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SCIENCE DIRECT
Physica B 329–333 (2003) 1653–1655
PHYSICA B
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Construction of an ultra low temperature STM with a bottom loading mechanism

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Abstract

We have constructed and tested an ultra-low temperature scanning tunneling microscope that works with an atomic resolution at ultra low temperatures ($T \geqslant 126$ mK) in magnetic fields ($B \leqslant 6$ T) in ultra high vacuum (UHV). Clean sample surfaces can be prepared by several different methods and characterized by low energy electron diffraction in situ of UHV. A unique bottom loading mechanism enables us to cool back to the base temperature within 3 h after changing the sample and scanning tunneling microscopy tips.

Keywords: Low temperature STM

The scanning tunneling microscopy and spectroscopy (STM/STS) technique has recently become a powerful tool for low temperature experiments. This is because, with this technique, we can study a variety of low temperature phenomena ranging from adsorbed two-dimensional solids to low T_c superconductors with extraordinary spatial resolutions.

Here we report the construction and test results of the first STM designed to work with an atomic resolution at ultra low temperatures (ULT) below 200 mK in magnetic fields up to 6 T. It has also capabilities of sample preparation and characterization in situ of UHV. After transferring the sample and tip to the STM head, it takes only 3 h to cool back to the base temperature. All these features make this new ULT-STM more versatile than previously constructed ones [1–3].

Since the designing of our ULT-STM has been already reported elsewhere [4] rather in detail, we will describe here mainly what have been changed from the original design.

We have replaced the long and rather thin (380 mm long and 20 mm in diameter) copper rod, which thermally and mechanically connects the STM head to the mixing chamber (MC) of dilution refrigerator (DR) (see Fig. 2 in Ref. [4]), with a more rigid copper cage with three support rods (200 mm long and 8 mm in diameter). It was necessary to do so in order to improve mechanical rigidity of the system. Otherwise, the atomic corrugation has not been observed because of large lateral vibrations of 0.6 μm amplitude at 5 Hz at the end of the thermal link.

The thermal link to cool the sample and tip below 200 mK is based on three sintered silver-powder heat exchangers (4 m^2 surface area) packed in an epoxy capsule which is filled with liquid ^{3}He (0.5 cm^3). This configuration was adopted to keep better thermal contact and electrical isolation between the sample, tip and MC. Each heat exchanger is sintered onto a silver wire (1 mm in diameter) and connected either to the MC, sample or tip. In order to improve the high temperature performance, a 25 μm thick Mylar sheet was inserted between the silver wire and MC. The sample temperature is monitored with a RuO_2 resistance thermometer attached to the silver wire, which connects the sample to one of the heat exchangers.

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0921-4526/03/$ - see front matter
doi:10.1016/S0921-4526(02)02446-8

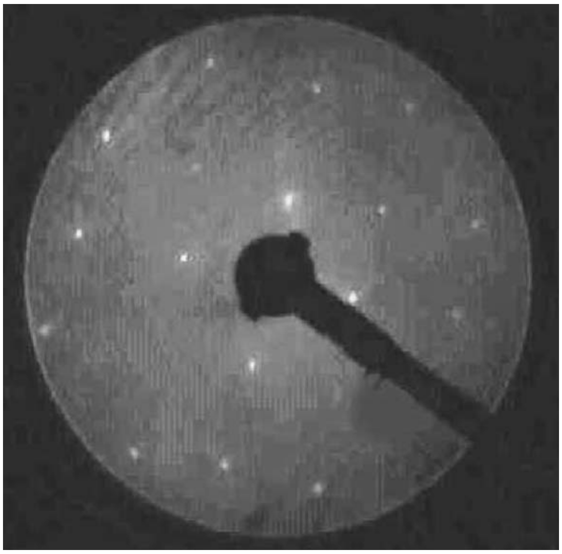

Fig. 1. The LEED pattern measured at room temperature with an electron energy of 193 eV for a Sr_2RuO_4 sample cleaved at $T = 8$ K.

The bottom loading mechanism for the sample and tip minimizes the access length from the UHV chamber to the STM head. We used a commercial DR[1] with a dewar which has demountable tails. The STM head is made of silver based alloys which are non-magnetic and have small nuclear-spin heat capacities at ULT and in high fields.

We have tested the cleavage mechanism, LEED characterization, precooling and sample/tip exchange mechanism using a Sr_2RuO_4 single crystal. All these procedures have been made in UHV environment. Fig. 1 is a LEED pattern of the cleaved sample surface. It clearly shows the four-fold symmetry of the SrO plane indicating the successful cleavage [5]. The sample and tip were precooled by liquid nitrogen and helium flow down to 100 and 7 K, respectively, within 40 min (see Fig. 2(a)). They were then transferred to the STM head (see the arrows v and vi in Fig. 2(b)). The STM head warmed up to 8 K temporarily by this transfer as well as by accompanying removal and installation of the 80 and 4 K radiation baffles. However, in 20 min, it cooled down to 2 K due to a large heat capacity of ^{3}He–^{4}He liquid mixture in the DR. During this exchange procedure, the ^{3}He circulation of DR was stopped, while the 1 K pot was kept running. It takes only 2 h for the MC to cool back to the base temperature (38 mK) after restarting the ^{3}He circulation.

Fig. 3 shows an STM image of 2H-$NbSe_2$ transferred to the STM head in the same manner as described above. This image was taken at 126 mK, the lowest sample temperature achieved in the present first cooling run. Individual Se atoms are visible as well as the 3×3 superlattice of the charge density waves (CDW) formation ($T_{CDW} = 33$ K). The measured temperature difference between the MC and sample indicates a large heat leak to the STM head of 20 μW at most. We believe that a major source of the heat leak is radiation from the 4 K baffle whose temperature is probably much higher than 4 K because of insufficient radiation shielding at the

[1] Oxford Instruments, model Kelvinox-100.

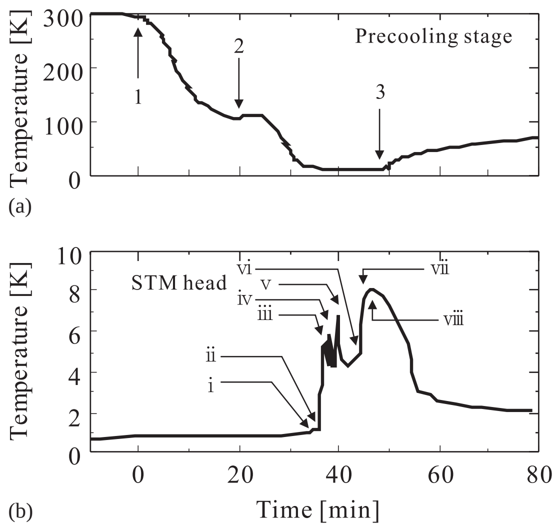

Fig. 2. (a) The time evolution of temperature of the precooling stage. The arrows indicate when we started precooling of the stage with liquid N_2 (1) and He flow (2) and when we stopped the He flow (3). (b) The time evolution of temperature of the STM head during sample/tip exchange. The arrows indicate when we removed 80 K (i) and 4 K radiation baffles (ii), sample (iii) and tip (iv), and when we installed new tip (v), new sample (vi) and the 4 K (vii) and the 80 K (viii) baffles.

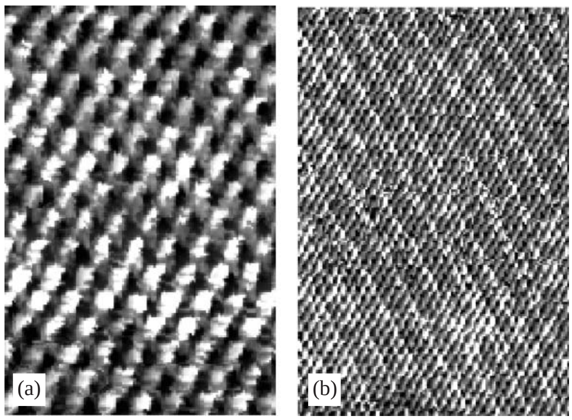

Fig. 3. The STM image of 2H-$NbSe_2$ taken at $T = 126$ mK with a PtIr tip (constant height mode; $I = 40$ pA, $V = -80$ mV, (a) 2.7×4.1 nm^2, (b) 9.0×13.6 nm^2).

dewar tail. This problem will be fixed in the next cooling run.

We thank Yoshi Maeno for providing us a Sr_2RuO_4 single crystal. This work was supported by Grants-in-Aid for Scientific Research from MEXT, Japan and ERATO project of JST.

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