Construction of a low-temperature STM with in situ sample cleavage

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1. Introduction

Operation of an STM at liquid-helium temperature allows topographic and spectroscopic measurements with little thermal noise as well as access to various phenomena which take place only at low temperatures. An important point to realize true vacuum tunneling is preparation of clean tip and sample surfaces. The presence of any adsorbates (perhaps except liquid helium) on either side is fatal because they are too stiff to sweep out with the tip and too thick to mediate a tunneling current. A sophisticated solution is to make both the sample-preparation part and the low-temperature STM part UHV compatible and then transfer the samples to the STM after they are prepared at room temperature [1,2]. Another solution is to clean the tip apex and cleave the sample after the entire system is cooled down to low temperature. In some applications the latter method is rather more favorable than cleaning at room temperature. For example, the high mobility of oxygen in high-$T_c$ superconductors at room temperature may make the surface deficient of oxygen before the sample is cooled down. In addition, the cold environment serves as a good cryogenic pump, and keeps a clean sample surface without complicated UHV equipment.

We constructed a low-temperature STM which fits within a 30 mm diameter space and is capable of sample cleavage at low temperature. The idea of the design is described in the next section. Details of mechanical design and operation are described in section 3. Installation and performance are described in section 4.

2. Design idea

The microscope head is designed based on the combination of a flip-manipulator-type sample holder [3,4] and a spring-loaded three-ball structure [5–8].

In the former structure, when the sample is apart from the tip, the sample holder assembly pivots about a catch and flips; then the sample surface is prepared apart from the tip. When the sample approaches the tip, a pick located close to the tip apex first touches the sample. From there the sample holder pivots about the pick; then vertical motion of the driving screw is reduced at the tip-and-sample gap by a factor $\sim 100$. A
drawback of this design is that the stress by the pick can damage the sample surface.

In the latter structure the sample holder assembly is sustained against the main body with a scanning tip using three balls and loading springs. The apex of the tip is located almost along the line drawn between two of the centers of the balls. While the third ball sitting on a lead screw moves vertically, the sample holder assembly pivots about the line. The motion of the lead screw is reduced by the lever mechanism at the tip-and-sample gap. This simple design allows reliable fine approach without damaging the sample, and is successfully adopted in many commercially available STM’s. We add to this structure a mechanism to flip the sample holder when the sample is apart from the tip, as in the flip-manipulator-type design. Once a large motion of sample holder assembly is obtained, it is easy to add a mechanism to cleave the sample.

The loading springs make the structure rigid and increase the resonant frequency of the microscope. However, if they are connected to fixed points on the main body as well as the sample holder, their tension will make it hard to flip the sample holder. To avoid the difficulty we added on the main body side a moving stage and hooked up the springs to it. The stage moves faster than the driving screw and releases the tension during the flip of the sample holder.

3. Design details

A cross section of the microscope in the initial state is schematically shown in fig. 1. Pictures of the corresponding state from different directions are shown in fig. 2. The microscope is designed to fit in a cylindrical space of 30 mm in diameter. At the bottom of the main body a couple of ruby balls are embedded. The height of their centers is adjusted to the bottom plane of the main body. The tubular PZT scanner, 9 mm in diameter, 27 mm long and 0.5 mm thick, is fixed to a cylindrical scanner base via a MACOR washer. The other end of the scanner is capped with a tip collar, where a tip holder with 1.4 mm diameter thread is screwed down. Whenever the tip is replaced its apex has to be adjusted to the bottom plane of the main body and ~ 0.5 mm inside of the line drawn between the centers of the ruby balls. To adjust the tip position the scanner base is relocated and then clamped to the main body. The scanner base also holds terminals of high-voltage PZT wirings.

Fig. 1. Cross section of the microscope, schematically drawn.
On top of the lead screw, 4 mm diameter and 0.25 mm pitch, is the third 3 mm diameter ruby ball. Rotation of the driving rod at the center of the microscope is geared down to the lead screw by a ratio of 5/12. Because the lever composed of the three balls reduces the motion by a ratio of 35, the rate of fine sample approach is 3 μm per rotation of the driving rod. The upper part of the lead screw has a left-handed screw, 4 mm diameter and 0.75 mm pitch. The counter-screw drives a moving stage vertically, 4 times faster than the ruby ball. Two loading springs are hooked up to the moving stage and pull up the sample holder against the main body.

On the sample holder V-shaped grooves run T-wise, which uniquely determine the horizontal alignment of the tip and sample. The sample is sandwiched with PTFE sheets and clamped to the sample holder. A ruby prism is also fixed to the sample holder, an edge of which is adjusted to attach to the side surface of the sample. A pair of guide rails run on both sides of the fixed prism on the sample holder. The running-blade assembly glides along the rails and cleaves the sample. The position of the running blade along the rail is determined by link with a swing arm, which rotates about a pivot located on the main body inside of the fixed ruby balls.

At the bottom of the main body a catch bar is located farthest from the tip. When the sample is away from the tip, the hook on the sample holder meets the catch bar; then the sample holder pivots about the bar. This rotation causes motion of the swing arm, hence the running blade runs across the sample and cleaves it. Development of the coarse-approach process is schematically shown in fig. 3. Pictures of the corresponding states are shown in fig. 4. The flip motion is finished when the paired ruby balls meet the V-grooves on the sample holder; thereafter the

Fig. 2. Photograph of initial state corresponding to fig. 1, viewed from different directions.
fine sample approach is performed. Here, both the swing arm and the running blade are folded away from the tip.

4. Installation and performance

The microscope is installed in a indium-sealed can of a cryostat, 38 mm in diameter and 300 mm long, similar to the one used in our previous work [9]. After the tip and sample are mounted, the can is sealed, evacuated down to $\sim 2 \times 10^{-7}$ Torr, $\sim 10$ mTorr of helium gas for heat exchange is introduced, then dipped into liquid helium. In order to avoid sticking of any movable parts at low temperature dozens of 1 mm diameter ruby balls are used as ball bearings, link pivots, or low-friction point contacts. Threads on the lead screw are lubricated with dry molybdenite powder.

Together with replacement of the micrometer head a few points are improved: Especially, Cu-Ni-based low-noise cables are employed for tip/sample bias and tunneling current detection.

As a result the performance of our STM is considerably improved. The rigid structure with loading springs made the microscope insensitive to external vibrations. Use of the new cable made the measurement durable against acoustic noise. An atomic image of graphite is reproducibly obtained at 4.2 K even without the air damper out of the helium vessel.

A major limitation of the present design is its range of fine approach. Corresponding to the vertical motion of the lead screw $\sim 4$ mm, the range is $\sim 110 \mu$m, i.e., the height of the sample

Fig. 3. Development of sample cleavage / fine approach, schematically drawn.
surface after cleavage must fall within this narrow range. When the sample is a layered material which is thin from the beginning, the condition is not severe. Otherwise a tedious trial-and-error is required until an acceptable cleavage is achieved.

In conclusion, we have constructed a low-temperature STM which is capable of in situ sample cleavage. Application to various low-temperature phenomena is to be reported separately.

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References