Design of a simple and compact scanning tunneling microscope

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A simple and compact scanning tunneling microscope (STM) for operation in air is described. The design of the STM unit is based on the inertial sliding systems described in the literature, with some modifications to improve the stability of the scan unit. The STM is built of two concentric piezo electric tubes, the inner one for scanning the tip and the outer one for moving the sample holder towards or away from the tip. The vibration isolation system, mechanical construction of the scan unit, probe tip preparation, analogue and computer control electronics have all been described in this article. An analogue feedback system has been used for the constant current imaging. Images of highly oriented pyrolytic graphite have been obtained in air both on constant current and constant height modes of operation. The atomic resolution images obtained show the hexagonal lattice of graphite clearly.

SCANNING tunneling microscopy (STM) invented by Gerd Binnig and Heinrich Roehrer[1] and their associates at the IBM Research Laboratory in Zurich in the early eighties has heralded a new era in the surface science. Unlike other microscopies like SEM, TEM, STEM and electron diffraction techniques which need ultra high vacuum, STM can function in a variety of environments like air, electrolytic media, high pressure systems in addition to UHV over a range of temperatures. These features make STM an ideal choice for surface science studies. The solid–liquid interface, so vital to studies as diverse as electrochemistry, biology and analytical chemistry is now accessible to atomic-scale analysis, and we can foresee developments as path-breaking as those that ultrahigh vacuum technology brought to vacuum surface science.

The basic principle of STM involves positioning of a chemically inert fine metal tip (W, Pt, Pt–Rh/Ir and Au) within a few angstroms of the sample surface so that the electron wave function of the tip and the sample overlap. Application of a low bias voltage between the tip and the sample at this close range causes electrons to tunnel across the gap resulting in a current of typically nA and less. For a typical metal barrier height of 4 eV, there is a change of current of an order of magnitude for a change of gap separation by 0.1 nm. This extreme sensitivity of STM means that the features which are of atomic dimensions can be accurately imaged provided the gap distance is accurately controlled. Actually the STM image corresponds to a contour map of local density of states (LDOS). Hence the image obtained from the data is topographic only as a first approximation so that we obtain the 3-D map of the surface atomic positions. More importantly, the image provides the surface electronic information (LDOS) and by varying the bias voltage at any fixed location of the tip, it is possible to get information on the spatially related barrier height which is related to the work function. This technique is known as scanning tunneling spectroscopy (STS)[2]. In recent times, STM has also proved itself to be an indispensable tool for electrochemical studies[3]. In the electrolytic systems, it is necessary to independently control the tip and sample potentials by means of a bipotentiostat and also minimize the faradaic current flow through the tip by proper insulation, except at the very end, so that the tunneling current is the dominating component.

We describe in this article a simple and inexpensive design of a scanning tunneling microscope based on an inertial sliding coarse approach mechanism[4] that we have built and are using. We are currently engaged in the process of converting this into an *in situ* STM for electrochemical studies.

Design

The major factors in the design of an STM involve efficient vibration isolation, minimum thermal drift, proper feedback control, atomically sharp tunneling tips and coarse positioning. Vibration isolation is generally accomplished by increasing the resonance frequency of the scan unit – such as using tube piezo scanners – so that it is completely isolated from the externally or internally generated low frequency vibrations[5]. The thermal drift is compensated by careful selection of materials and a symmetric design of the scan unit. Very sharp – ideally with a single or a few atoms at the tip – tunneling probes can be obtained from electrochemically etched inert metal wires such as Pt–Rh or W. The essential requirements for the optimum feedback in the constant current mode of operation have been now fairly well understood after the much quoted work of Park and Quate[6]. However, it is the long range one dimensional translation known as coarse positioning which has proved to be a major challenge ever since the designs of home-made STMs started. This is because the piezo scanner usually has a total scan range of only a few micrometers and it is therefore necessary to use a mechanical means of coarse approach to bring the tip close to the sample without encountering a disastrous 'tip crash'. As the piezo that is used in STM is normally quite small, it is the coarse approach module that decides the overall size of the STM. However, in order to minimize the 'mechanical loop', and increase the resonant frequency, it is essential to decrease the overall size. However, among the several designs that are based on stepper motor control[7], inchworm control[8] and inertial sliding mechanism[9], it is the last one which we have chosen as it has been proved to be the least expensive and simple in design. In the inertial sliding arrangement the stage is placed in a position such that when a suitable waveform is applied, the stage moves relative to its starting position by a small increment. This is because the large acceleration provides enough inertial forces that exceed static friction during the falling portion of the saw tooth waveform, while during the slow rise of the waveform the stage essentially follows the piezo motion.

Coarse positioning

Among the several inertial sliding mechanisms reported in the literature, we have used the one based on that of Agrait[10], but with several modifications to improve the stability of the scan unit. While we have used horizontal motion of the sample stage, our design of the scanner is based on two concentric piezo electric ceramic tubes having a common base made of cylindrical PVC block. The piezo tubes (PZT-5H, EBL Division, Stavely Sensors) have silver metallized coating both on the inside and the outside. The outer piezo is 25 mm long and 12.5 mm outer diameter while the inner one is of the same length, but of 9.375 mm diameter. The outer surface of the smaller tube is sectioned into four metallized quadrants. From the analysis of Chen[11], the X and Y displacements are given by, $K_x = K_y = 2\sqrt{2}\, d_{31} VL^2/\Pi Dh$, and Z displacement is given by, $K_z = d_{31} VL/h$, where K_x, K_y, K_z are respective diplacements, d_{31} is the piezo coefficient, L, D and h are respectively the length, diameter and the thickness of the piezo tubes and V is the applied voltage.

The Z response of outer piezo is 5.8 nm/V while that of the inner one is 8.6 nm/V. The inner piezo displacement in the X–Y direction is 20.8 nm/V. The maximum X–Y motion is 2.5 micron for an applied voltage of about 120 V.

A glass tube of about 20 mm length and 12.5 mm dia is attached to the other end of the piezo using cyano acrylate 'super glue'. The glass tube has two grooves etched along its entire length over which the sample stage moves. The sample stage is a split brass block, the two halves of which are held in position by a pair of spring loaded stainless steel pins. The sample holder which is inserted into the glass tube, slides on the etched railings with two stainless steel pins glued to it on either side as shown in Figure 1 *a*. The spring tension should be such that the sample holder slides smoothly, if gently pushed manually, and at the same time should stay in position when no force is applied. We have, however, found from experience, that spring tension is not at all critical and at worst affects only the rate of motion.

The inner piezo tube holds the tip wire coaxially. This is achieved by inserting the tip into a syringe tube which in turn is positioned at the centre of the bakelite frit that is glued to the inner piezo by super glue. The coarse positioning is achieved by applying a saw tooth voltage to the outer piezo tube whose inside metallized portion is grounded. This compresses the tube resulting in the glass tube containing the sample holder being brought closer to the tip. During the sudden release of the tube during the falling portion of the saw tooth waveform, the glass tube which is attached to the piezo quickly retracts, while the sample holder cannot due to

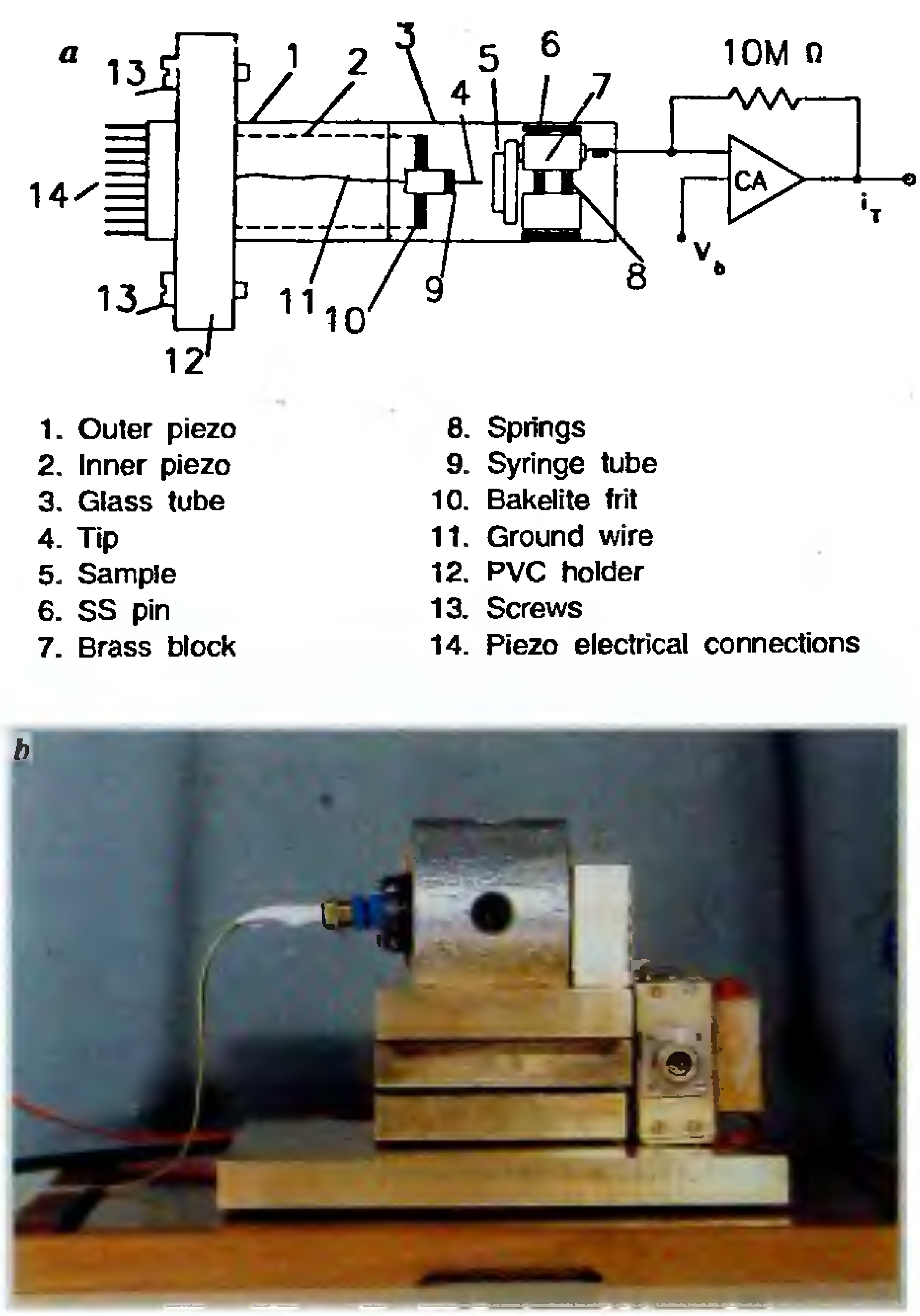

Figure 1. Scan unit of the STM. ***a***, Details of the parts; ***b***, Photograph of the unit mounted on the vibration isolation stack.

inertia. This results in the sample holder being translated towards the tunneling probe by one step. By the application of a train of saw tooth pulses of about 120 V amplitude and fast scan rate, it is possible to attain speeds of the order of 1 mm/s both towards and away from the tip. But normally the sample holder is moved at a rate of 0.1 mm/min after visually positioning the sample holder at a distance of about 0.5 to 1 mm separation from the tip using a hand magnifier. Then by the application of a slow ramp the sample stage is translated in small increments until the tunneling current is detected. We have calibrated the movement of the sample stage, at different sweep rates, by laser interferometry. Normally the movement of the sample is observed only above a threshold voltage of about 20 V. This is not a handicap though, as the rate of motion can be easily controlled by varying the sweep rate of the saw tooth waveform. Once the tunneling range is reached, the X–Y scan of the tip is started by the application of an asymmetric voltages of $+X-Z$ and $-X-Z$ on the two opposite sides of the metallized quadrant and $+Y-Z$ and $-Y-Z$ on the other two opposite quadrants of the piezo ceramic tube. The axially symmetric design of the unit along with the piezo tubes of the same length attached to a common base, minimizes the thermal drift considerably. The electrical contacts to the sections of the piezo tube are provided by soldering with low melting indium solder. A photograph of the scan unit resting on the stack of brass plates is shown in Figure 1 *b*.

Tip preparation

The STM tips are prepared by electrochemcial etching. The Pt-Rh (13%) wire of 0.25 mm dia is ac etched in a solution containing 5% each of sodium chloride and sodium nitrate while the tungsten tips are prepared by etching in 5% potassium hydroxide. Owing to 'necking effect', the lower portion of the tip immersed in the electrolyte drops, leaving a sharp tip. One can see a bright glow at the end of the tip wire as it detaches and drops resulting in the current cut off. The tip is gently removed after switching off the ac source without causing any vibration to the electrolyte. The tip is then dipped in 5% hydrofluoric acid to remove any surface oxides, rinsed in water and dried before inserting into tip holder. The aspect ratio of the conical end can be controlled by the depth of immersion into the electrolyte. The tips thus prepared have excellent reproducibility.

Vibrational isolation

The rigidity of the scan unit is the most important factor in the vibration isolation of the system. Hence the scan unit (Figure 1 *b*) is mounted inside a nylon block with a hole and is secured by means of two screws to a cylindrical PVC block. The entire assembly, screwed onto a thick brass plate, is mounted onto a stack of three more brass plates, each separated by a viton rubber O-ring. The stacked plates rest on a wooden plank and the whole assembly is suspended using bungee chord to the ceiling of the building. The wooden plank rests gently on to an inflated car tube placed on a granite slab. The entire unit is kept on a rigid teak wood table. The stacked plates assembly with viton spacer is effective at frequencies above 50 Hz and the bungee chord suspension system damps low frequency vibrations[12]. This vibrational isolation is extremely effective in spite of the fact that there is a road of a moderately busy traffic situated adjacent to the building that houses the STM.

STM electronics

The STM can be operated either in constant height or constant current mode of operation. In the former mode the tip is scanned over the sample in the same plane, resulting in the variation of the tip sample separation, which in turn changes the tunneling current. In the constant current mode of operation the tip is scanned such that the gap separation is always maintained constant at a reference value, by applying a feedback voltage to the inner piezo. The feedback voltage needed for maintaining the tunneling current constant is a measure of the surface topographical changes.

A block diagram of the controlling electronics is shown in Figure 2.

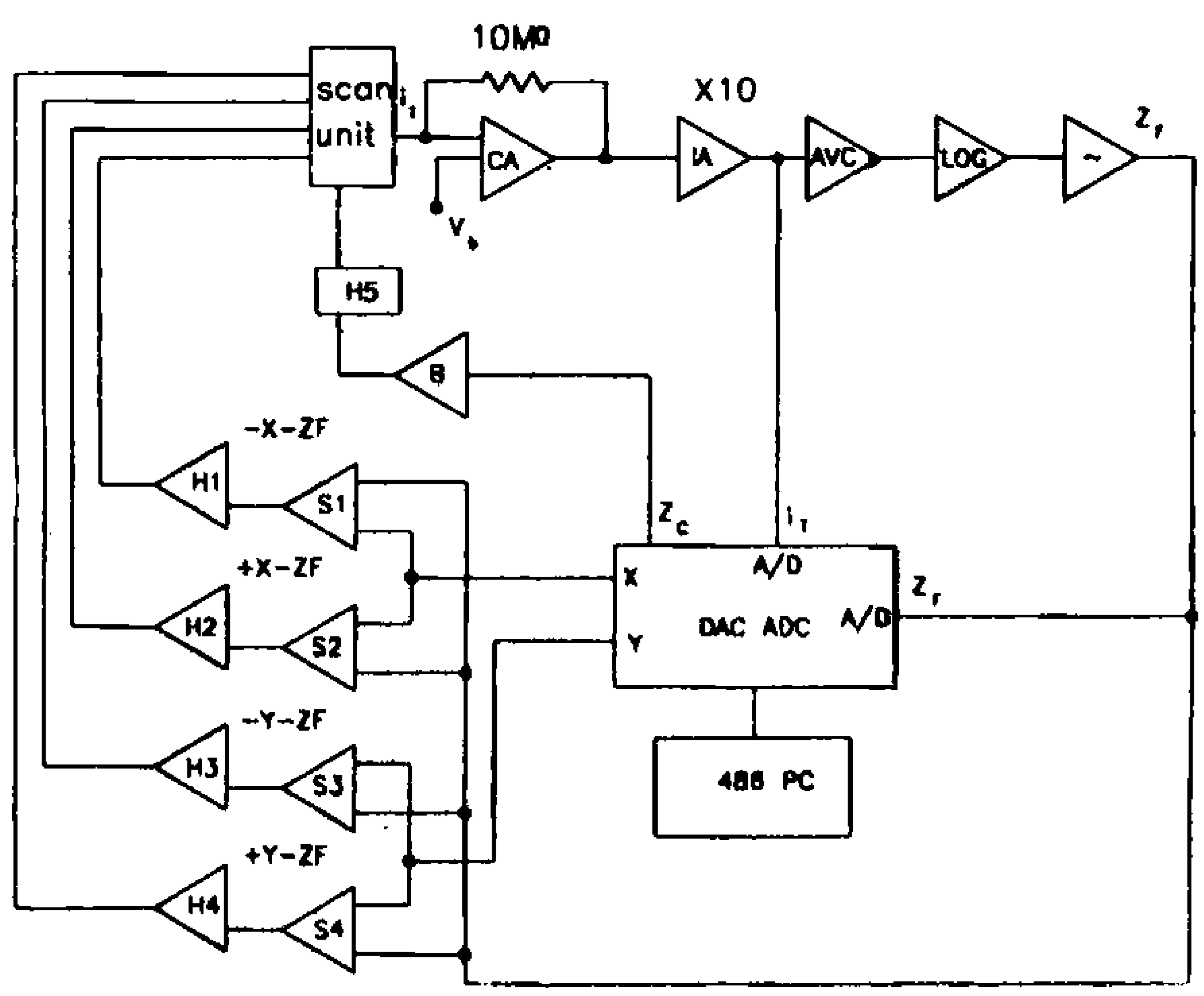

Figure 2. Block diagram of the scanning tunneling microscope electronics. CA, Current amplifier; IA, Instrumentation amplifier; AVC, Absolute value circuit; LOG, Logarithmic amplifier; ∫, Integrator; H1, H2, H3, H4, H5, High voltage amplifiers; S1, S2, S3, S4, Summing amplifiers; B, Buffer amplifier; ADC/DAC, Analogue to digital converter and digital to analogue converter and A/D and D/A are respective inputs; Z_f and Z_c, Z feedback voltage and Z course adjustment voltage respectively.

The electronics essentially comprises of the current amplifier, analogue feedback system, data acquisition using A/D and the piezo drivers comprising of the D/A card interfaced to a PC and high voltage amplifiers.

Current amplifier

The current amplifier is a high impedance operational amplifier AD 529 where the current is converted into voltage over a high resistance (10 MΩ metal film). This provides an output voltage of 10 mV for a current of 1 nA. The bias voltage, V_b, is applied to the sample from a battery through the non-inverting input of the current amplifier. The current amplifier is placed inside a grounded aluminum box and mounted close to the tunneling junction in order to minimize the capacitance contribution and consequent noise introduced by any lengthy cable. All the wires which connect the current amplifier to the rest of the circuit are well shielded and clamped at several places to minimize any vibration.

The feedback unit

The feedback unit essentially compares the actual tunneling current with a user specified reference current. If the measured tunneling current is too large, the feedback control system generates a voltage which is applied to the tube scanner to pull the tip back and *vice versa*. The piezo electric tube expands linearly with the applied voltage, which is infact directly proportional to the changes in the vertical tip position. This 'error voltage' therefore represents the surface height.

The output of the current amplifier is further amplified (X10) by means of an instrumentation amplifier (AD 624). An absolute value circuit (AVC) comprising two low noise OPA 27 op-amps provides the positive voltage to the logarithmic amplifier (AD 759 N). The logarithmic amplifier is used, as it linearizes the exponential behaviour of the current response which in turn improves the stability of the feedback loop. Besides, the reference tunneling current can be set by varying the input resistance of the log amplifier. In our circuit the reference current can be varied by means of a 10-turn potentiometer to any value between 10 pA and 10 nA. The output of the log amp is fed to the integrator whose time constant can be varied by a variable input resistance. This in effect adjusts the integral gain of the feedback circuit. The changes in the integrator output voltage actually represents the variation in Z voltage that is to be applied to the tip piezo to maintain the tunneling current constant at a reference value. This value, therefore, corresponds to the topographic information at any one location (x, y). The array of numbers collected, as the tip is scanned, represent the surface height information and is stored in a computer for analysis and display.

Computer control and data acquisition

A personal computer IBM compatible 486 DX interfaced with a Keithley 500 A measurement and control system containing a 16 channel, 16 bit A/D card and two 2 channel 16 bit D/A cards is used for data acquisition and control. While the assembly language driver routines are supplied by the manufacturers of the card, the measurement and control software has been written in Quick BASIC. The feedback signal from the integrator is fed to one of the channels of the A/D. The current amplifier output representing the tunneling current and the logarithmic amplifier output which provides the logarithm of the tunnelling current are fed to another two channels of A/D respectively. Two of the four D/A outputs are used for coarse positioning and Z control respectively. The other two D/A outputs are used for X and Y control. The tip scan and data acquisition proceed concurrently and synchronously. Both the data acquisition and X and Y scans are performed as background operation as this is the fastest mode of operation. The STM line scan image is displayed in real time.

High voltage amplifiers

A set of five high voltage amplifiers drive the piezo ceramic tubes for coarse positioning and x and y scanning. We have used APEX PA42 high voltage operational amplifiers[13] which are simple and convenient to handle. This amplifier has maximum voltage operation of 350 V and output current of 120 mA. The frequency response of 40 V/uS is adequate considering the fact that for the inertial slider the saw tooth to be applied needs to have a fast settling time. The X and Y outputs of the D/A are inverted by op-amp inverter (Burr Brown, OPA 27), to get $-X$ and $-Y$ outputs. The Z feedback voltage from the integrator is added to X, Y, $-X$ and $-Y$ at the inputs of the four PA 42 op-amps (at a gain of 10) to get the asymmetic voltages needed for the piezo X and Y controls and also for controlling the tip position in the constant current mode of operation. In the constant height mode the time constant of the integrator is made very large compared to the scan rate (about 10 Hz), that the feedback cannot respond to the changes in the tunneling current arising from the changes in the surface topography.

Operation of the STM

Operation of the STM consists in first positioning the sample holder as close to the tip as possible (usually about 0.5 mm) by visual inspection taking care not to touch the tip inadvertently. The tunneling tip is fully withdrawn by the application of maximum voltage (+ 120 V) to the inner piezo. A train of saw tooth pulses is applied to the outer piezo by a high voltage amplifier

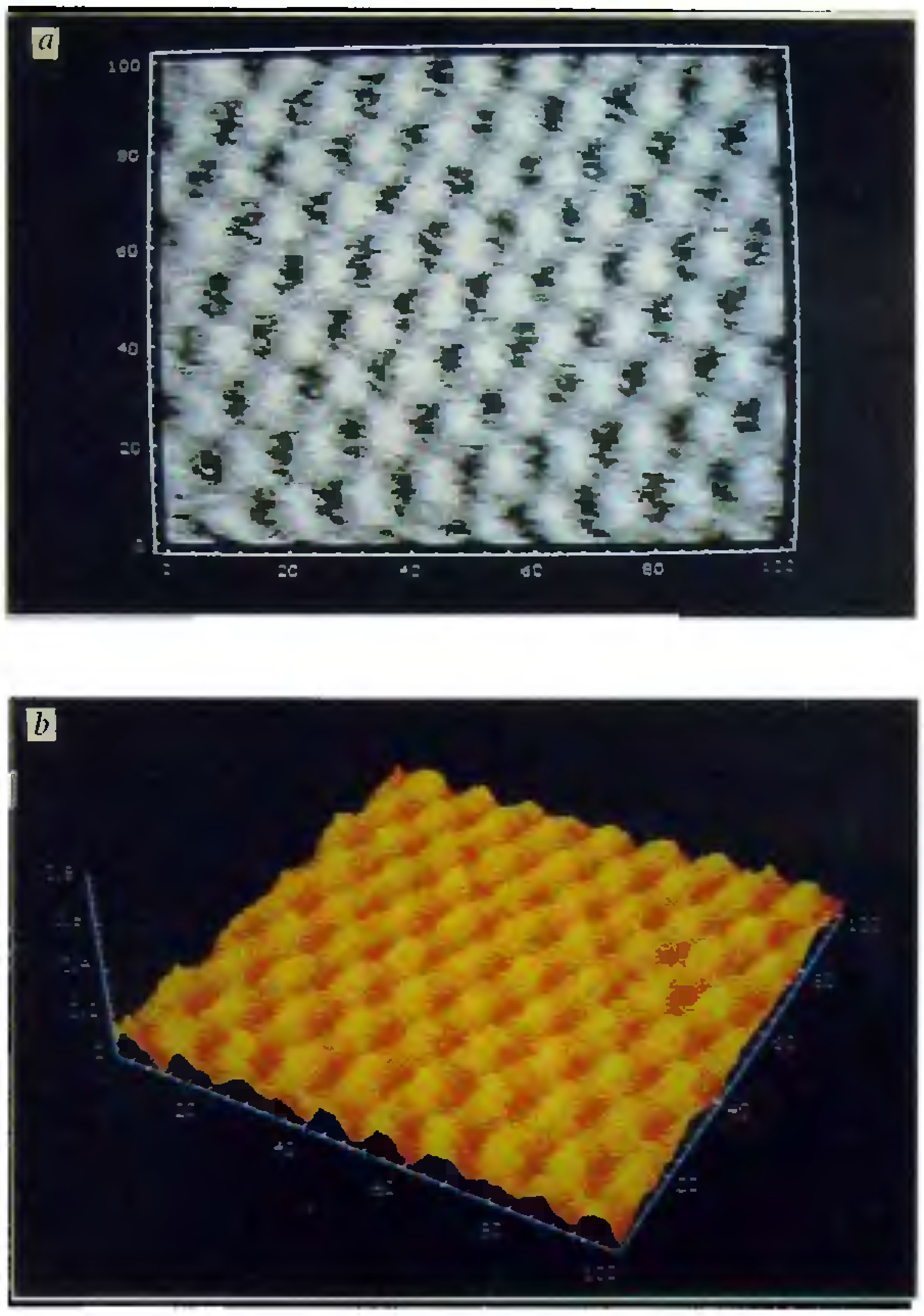

Figure 3. *a*, 2 nm × 2 nm scan image of highly oriented pyrolytic graphite. Scan mode: Constant height. Tunneling current, 1 nA, Bias voltage, 100 mV, Scan rate, 10 Hz; Tip, Tungsten. *b*, 3-D view of the image shown in Figure 3 *a*.

operating at a gain of 10, so that the sample holder approaches the tip in slow steps at the rate of about 0.1 mm/min. After every step of the sample holder, the inner piezo is stretched in small increments to detect tunneling current while the feedback is on. The sequence is repeated till the tunneling current is detected and immediately the *Z* movement of the sample is stopped by a trigger and the *X–Y* scanning is started. The *X* scanning is carried out twice, once in the forward direction and again while retracing the scan. Then a *Y* offset is provided and the data collected. This procedure is adopted to avoid any sudden tip movements as it occurs if a saw tooth is employed, which produces oscillations that adversely affect the image quality. A total of 20,000 data are thus acquired (200 × 100) and stored in a file. The data corresponding to forward *X* scan (100 × 100) is used for image display. While the line scan images can be displayed in real time, the 3-D images and the density plots are displayed using *Mathematica* software package after correcting for the plane tilt, but without any further processing.

Discussion

The reliability and stability of STM has been tested using a freshly cleaved Highly Oriented Pyrolytic Graphite (Type ZYA, Advanced Ceramic Co, USA). A 4 mm × 4 mm sample is stuck to the sample holder using silver epoxy paste which provides the electrical contact. Figure 3 *a* shows the raw data image of the HOPG surface (2 nm × 2 nm) while Figure 3 *b* the 3-D view of the same surface, obtained with a tunneling current of 1 nA and a bias voltage of 100 mV and scan rate of 10 Hz in the constant height mode of operation. A tungsten tip is used as a probe. The hexagonal symmetry pattern of graphite lattice is clearly seen. It is well known that the STM provides the electronic density of states rather than the actual positions of the atoms. Three alternate carbon atoms of the top layer of hexagonal graphite are bonded with the lower layer carbon atoms which are exactly below them (A sites) and have lower electron density and appear as dark spots in the STM image. This is in contrast to the other three carbon atoms represented by bright spots that have higher electron density as they are located right above the central hole of the lower layer hexagon (B sites)[14]. The distance between two bright spots corresponds to 0.250 nm and the C–C distance is 0.145 nm which is consistent with the values reported earlier for surface structure of graphite[14].

To summarize, we have designed and successfully tested a simple and compact scanning tunneling microscope and shown that it can provide atomic resolution image of graphite in air. This STM has presently been modified to be used as *in situ* STM for electrochemical studies.

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