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A compact low temperature scanning tunneling microscope

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Abstract. We describe the design and fabrication of a compact Low Temperature Scanning Tunneling Microscope (LT-STM) together with a dipper cryostat for cooling the STM down to liquid helium temperatures. The STM, based on the piezo-tube walker as coarse approach mechanism, is suspended inside a cryostat vacuum can using three soft helical springs. The can is dipped into a liquid helium storage container for cooling the STM. Its compact size makes it less susceptible to mechanical vibrations and so the STM works with atomic resolution with a simple spring suspension. We demonstrate the performance of this STM for atomic resolution imaging and tunneling spectroscopy by observing the $3 \times 3$ charge modulation and the energy gap in the Charge Density Wave (CDW) phase of 2H-NbSe$_2$ at liquid helium temperatures.

1. Introduction
Ever since its invention by Binnig and Rohrer [1], the STM has advanced into a powerful tool for probing the exotic electronic properties of various conductors together with their surface topography. In particular, it has revealed some very interesting physics in several strongly correlated electron systems such as cuprates [2] and manganites [3] where electronic inhomogeneities seem to play a critical role. STM is the most suitable tool to probe such inhomogeneities down to atomic scales. While a sophisticated low temperature UHV-STM system can be used for such studies with in situ sample cleaving and preparation facilities, such systems tend to get bulky, complicated, and expensive. However, a robust, sensitive and high vacuum LT-STM is essential to access the phases of interest and to reduce the thermal noise for improved spectroscopic energy resolution.

The essential components of an STM are its coarse-approach mechanism, the fine xyz-scanner, the tip and sample holder, and the control electronics with software. Fairly advanced electronics and softwares for STMs are available commercially and the design and fabrication of the components other than the coarse approach is quite straightforward. However, getting a reliable coarse approach in a LT-STM is the most difficult part. The coarse approach also dictates the size of the STM and hence its sensitivity to external mechanical vibrations. The most common coarse approach for ambient STMs is micrometer screw driven by a stepper motor, however, the same cannot be used for LT-STMs. A number of LT-STMs [4, 5, 6] have been reported in the literature with a coarse approach based on piezoelectric elements. In particular, majority of these designs are based on the inertial sliding mechanism [4] and some are stick-slip [5] or grab-and-pull [6] type. Our motivation was to make a simple and user-friendly LT-STM.
system that can be cooled down to liquid helium temperatures by using a liquid helium storage-cum-transportation vessel. We have set-up a compact and user-friendly low temperature STM system using a stick-slip like coarse-approach called piezo-tube walker [7]. In the earlier work [7] one of the authors has described the working mechanism of piezo-tube walker and used it in the ambient STM. In this paper we have shown that the same coarse approach mechanism can also work in the low temperature STM with a proper vibration isolation. Performance of this type of LT-STM is apparent from our results.

2. Experimental setup

The internal diameter of the neck of the liquid helium container that we want to use for cooling this STM is 50mm. To avoid sample contamination and freezing of the coarse approach, we need to keep the STM in high vacuum while cooling. Thus, out of this 50mm diameter space the vacuum can and the vacuum space would occupy about 20mm. Thus the STM has to be small enough to fit inside a circular hole of diameter 30mm. The design of the STM that we use here is similar to the one described in an earlier paper [7] except for a few changes in terms of further size reduction and the material selection for low temperature compatibility.

The walker cavity in this STM is made of stainless steel (SS) but for size reduction, the bottom part of the cavity is one single piece as opposed to the earlier design [7] that used two pieces. Since we cannot access the internal faces of the cavity for polishing anymore, we fix thin polished alumina strips on the cavity walls. So the frictional contact is now between these alumina strips and sapphire discs fixed to the tube. Also, instead of long helical springs for putting the cavity together, we use much smaller copper-beryllium finger springs for further size reduction (shown in index 8 of Fig.1a). The xyz-scanner tube [9] of outer diameter 6.35mm and wall thickness 0.508mm is coaxially fixed on the inside of the walker tube using a spacer. A ground shielded tip holder is mounted at the outer end of the scanner tube while the sample-holder is placed at the end of the cavity facing the tip. This STM works in any orientation from horizontal to vertical. To minimize the threshold voltage for walker motion and the forward-backward asymmetry the sliding force has to be appropriately adjusted. The overall size of this STM is significantly smaller than the original one so that it fits inside a cylindrical hole of 30mm internal diameter and 45mm length. The walker electronics is home built and uses a micro-controller that controls six optical switches. 5ms after zero crossing detection of the 50Hz line sine-wave, the switches are turned on one by one at 100µs interval near the peak of the sine-wave. The six voltages then decay to zero with the 50Hz sine-wave. The output voltage polarity as well as its amplitude are adjustable between 45V and 300V.
This STM is suspended from a circular brass flange, which is a part of a dipper cryostat (Fig. 1b), using three soft springs (Fig. 1a). Combined with the STM mass this spring mass system has a natural frequency of less than 10 Hz. The compact STM itself has quite a high natural frequency (>5 kHz) and so this spring mass system easily filters out the high frequency noise to which the STM is sensitive. The dipper cryostat (Fig. 1b) consists of a 12.7 mm O.D. stainless steel tubing with a homemade vacuum electrical feed-through on top after a TEE to provide a pumping port. The central part of the SS tubing passes through a vacuum sealed compression port welded on a brass flange that seals on top of the liquid helium storage container. The lower end of this tubing ends into a brass flange that seals on a brass can using indium wire as vacuum seal. Inside the vacuum can a thick wall copper tubing is fitted into the SS tubing. On the free end of this copper tubing (see Fig. 1a) we mount another brass flange on which the STM is suspended. This copper tubing helps in bringing a decent cooling power to this brass flange and we provide a heat link between this flange and the STM using soft copper wires. This is required, as we do not put any helium exchange gas to avoid the sample surface contamination and freezing of walker. In fact we are putting a small amount of activated charcoal in the can to get rid of any residual gases at low temperatures.

We use phosphor-bronze wires inside the SS tubing for making connections from the top flange to the STM except for the sensitive tunneling current and bias voltage connections for which we are using thin stainless steel coaxial wires. The wires near the STM are softly coupled to keep the vibration isolation effective. The tunneling current preamplifier is placed just outside the hermetic feedthrough in ambient. Other than the homemade walker electronics, the rest of the STM control electronics and software are commercial. A TTL pulse is used from this control electronics to drive the walker for automatic tip approach. The STM tips are made using either tungsten [10] or platinum(0.8)/iridium(0.2) [11] wire of 0.25 mm diameter using electrochemical etching. The samples are fixed on the sample holder screw using a conducting epoxy.

3. Test and Operation
The STM was first tested in ambient conditions after suspending it vertically from the cryostat for atomic resolution on HOPG. As shown in Fig. 2a, we can clearly resolve the three sites (in the line-cut) on HOPG arising from the AB type stacking of the hexagonal graphene layers [12]. Using this image the STM was calibrated for its x-y motion and z-calibration was done using a Michelson interferometer and He-Ne Laser by observing the fringe shift. At room temperature the maximum x-y scan area is 3.5 µm × 3.5 µm and we can move a maximum of 2 µm in z-direction with the present control electronics. After sealing the cryostat can and pumping it to a vacuum of <0.05 mbar, we insert the cryostat into liquid nitrogen container. With the copper wire heat link, we can reach a temperature of 80 K in less than 15 hrs. Due to differential thermal contraction of various STM parts with cooling there is a net drift of the tip towards the sample. Due to this drift we cannot do any imaging during the cooling. At a temperature of 200 K the z-drift is of the order of 0.1 µm/K and the sign of the drift reverses around 120 K. We imaged HOPG and some other layered compounds at liquid nitrogen temperatures. Since the d31 parameter of the piezo-tube is temperature dependent, we can scan a maximum area of 1.3 µm × 1.3 µm and for the same reason, the piezo-tube walker’s threshold voltage also rises. At room temperature the walker works at 45 V step voltage while at 80 K we need 120 V step and at liquid helium temperatures it is 240 V.

After pre-cooling in liquid nitrogen we insert this cryostat into a liquid helium storage container and in less than 15 hrs we reach a stable temperature of 8 K. During this temperature change the tip drifts away from the sample. We have imaged HOPG and 2H-NbSe2 at this temperature. The maximum scan area at 8 K is 0.6 µm × 0.6 µm. NbSe2 undergoes a CDW transition at 35 K and at 8 K we see the 3 × 3 CDW charge modulation as shown in Fig. 3a, which is more clearly visible in the Fourier transform in Fig. 3b. Tunneling spectroscopy was performed.
by recording the I-V spectra and numerically differentiating them we get the dI/dV-V spectra. This dI/dV at V is directly proportional to the local density of electron states at energy eV with respect to the Fermi energy. Therefore, we expect a corresponding depression in the dI/dV-V spectrum due to the CDW gap at the Fermi energy. This spectrum is shown in Fig.3c as well and we clearly see signatures of the CDW energy gap (\(2\Delta \approx 80\text{meV}\)) in the tunneling spectra. The magnitude of this gap value is consistent with the reported value in the literature [8].

4. Conclusions
In conclusion, we have designed and fabricated a LT-STM based on the piezo-tube walker together with a simple dipper cryostat for cooling it to liquid helium temperatures. We have tested this STM down to 8K in liquid helium for atomic resolution imaging and tunneling spectroscopy on HOPG and on CDW phase of 2H-NbSe\(_2\) single crystals at 8K.

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References
[1] Binnig G, Rohrer H, Gerber Ch, and Weibel E 1982 *Phys. Rev. Lett.* 49 31.
[2] Hanaguri T, Lupien C, Kohsaka Y, Lee D H, Azuma M, Takano M, Takagi H, Davis J C 2004 *Nature* 430 1001-5.
[3] Renner Ch, Aeppli G, Kim B G, Soh Y Ah, Cheong S W 2002 *Nature* 416, 518-21.
[4] Pohl D W 1987 *Rev. Sci. Instrum.* 58 54-7.
[5] Binnig G and Rohrer H 1982 *Helv. Phys. Acta* 55 726.
[6] Okumura A, Miyamura K and Gohshi Y 1988 *J. Microsc.* 152 631.
[7] Gupta A K and Ng K W 2001 *Rev. Sci. Instrum.* 72 3552.
[8] Wang C, Giambattista B, Slough C, Coleman R and Subramanian M 1990 *Phys. Rev.* B 42 8890.
[9] Binnig G and Smith D P E 1986 *Rev. Sci. Instrum.* 57 1688-89.
[10] Lindahl J, Takanen T and Montelius L 1998 *J. Vac. Sci. Technol.* B 16 3077.
[11] Oliva A I, Romero G A, Pena J L, Anguiano E, Aguilar M 1996 *Rev. Sci. Instrum.* 67, 1917-21.
[12] Park S I and Quate C F 1986 *Appl. Phys. Lett.* 48, 112.