Cryostat for *in situ* scanning tunneling microscopy studies of film growth at low temperatures

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This article describes a low temperature scanning tunneling microscope (STM) system which is designed to study film growth at very low substrate temperatures $(4\ \mathrm{K}<T_S<77\ \mathrm{K})$. A simple tripod design with the addition of a sample manipulator, is implemented as the STM head. In this system, a metal film can be thermally deposited on a conducting or an insulating substrate held at cryogenic temperatures and be probed *in situ* by STM. *In situ* and room temperature images of a Pb film grown on a 4 K substrate are presented. © *1997 American Institute of Physics.* [S0034-6748(97)01111-8]

I. INTRODUCTION

The scanning tunneling microscope (STM), developed by Binning *et al.*,[1] is capable of measuring surface structures with atomic resolution in real time. The determination of microscopic details on surfaces, made possible with the STM, has given researchers valuable insights into various surface science problems.

Numerous STM experiments have investigated the growth of thin films and revealed a great deal of information on the physical mechanisms involved. That work[2–4] has concentrated on growth regimes in which thermal adatom diffusion is rapid and structures form under near equilibrium with the vapor. We are interested in studying the structure of metal films grown on very cold substrates where adatom diffusion is absent. These films, which have been referred to as quench condensed, have been used in numerous studies of the effects of strong disorder on electronic transport.[5] Very little is known about their structure. Thermal annealing to temperatures as low as 20 K has been shown to change the structure of some of these films irreversibly. Thus a complete understanding of the growth mechanisms involved at low temperatures requires *in situ* studies.

A STM for studying film growth at low temperatures must allow the user to manipulate the sample stage at cryogenic temperatures to expose it to the thermal deposition source and to bring it within tunneling distance from the tip in a controlled manner. In these experiments, the substrate has to be strongly thermally anchored to the cryogen bath, since it has to be kept at low temperatures even during the film deposition. Most current STM systems with low temperature sample manipulation capabilities[6–8] use piezoelectric walkers for approach mechanisms. These designs rely on weak thermal links such as cold fingers[6,7] or exchange gas to cool their sample stages.[8]

In this article, we present a cryostat design where a thin film can be quench condensed on a substrate held at cryogenic temperatures $(4\ \mathrm{K}<T_S<77\ \mathrm{K})$ and probed *in situ* with a STM. To link the substrate strongly to the liquid He bath, we have implemented a mechanical approach mechanism in the STM head. We present topographs of quench-condensed lead films that illustrate the system performance.

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II. INSTRUMENT DESCRIPTION

The STM head shown in Fig. 1 is based on an ultrahigh vacuum (UHV) STM head design by Schmid and Kirschner.[9] Three stainless steel springs hold the sample plate in rigid contact with three fine pitched (1/4-80) screws with rolling ball tips[10] installed in a stainless steel block that houses the piezo tube scanner. The sample plate is made of stainless steel except for the area in direct contact with the substrate. This part is made of oxygen-free high-conductivity copper and is thermally anchored to the cryogen bath to ensure proper substrate cooling. The three screws are arranged at the corners of an equilateral triangle. The two screws near the front end are for coarse sample-to-tip approach and the screw at the rear serves as the fine-approach adjustment. This screw tilts the sample toward or away from the tip about the pivot line defined by the two coarse-approach screws. The line connecting the two front screws, the pivot line, is very close to the tip so that the mechanical advantage of this lever system reduces the motion of the tip by a factor of 10 relative to the fine-approach screw motion.

The STM head is rigidly fixed to a copper platform in a 4 in. diameter, 8 in. long stainless steel can at the end of a cryostat [Fig. 2(a)]. The platform hangs from three 1/2 in. diameter copper rods screwed into the 6 in. conflat flange

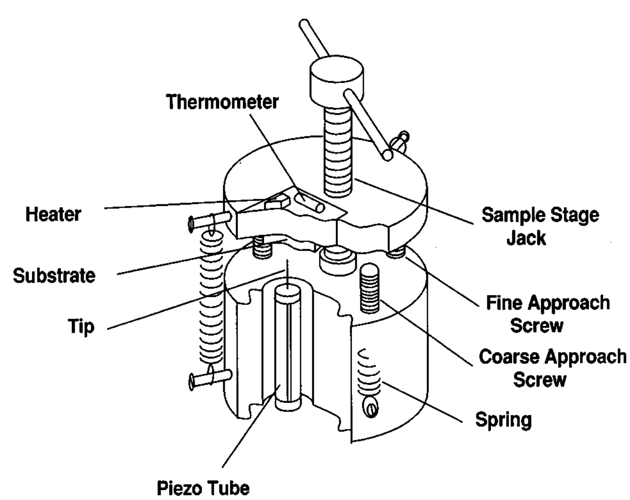

FIG. 1. Illustration of the STM head.

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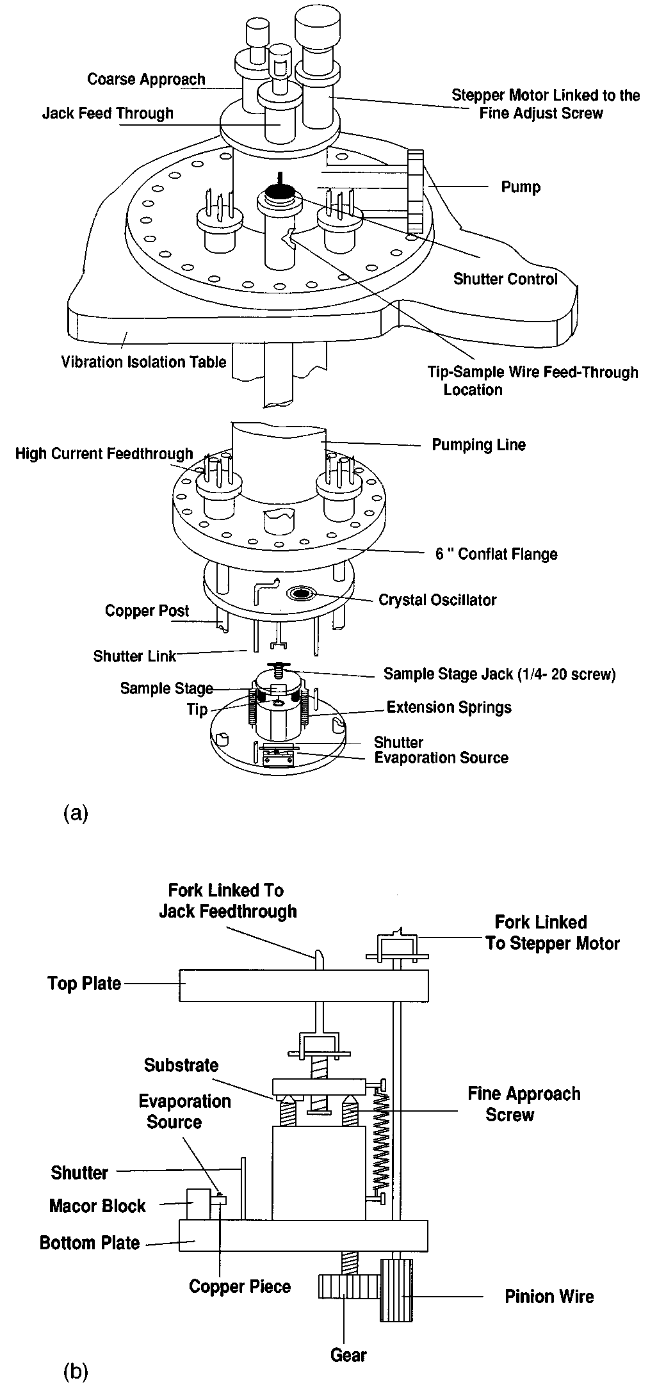

FIG. 2. (a) Illustration of the cryostat. (b) Close up showing the mechanical links to the approach screws.

that seals the can. A copper braid approximately 3 in. in diameter and 1 in. long connects the copper sample mount to a copper plate that is in direct contact with the cryogen bath via a solid copper rod. A 3 in. diameter stainless steel pumping line extends 34 in. from the 6 in. conflat flange to the room temperature portion of the cryostat. This line is used for evacuating the can. A second 1 in. diameter stainless tube runs parallel to the first for bringing wires to the quartz crystal microbalance and the STM. The whole cryostat sits on a commercial vibration-isolation system.[11]

The fine-approach screw is linked via gears, a fork, and an UHV feedthrough, to a stepper motor[12] that operates at room temperature [Fig. 2(b)]. Commercial software and electronics[13] generate the pulses to drive the motor. The minimum step of 1.8° corresponds to tilting the sample 60 nm towards the tip. A 1 in. PZT-5H[14] tube of 0.03 in. wall thickness and 0.25 in. diameter is implemented as a scanner which, with current electronics, has a scan range of more than 20 μm in the x and y directions and 2.5 μm in the z direction at 300 K. At 4.2 K the scan range and the z range are reduced to 4.5 $\mu\text{m}\times 4.5\ \mu\text{m}$ and 400 nm, respectively. One of the coarse-approach screws is also linked to a room temperature feedthrough in the same way as the fine-approach screw [not shown in Fig. 2(b) for clarity]. Adjustments in the fine-approach screw and this coarse-approach screw are used to translate the tip to different regions on the sample. These two screws tip the sample around different axes. With these adjustments, regions of a sample as far apart as 20 μm have been imaged.

Prior to closing and evacuating the can, the tip and the substrate are brought into tunneling distance (or very close proximity with the help of an optical microscope in the case of a nonconducting substrate) by adjusting the three screws. After sealing the can a 60 ℓ/s turbo pump evacuates it to pressures as low as 2×10^{-7} Torr at room temperature. The system is cooled down by immersing the can in liquid nitrogen or helium. The estimated pressures inside the can are less than 5×10^{-8} Torr and 1×10^{-9} Torr at 77 K and 4.2 K, respectively. Since the entire cryostat is immersed in the cryogen during an experiment, the walls of the cryostat provide effective cryopumping for the sample stage which stays warmer than the walls during the cooldown.

Once the desired temperature is reached, the sample plate may be lifted off the ball tips on the three approach screws by a jack screw in order to bring the substrate into thermal deposition position. This low pitched screw (1/4-20) (Figs. 1 and 2) passes through the center of the sample plate and is linked to a room temperature feedthrough that can withstand a considerable amount of torque. When engaged, the jack lifts the sample plate up against the pulling force of the springs. This upward motion of the sample stage ($\cong$0.5 in.) is enough to bring the sample into the line of sight of the evaporation source. Because of the springs, the upward movement of the plate is very reliable and reversible. After a deposition, the plate is lowered onto the three approach screws by unscrewing and disengaging the jack.

The thermal evaporation source sits 2.2 in. below the sample stage and 0.6 in. away from the STM body. It consists of two small copper blocks attached to a Macor block of dimensions 0.25 in.$\times$0.5 in.$\times$0.5 in. to electrically isolate them from each other and the rest of the cryostat. The copper pieces have tapped holes on them for attaching high current wires and the tungsten wire holding the premelted metal ball. The high current wires are run outside the cryostat and enter the cryostat through the high current feed throughs on the 6 in. conflat flange. Inside the cryostat, the wires are isolated from the rest of the system via ceramic beads. Up to two different sources can be implemented to make successive depositions of different materials. Depending on the melting point of the evaporated metal, currents of approximately

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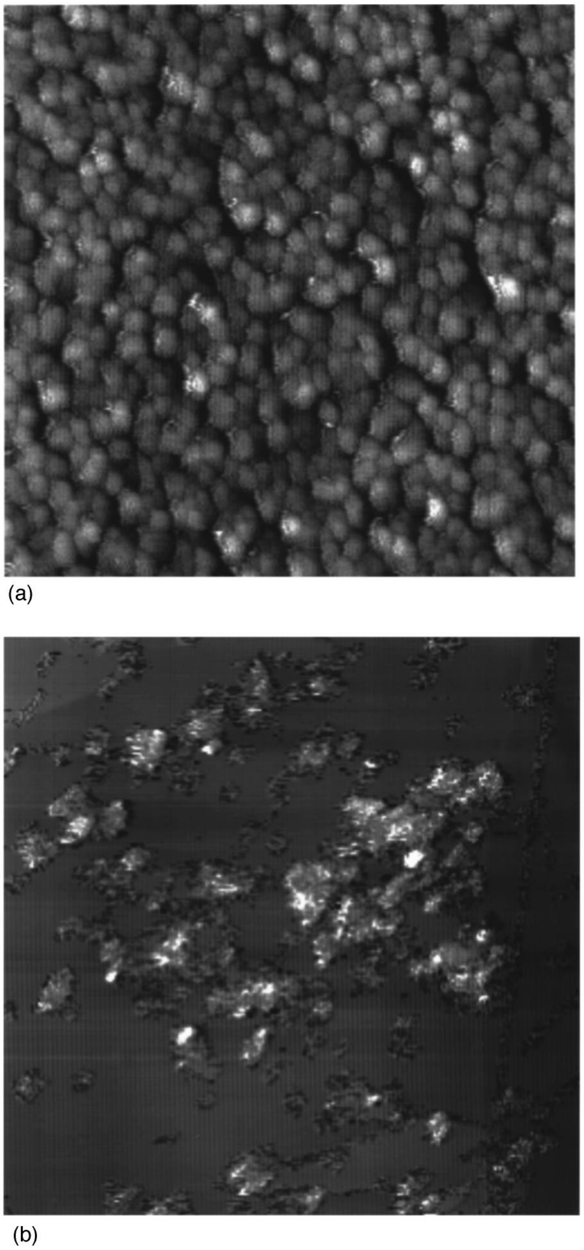

FIG. 3. (a) Topographic image of a 4.3 nm thick Pb film deposited on HOPG ($T_S < 12$ K during deposition). The image was taken at 4 K with $I_T = 10$ nA and $V_T = 0.25$ V. The scan area is 500 nm×500 nm. The height range is 2 nm from black to white. (b) The same film annealed to room temperature and imaged in high vacuum with $I_T = 9.4$ nA and $V_T = 0.59$ V. The scan area is 1000 nm×1000 nm. The height range is 4 nm.

5–12 A are run through the tungsten wire corresponding to an input power of 20–50 W. During evaporation, the incident particles hit the substrate surface at a slight angle (∼15°–20°) to the substrate normal. There is a room temperature linked shutter system which shields the sample from the evaporation source when closed. The shutter allows the evaporation source to be seen by the crystal microbalance at all times to ensure constant deposition rates.

III. OPERATION

With commercial electronics and software,[13] the STM system has been operated successfully at room temperature, 77 K, and 4 K. Terraces and monatomic steps have been observed on gold films deposited at room temperature.

With this system, we have investigated the morphology of metal films deposited on various substrates held at 4 K $< T_S <$ 77 K. The initial cool down to 4 K takes about 6 h. There is a moderate thermal drift of 50 μm in the tip sample distance during the initial cool down. To avoid tip crashes, the sample is backed 10 μm by the fine-approach screw before the sample stage jack is engaged for the deposition. Substrate heating of 5 to 15 K is observed during the depositions at 4 K depending on the melting point of the deposited material. After the film has been deposited, it is necessary to wait approximately 30 min for the system to reach thermal equilibrium. The turbo pump is turned off during the STM operation.

Figure 3(a) shows the structure of a thin quench-condensed Pb film on highly oriented pyrolytic graphite (HOPG) ($T_S < 12$ K during deposition), as imaged at 4 K. Annealing the film to 300 K [Fig. 3(b)] changes the morphology drastically.

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[12] The stepper motor and the driver card can be acquired from C&H Sales Company (P.O. Box 5356, Pasadena, CA 91117-9988) inexpensively.
[13] Park Scientific Instruments, 1171 Borregas Avenue, Sunnyvale, CA 94089.
[14] Staveley Sensors, 91 Prestige Park Circle, East Hartford, CT 06108.

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