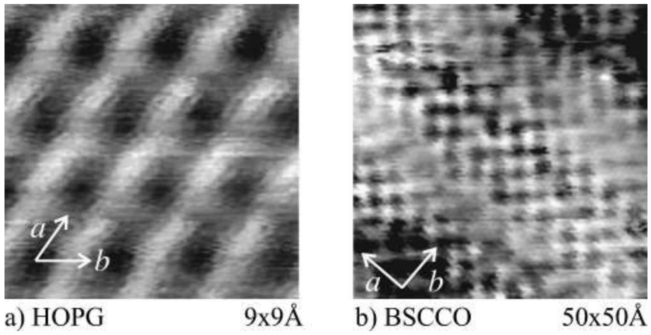

FIG. 2. Raw data topographic images obtained in constant current mode at 275 mK with electrochemically etched Ir tips: (a) *ex situ* cleaved HOPG with $R_t = 0.25\,\mathrm{G\Omega}$. The atomic corrugation is about 1.6 Å. (b) *in situ* cleaved BSCCO at $B = 6$ T with $R_t = 0.7\,\mathrm{G\Omega}$. The atomic corrugation is about 1.3 Å.

a heater directly on the STM could considerably shorten this delay.

Operating the STM generates no detectable heat dissipation, except when driving the inertial slider[18] to move the sample with respect to the tip. Applying a high ac voltage (typically 150 V_{pp} at 1500 Hz) to the piezotube, generates a heat leak in the order of 100 μW on the ^{3}He pot. At 275 mK it can lead to a peaked temperature rise of about 10 mK at the ^{3}He pot. Due to the high heat capacity of the ^{3}He pot at very low temperatures compared to that of metals, the base temperature is recovered within a minute after stopping the slider.

IV. UHV AND MANIPULATION

Ultrahigh vacuum is achieved by a 300 ℓ/s ion pump and by the cryopumping of the large cryostat surfaces extending into the UHV space (Fig. 1). One reaches a cryogenic vacuum of 10^{-11} mbar at the low temperature sample location. To access the STM inside the UHV space, an UHV platform for *in situ* manipulations was designed. It allows the removal of the 1 K shield around the STM, to transfer tips and samples to the STM, and to cleave the samples at room temperature. In order to control the spot where the tip is going to tunnel on the sample, a stepping motor[22] driven mechanism performs the sample transfer with the possibility to locate precisely (2.5 μm/step) the sample with respect to the tip along the transfer axis. Displacements can be controlled with submicronic resolution using a scanning electron microscope (SEM). As the STM scan size at 300 mK is about 4 μm, we can access any surface structure along the transfer axis.

V. SYSTEM PERFORMANCES

To prove the system stability and to demonstrate that high quality tunneling spectroscopy is achieved at very low temperatures in UHV, we present familiar results obtained on highly oriented pyrolitic graphite (HOPG) and on two different high-T_c superconductors.

Preliminary tests were performed on HOPG at both room temperature and very low temperatures, to achieve a proper calibration of the microscope. All data shown here were taken with electrochemically etched Ir tips mounted perpendicular to the ab plane of the sample. In Fig. 2(a), we show a raw data topographic image of HOPG taken at 275 mK at constant current. The hexagonal carbon β-site lattice ($a_0 = 2.46$ Å) is clearly resolved. We further obtained atomic resolution on an *in situ* cleaved $Bi_2Sr_2CaCu_2O_{8+\delta}$ single crystal at 275 mK in a field of 6 T [Fig. 2(b)]. It is relevant to point out that a true UHV environment is necessary to obtain this result. The ab plane atomic lattice ($a_0 \approx b_0 = 5.4$ Å) and the modulation along the b axis (≈ 26 Å) of the BiO plane are distinctly observable.

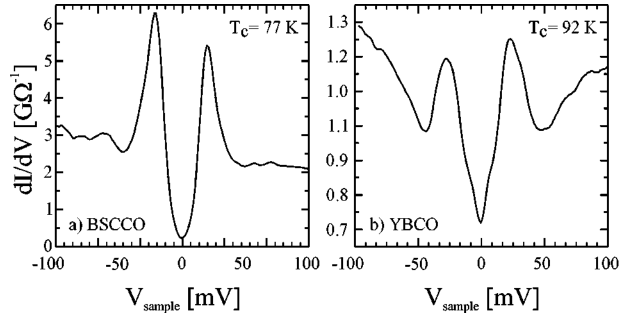

FIG. 3. Differential tunnel conductance spectra obtained at 275 mK in zero field using a lock-in technique and electrochemically etched Ir tips: (a) *in situ* cleaved BSCCO with $R_t = 0.5\,\mathrm{G\Omega}$; (b) YBCO with $R_t = 0.7\,\mathrm{G\Omega}$.

We further demonstrate the ability to perform stable spectroscopy on complex materials at very low temperatures in UHV, by showing reproducible differential tunnel conductance spectra taken at 275 mK on an $YBa_2Cu_3O_{7-\delta}$ [YBCO, Fig. 3(b)] and on an *in situ* cleaved $Bi_2Sr_2CaCu_2O_{8+\delta}$ [BSCCO, Fig. 3(a)] single crystal. The measured differential tunnel conductance, which is proportional to the local density of states (LDOS),[3] clearly shows the well-known superconducting gap. The large and controlled temperature range together with the magnetic field will be essential to investigate with more details the temperature and field dependence of the LDOS of high-T_c superconductors, especially in the intermediate state.

In summary, we have presented a unique STM setup, which extends conventional spectroscopic studies of superconductors to sub-Kelvin temperatures and high magnetic fields in a true UHV environment, thus giving the exciting opportunity to explore superconductivity and other low energy electronic phenomena in a large range of the H-T phase diagram, which so far has barely been studied by this technique.

ACKNOWLEDGMENTS

The authors thank J.-G. Bosch and the machine shop for their help in designing and manufacturing the STM and the UHV platform. We also warmly thank B. Revaz and A. Erb for supplying excellent BSCCO and YBCO single crystals. Financial support from the Swiss National Fund for Scientific Research is gratefully acknowledged.

[1] G. Binning and H. Rohrer, Helv. Phys. Acta **55**, 726 (1982).
[2] For an introduction see, *Scanning Tunneling Microscopy I-III*, Springer Series in Surface Science, edited by H.-J. Güntherodt and R. Wiesendanger (Springer, New York, 1992).
[3] J. Tersoff and D. R. Hammann, Phys. Rev. B **31**, 805 (1985).

[4] H. F. Hess, R. B. Robinson, and J. V. Waszczak, Phys. Rev. Lett. **64**, 2711 (1990).
[5] I. Maggio-Aprile, Ch. Renner, A. Erb, E. Walker, and Ø. Fischer, Phys. Rev. Lett. **75**, 2754 (1995).
[6] S. H. Pan, E. W. Hudson, A. J. Gupta, K.-W. Ng, and J. C. Davis (unpublished).
[7] Ch. Renner, B. Revaz, J.-Y. Genoud, K. Kadowaki, and Ø. Fischer, Phys. Rev. Lett. **80**, 149 (1998).
[8] A. Matsuda, S. Sugita, and T. Watanabe, Phys. Rev. B **60**, 1377 (1999).
[9] Y. M. De Wilde, M. Ivarone, U. Welp, V. Metlushko, A. E. Koshelev, I. Aranson, G. W. Crabtree, and P. C. Canfield, Phys. Rev. Lett. **78**, 4273 (1997).
[10] Ch. Renner, B. Revaz, K. Kadowaki, I. Maggio-Aprile, and Ø. Fischer, Phys. Rev. Lett. **80**, 3606 (1998).
[11] A. M. Troyanovski, J. Aarts, and P. H. Kes, Nature (London) **399**, 665 (1999).
[12] H. Fukuyama, H. Tan, T. Handa, T. Kumakura, and M. Morishita, Czech. J. Phys. **46**, Suppl. S5 2847 (1996).
[13] S. H. Pan, E. W. Hudson, and J. C. Davis, Rev. Sci. Instrum. **70**, 1459 (1999).
[14] For recent results and references therein, T. M. Riseman, P. G. Kealey, E. M. Forgan, A. P. Mackenzie, L. M. Galvin, A. W. Tyler, S. L. Lee, C. Ager, D. Mc Paul, C. M. Aegerter, R. Cubitt, Z. Q. Mao, T. Akima, and Y. Maeno, Nature (London) **396**, 242 (1998).
[15] For a review about magnetic superconductors, M. B. Maple, Physica B **215**, 110 (1995).
[16] H. W. Meul, C. Rossel, M. Decroux, Ø. Fischer, G. Remenyi, and A. Briggs, Phys. Rev. Lett. **53**, 497 (1984), see also Ref. 15.
[17] Ch. Renner, Ph. Niedermann, A. D. Kent, and Ø. Fischer, J. Vac. Sci. Technol. A **8**, 330 (1990).
[18] Ch. Renner, Ph. Niedermann, A. D. Kent, and Ø. Fischer, Rev. Sci. Instrum. **61**, 965 (1990).
[19] M. Kugler, Ch. Renner, V. Mikheev, G. Batey, and Ø. Fischer, Physica B (to be published).
[20] U.S. No. Patent 5829270, Heliox^{UHV} Product Information, Oxford Instruments.
[21] Highly thermal conducting materials, Caro-Bronze alloy (91.7% Cu, 8% Sn, 0.3% P), Walter Looser AG, CH-8031 Zurich; alumina ceramics (Degussit), Friedrichsfeld GmbH, D-6800 Mannheim; Thermally and/or electrically conductive UHV epoxy glues EPO-TEK 417/H70E, Polyscience AG, CH-6330 Cham.
[22] UHV stepping motors, Arun Microelectronics Ltd., Arundel, West Sussex, UK.