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A scanning tunneling microscope with a wide sampling range

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Construction of a simple scanning tunneling microscope (STM) is described. This STM is suitable for atmospheric, controlled atmosphere, and high vacuum (but not UHV) work. This STM is especially well suited for determining surface topography on the 0.1 nm scale when images must be obtained over a wide sampling region (mm). Interchangeable piezo heads allow the STM to be used either for atomic resolution or for large (800×800 nm) area scans. Atomic resolution pictures of the graphite surface demonstrate that this design is suitable for use with structures smaller than 0.1 nm. An image of a thin film of Au, deposited on pyrex, is also presented.

I. INTRODUCTION

The scanning tunneling microscope (STM) was first demonstrated in 1982[1] and now serves as the focus for a rapidly growing group of surface sensitive techniques. The general design requirements and some recent STM implementations were recently reviewed.[2] Over the years, the trend has been to decrease the size and increase the rigidity of the microscope in order to minimize the amount of external vibration isolation required. Some very recent designs[3-5] have resulted in truly pocket-size microscopes requiring no special vibration isolation. In order to reduce the size of the STM, large *x-y* translational motions have been sacrificed. Thus, these very compact designs are poorly suited for applications requiring that images be taken at various sampling points distributed over a wide area.

Very recently, Stupian and Leung[6] described an STM that utilized a motorized micrometer to provide the coarse approach mechanism. This STM was small and rigid enough to require only minimal vibration isolation. Their implementation did not provide a means of wide *x* or *y* adjustment, and had the added disadvantage that the sample rotated in concert with the coarse advance motion. Their STM demonstrated that a motorized precision screw could be successfully coupled with a tube scanner to produce a useful STM.

In the present article, we describe the logical next generation device beyond the Stupian and Leung design. Our STM can produce good images of graphite on the atomic scale, and also of deposited films on a scale approaching micrometers. In comparison with Stupian and Leung's design,[6] ours is more compact, maintains a fixed orientation of the sample, and provides for several millimeters of travel parallel to the tip motion. Our primary use for this instrument is to study the morphology of deposited films as a function of deposition parameters. The uniformity of these films, over relatively large regions, is also an issue. Thus, we generally take pictures of the order of 10^4 nm^2 every several micrometers over a distance of a few millimeters.

II. DESCRIPTION

A. Mechanical design

Approach of the sample within tunneling distance of the tip is accomplished through the use of a commercial, miniature motorized translator.[7] This device is about the size of a deck of cards, has 0.5 in travel, and a step resolution of about 20 nm. It is basically a rectangular frame housing a dc motor stage driven on a precision lead screw. Applying 5 V dc produces rapid movement (about 1 mm/s) while 12 ms pulses of 5 V amplitude produce about 20 nm jogs. This stage also acts as the base for the STM. All mechanical parts are bolted to it. Figure 1 is a top view of the microscope.

To the outer part of the stage is bolted an L-shaped frame that houses the piezoelectric tube scanner, tip assembly, and most of the wiring. We actually have two of these frames. One is equipped with a 0.5×0.25×0.02 in. (wall) PZT-5A[8] tube used for fine work. The *z* displacement of this tube is calculated as 4.3 nm/V and the *x-y* displacement is determined to be 14.0 nm/V by calibration with graphite. The second frame is equipped with a 0.75×0.25×0.02 in.[3] (wall) PZT-5H[8] tube used for coarser work. The displacement per volt of this tube is a factor of 2.8 larger than the smaller tube. The outer electrodes were cut in the common four-quadrant fashion.[2-6] Each piezo was glued to a Macor block and the block was attached to the frame with screws. Electrical connection to the nickel coating on the piezoelectric tubes was made using Cerroceal No. 35 solder, a noncorrosive flux, and a soldering iron set at 330 °F. The quality of the solder joint is greatly improved by careful precleaning of the electrode surface with a pink rubber eraser. The Macor pieces were machined in such a way as to make the tip position relative to the motor stage independent of the piezo used. A grounded metal cylinder surrounds the piezo tube. Assembly details are shown schematically in Fig. 2.

The tip assembly comprises a Macor disk and a stainless-steel collar. The steel collar is drilled with a 0.225 through hole and is equipped with a 0-80 set screw. This collar is identified as B in Figs. 1 and 2. The tips used are 0.5 mm diameter, W or Pt/Rh wire. The tungsten tips are sharpened by electrochemical etching. The Pt/Rh tips are cut with dykes.

The sample holder assembly is mounted on the motor stage. This unit is designed to hold conventional SEM stubs. The stubs are held in place by a set screw. The vertical position of the front part of the sample assembly is controlled by a differential screw (E in Figs. 1 and 2). This screw is an 8-36/8-32 combination that provides an effective pitch of

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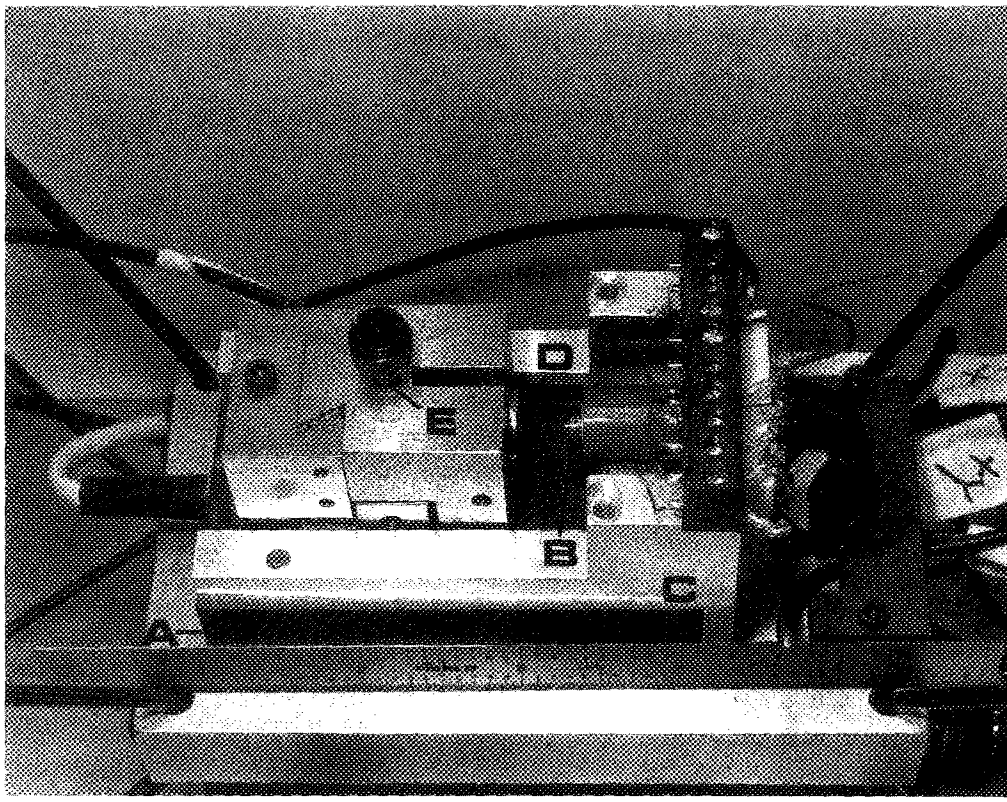

FIG. 1. Photograph of the STM. (A) identifies the motorized stage, (B) is the 0.5-mm-diam W tip and the tip holder, (C) is the L-shaped frame, (D) is the SEM stub sample mount, and (E) is the differential screw.

> 150/in. over a distance of about 3 mm.

The microscope sits on a mild steel plate suspended from four viton bands about 4 in. long. The viton bands are attached at the inner top of a mild steel box that encloses the STM and preamplifier. This box is 1 in. thick and provides excellent isolation at acoustic frequencies.[9] It can also be used to provide a controlled environment chamber for the STM. The STM and its enclosure are supported on a Newport laser table (4 ft×8 ft×8 in.) and three pneumatic legs providing both vertical and horizontal isolation.

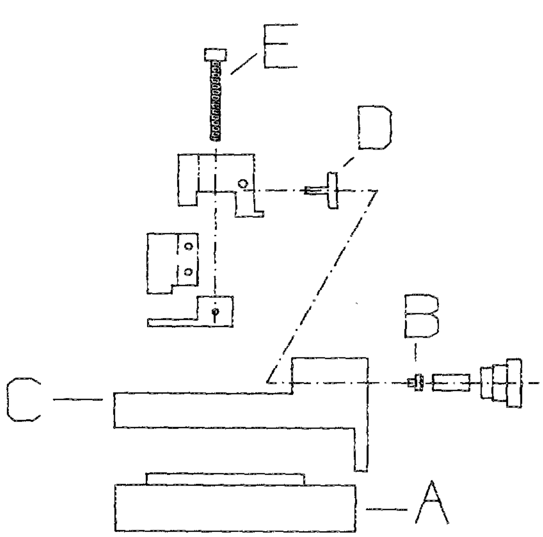

FIG. 2. Exploded view of basic features of STM construction. (A) is the motorized stage, (B) is the tip holder which is directly glued to the piezoelectric tube, (C) is the frame, (D) is an SEM stub used for sample mounting, and (E) is the differential screw of the Y-axis translation stage.

B. Electrical design

Figure 3 provides a schematic of the electrical connections used. Unlike Stupian's design, we rely entirely on the D/A converters to provide X, Y, and Z_c (coarse set) piezo drive voltages. The lines marked X_c,Y_c, and Z_c are controlled by ± 10 V 12-bit D/A converters. The X and Y lines are controlled by ± 2.5 V 12-bit D/A converters. These all reside on a Metrabyte DDA-06 interface card.[10] The DDA-06 is also used to produce 5 V pulses of variable duration. These pulses pass through a current amplifier and then drive the dc motor of the motorized stage, thereby effecting the coarse tip approach.

The X, Y, and Z voltages are fed into the four-quadrant piezo driver designed by Stupian and Leung (Fig 3 of Ref. 6). This driver provides $20\times Z_c + Z_f + X_c + X$, $20\times Z_c + Z_f - X_c - X$, $20\times Z_c + Z_f + Y_c + Y$, and $20\times Z_c + Z_f - Y_c - Y$ voltage to each of the four quadrants. The inner electrode is maintained near ground potential to assist in shielding the tip wire. Z_f is the error voltage, or feedback voltage, required to maintain a fixed surface to tip distance.

The tunneling current is amplified by a homemade two-stage amplifier based on the OPA111 opamp. Our preampli-

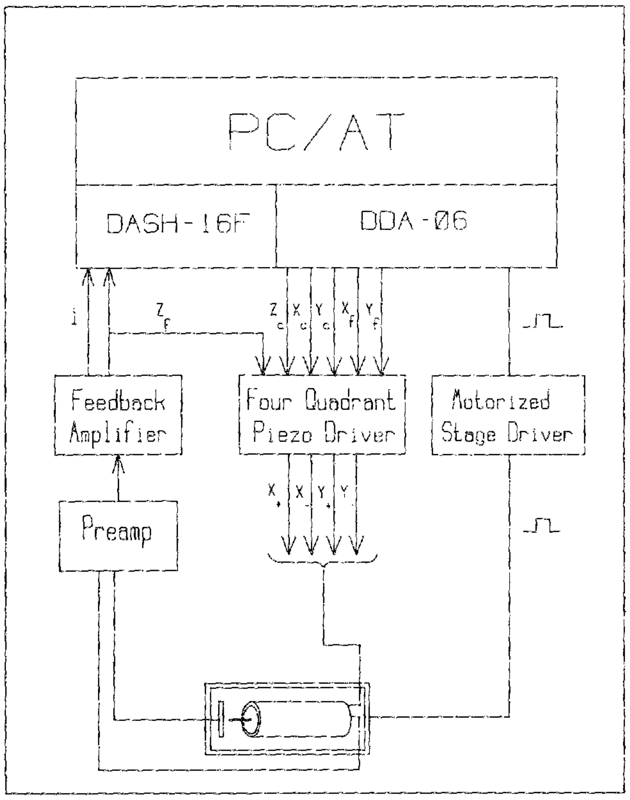

FIG. 3. Schematic of STM electrical system. The preamp and bias circuit are housed within an acoustic vibration isolation box which also houses the STM.

fier has a gain of 2×10^8 V/A and a bandwidth of 45 kHz. The preamplifier and bias voltage circuitry are contained in the steel acoustic shielding box which also provides useful electrical shielding. The bias and preamplifier are powered by two 9 V batteries.

The tip current signal for the preamplifier serves as input to the feedback amplifier. Our feedback amplifier is a copy of one designed for Paul Hansma's group at UC Santa Barbara. It behaves as a logarithmic amplifier for small signals but becomes linear for large error signals. The buffered current signal is monitored by one A/D channel of a Metrabyte DASH-16F high-speed interface board. The feedback voltage, Z_f, is also monitored by a second A/D channel of the DASH-16F.

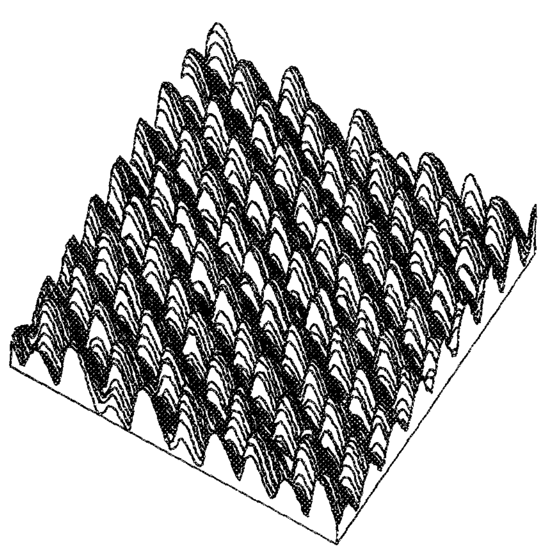

FIG. 4. 2.1 by 2.1 nm perspective view of the surface of monochromator grade graphite. The specimen was biased at -100 mV and the tip current was maintained at 2.0 nA.

C. Computer control

Data acquisition is controlled entirely by the computer through a mixed-language control program. All of the time, critical data taking and storage steps are written in assembly language. The main program is written in MICROSOFT QUICKBASIC and accesses the assembly routines through a QUICKLIBRARY. Thus, the convenience and power of basic programing need not cause a speed penalty. In order to stay within the 40 kHz bandwidth of our preamplifier, we typically introduce 40 to 200 NOP instructions per D/A step.

Data acquisition begins by first manually bringing the tip within about 0.02 mm of the sample. This is accomplished through the use of a manual pulse control on the motor stage control. The operator monitors the tip-sample distance using a small, 50-power pocket microscope (Edmunds). Once the sample and tip are close, programmed coarse approach is initiated. The voltage Z_c is ramped from 0 to -160 V in steps of 0.08 V. After each step, the tunneling current (i) line of the A/D converter is tested. If an operator-selected current level is present, all motion stops. If the set point is not reached, Z_c is again decreased. If Z_c reaches -160 V, Z_c is set to zero, a single 12 ms 5 V pulse is sent to the motor stage,and the sequence is reinitiated. This procedure gives a reliable and "crash free" approach of the sample to the tip. The operator may then select to increment or decrement Z_c in order to set the quiescent point of Z_f near 0 V.

In order to obtain a single STM scan, the operator selects a scan width, delay time, and data type desired. The latter choice selects whether Z_f (height at fixed current) or i (for fixed height scans) is to be stored as a function of x and y position. The A/D can be read from one to eight times for each point in order to reduce the reading error. Loop gain, low-pass and high-pass filters, and current set point are manually selected by potentiometers located on the feedback amplifier. Data are acquired in both directions, but saved as separately named files. Typical scan times range from 10 s to 6 min.

After the picture is stored, it is displayed as a seven-level color "bird's eye" view plot. This type of picture can be drawn very rapidly on our EGA display and provides a satisfactory indication of the quality of the picture. At this point, the operator may elect to save the data as is or to remove a least-squares plane prior to saving. The latter feature is especially useful for removing thermal drift from slow wide area scans.

Separate viewing and plotting programs have also been written. Data can be presented in x vs $y+z$ format, in colored overhead view format, or as three-dimensional projec-

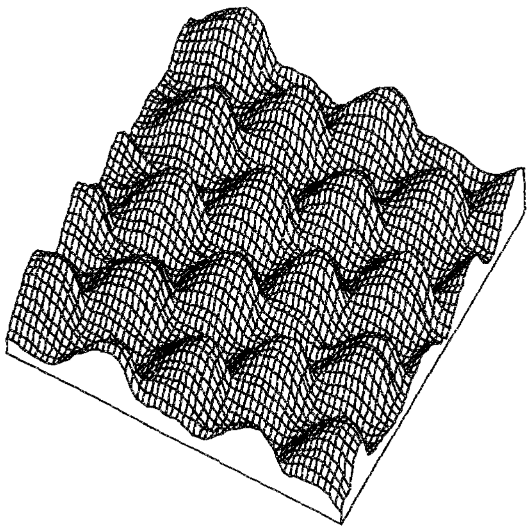

FIG. 5. 1.0 by 1.0 nm net drawing of the STM picture obtained from ordered graphite. The height variation is 0.4 nm and the sample was biased at negative 100 mV. The current was held fixed at 1.0 nA.

tions with hidden line removal. A simple data smoothing routine may also be employed.

III. RESULTS

Figure 4 shows a perspective view (30° rotation and 80° tilt) of a 2.1×2.1 nm^2 segment of the surface of monochromator grade graphite[11] obtained with our microscope using the short PZT-5A scanner. This is a true, constant current picture (2.0 nA) requiring a total of 1 min to acquire. The amplitude of the features observed is 0.9 nm. The data have not been smoothed or filtered in any way. The W tip was biased at + 100 mV relative to the graphite. The periodic features have a 0.246 nm spacing and do not correspond to individual atoms.[12–14] This spacing was used to calibrate the x- and y-axis sensitivity of the two piezoelectric tube scanners used.

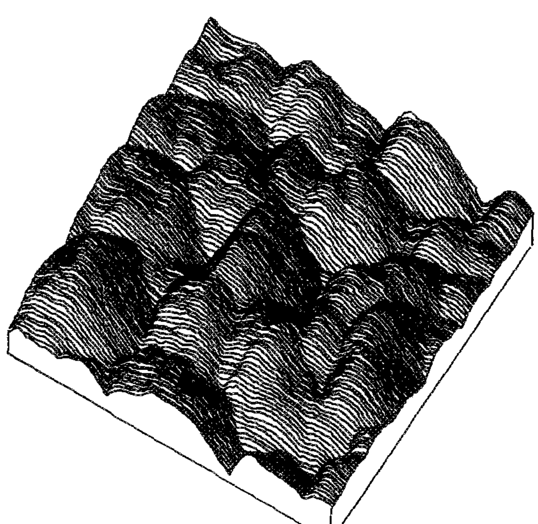

FIG. 6. 68.0 by 68.0 by 5.0 nm high picture of the surface of a 10.0 nm resistively deposited gold film supported on a microscope cover slip. The figure has been rotated 30° and tilted up by 80°.

Figure 5 is a net plot of the surface of graphite obtained from an inexpensive piece of ordered graphite. To the eye, this sample appeared to have an irregular surface. Small regions, however, showed good ordering. The data were smoothed once using the gradient method provided in the PLOT88 package.[15]

Figure 6 is a perspective-view (30° rotation and 80° tilt) picture obtained from a microscope cover slip that was coated with 10.0 nm of Au. The Au was deposited from an alumina-coated filament at a rate of 0.1 nm/s. The substrate was maintained at room temperature during the deposition. Figure 6 shows a 68.0×68.0 nm^2 segment of the surface. The maximum height excursion in the picture, lowest valley to highest peak, is 5.0 nm. The "rolling hills" observed are characteristic of thin films of Au deposited on flat substrates at room temperature.[3,5,16,17] The tip was biased at 100 mV and the current was held constant at 0.5 nA.

The figures were drawn with the use of a commercial plotting subroutine package, PLOT88.[15] Our main FORTRAN plotting routine reads data files, requests orientation information, calls the PLOTT88 package, and then produces plots on either a Hewlett Packard ColorPro plotter or on an EGA display.

The least desirable attribute of this STM is the poor thermal stability. After a sample change, a period of 30 to 90 min is often required to reduce thermal drift to acceptable levels. Replacing the motor stage frame with a steel frame should improve the thermal stability of this STM. Even greater improvement would result from the use of a short invar insert (equal in length to the piezoelectric tube) in the motor stage side walls and in the frame.[18]

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