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Design and Construction of a Cost Effective Computer Controlled Scanning Tunneling Microscope and Probe Production Technique

KEYWORDS
Nanotechnology, Scanning Tunneling Microscope, STM Probe, Computer control, AVR Microcontroller

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ABSTRACT *Working in the field of Nano technology requires instruments that can visualize and manipulate nanostructures. A Scanning Tunneling Microscope (STM) can analyze a sample surface at nano-scale and produce its topographical image. The design of the STM is based on Screw-Lever Motion demagnifier described in the literature. The scanner uses three orthogonal stack piezo cubes joined together with a metallic tripod. Images of highly oriented pyrolytic graphite have been obtained in air with constant current mode. The atomic resolution images obtained clearly show the hexagonal lattice of graphite.*

In this article, we describe design and construction of a robust low cost computer controlled constant current imaging STM. We also explain a simple method used to produce probes (tips) for the microscope. Major sections such as vibration isolation system, scan unit, analogue and computer control electronics have all been described. Data acquisition system designed and built is very low cost and easily available to everyone. The software that controls the system was written in Delphi language. To achieve cost effectiveness, expensive components and royalty controlled software have been avoided. The STM designed is therefore low cost, efficient and suitable for students, researchers and low budget institutions.

I: INTRODUCTION

Scanning Tunneling Microscope (STM) is an instrument that utilizes tunneling current (a quantum mechanical phenomenon) to analyze sample surface at nano-scale and produces topographical images of the sample surface [1]. Tunneling occurs when a chemically inert fine metal (W, Pt, Pt-Rh/Ir, Au) sharp conductive probe approaches a conductive surface which is clamped at a certain potential difference, without touching the sample (Figure 1).

At this point, as electrons jump (tunnels) through the distance between sample and tip, an extremely small current flows [2]. When Tunneling current between sample and tip is established, feedback controller circuit and piezoelectric transducer adjust the distance such that a steady tunneling current flows between sample and tip. As the tip scans the sample, topographic image of its surface is obtained by measuring the variations in the tunneling current. Resulting images could have atomic resolution if the gap distance can be accurately controlled. Actually, STM images correspond to a contour map of local density of states (LDOS) [3]. 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.

First STM was constructed in 1981 by two scientists named Binning and Rohrer [1] and today these microscopes are basic instruments for any nanotechnology lab.

The aim of this research was to design and construct a robust and cost effective computer controlled scanning tunneling microscope which is suitable for analyzing nanostructure surfaces. This prototype microscope was constructed by simple, low cost and easily available components found in every instrumentation laboratory. It was designed for constant current mode suitable for surface analysis. The overall design idea of this system is sketched in Figure 1.

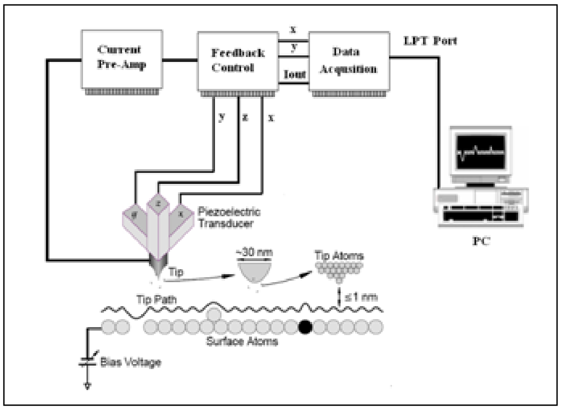

Figure 1: Sketches of STM design

Our design effort focused on seven major components; vibration isolators, scanner, tip approach system, probe tip fabrication method, control circuit, data acquisition board (DAC) and computer control software. Our simple vibration isolator, despite its simplicity, proved its efficiency when obtaining atomic images of graphite surface. The scanner, probe tip positioning system and electronic control were adopted from previous works and appropriate improvements were incorporated. The tip fabrication method was adopted from previous work and was improved. Computer interface board was designed and built based on an AVR microcontroller and computer image analysis software was specifically designed for the system.

An important characteristic of this prototype microscope is its portability and reliability. It can acquire atomic images of sample surfaces quite easily.

STM DESIGN

Major factors in design of an STM involve vibration isolation, proper feedback control, minimum thermal drifts, use of sharp probe tips and probe's coarse positioning mechanism. Vibration isolation is generally accomplished by increasing the resonant frequency of the scan unit which means scanner must be as small as possible so that it is completely isolated from the internally or externally generated low frequency vibrations. Thermal drift is compensated by careful selection of materials and a symmetric design of the scan unit. Because our system works in ambient condition thermal drift is not a major problem. Very sharp tunneling probe can be obtained from electrochemically etched inert metals like Wolfram. However, probe coarse positioning is the major challenge in the design of home-made STMs. This is because the piezo scanner usually has a total scan range of a few microns and it is therefore necessary to use mechanical means for coarse approach to bring the tip close to the sample without encountering a disastrous 'tip crash'. We used block stack piezo, PSt 150 /2x3/5 by Piezomechanik GmbH Germany which has static sensitivity of 7/150 micrometer/V [4] so if we drive it with 20 volts we could have 2 micrometer scan range.

To keep system cost down, we built a simple computer interface board which consisted of a microcontroller, an ADC, two DAC units and a parallel port interface. Computer control software and image processing was written in Delphi language [5] and free available charting packages [6] are used for display imaging and 3d reconstruction of the sample surface.

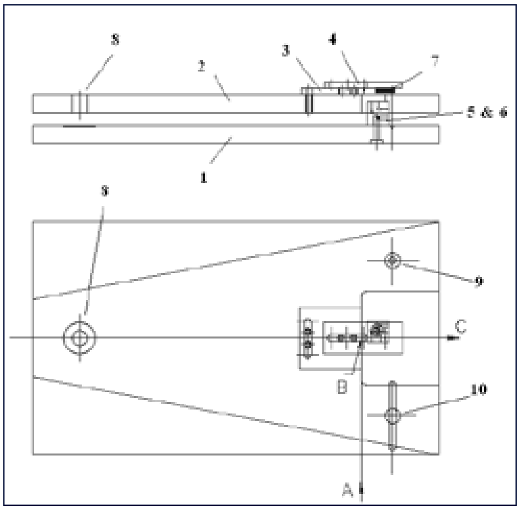

Figure 2: STM coarse/fine probe positioning mechanism , 1) lower plate: Specimen holder, 2) upper plate: holds scanner, 3,4) Scanner holder arm, 5) Scanner Block: holds piezo electric blocks, 6) Tripod: tip is placed here, 7) Magnet , 8)Fine positioning screw, 9,10) Coarse positioning screws.

PROBE COURSE MECHANISM

The simplest method for probe coarse positioning is screw-lever motion demagnifier (Figure 2) adopted from SXM project [7].

In this approach, three micrometer screws, AJS 0.5 by Newport USA (Figure 3) are used. Two of which serve for rough probe positioning and the third is used for fine position tuning. As our scan unit is small, 20*20*20 mm in size, the overall system size is determined by course positioning system. Among several system designs, we choose a simple one in concept, which uses two aluminum plates in parallel. The scanner and the probe is attached to the upper plate which is used for course probe positioning while the sample sits on the lower one (Figure 5).

Figure 3: High precision adjustment screws

The screw-lever motion demagnifier used has a leverage factor of 1/200. It has a coarse micrometer screw thread of 0.5 millimeter, yielding fine micrometer screw thread of 2.5 micrometer (Figure 2).

VIBRATION ISOLATION

The rigidity of the scan unit is the most important factor in vibration control of the system. To reduce the effect of low frequency vibrations, we used stacked plate ellastometer vibration damping system (Figure 4) which consists of three heavy granite slabs stacked on each other and are separated using viton damping [8, 9].

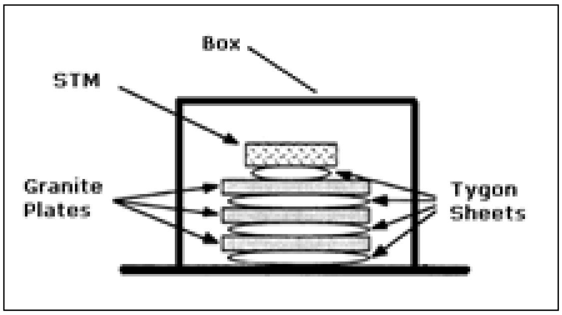

Figure 4: – Vibration Isolation System

To reduce the effect of acoustic noise, the whole mechanical part is enclosed on a metallic box which is electrically grounded (Figure 5).

Figure 5: STM mechanical components sitting on the vibration isolation system inside a metallic box.

TIP PREPARATION

The STM probe (tip) is the most important factor which determines lateral resolution of the system [10, 11, 12, 13]. We prepare our tips by electrochemical etching of W wire (Wolfram) with diameter of 0.3 mm with DC drop off technique. In this method, tip is immersed in a 2 Mol solution of KOH by 2 millimeters; a DC voltage is applied between tungsten wire as anode and another electrode as cathode (Figure 6). Owing to 'necking effect' the lower portion of the tip which is immersed in the electrolytic solution drops and a sharp tip remains at top. The tip is then removed and rinsed in water and dried before inserted into the tip holder. In this method tip sharpness depends on etching process cut off time which is controlled by an electronic circuit (Figure 7) that has been incorporated into the electronic control box of the STM.

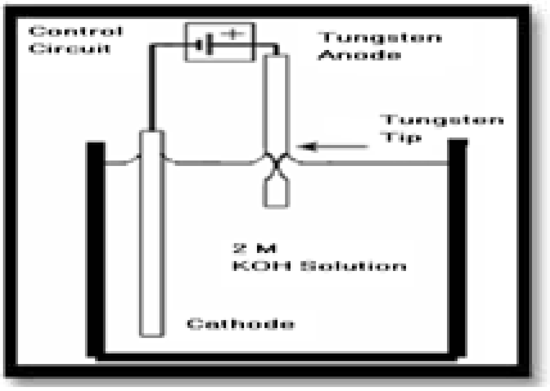

Figure 6: Electrochemical etching of Tungsten wire for probe preparation

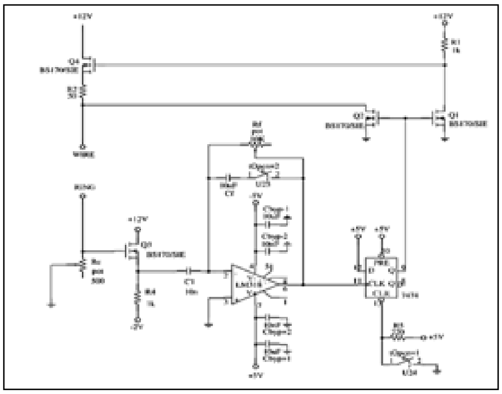

Figure 7: Circuit to control the cut off timing of the probe etching process

Electro-polishing current is essentially controlled by Rc pot. As the wire tip is shaping (rupture) the change in current at the gate of Q3 forces the integrator to produce a peak voltage that makes the flip flop change state and consequently turn Q1 and Q2 on. Consequently the gate voltage on Q4 is then reduced and eventually the supply voltage to the electrochemical process is cut off within 160 ns.

STM ELECTRONICS

Our STM can operate in constant current mode. In this mode of operation, the gap distance between tip and sample surface is always maintained constant by applying a feedback voltage to the Z piezo. However, to overcome the challenge of "tip crash" when approaching the sample and for positioning of the tip within the current tunneling distance, efficiently and routinely, We devised a simple visual method for tunneling screening: Two small LEDs are connected to the output of the integrator controller in opposite directions (back to back) to monitor saturation of the integrator, so when they both lit off, tunneling is established. A block diagram of the controlling electronics is shown in Figure 8.

CURRENT AMPLIFIER

The current amplifier is a high impedance low noise JFET input operational amplifier LF411 (Figure 9). Current is converted into voltage over a high resistance (10 MΩ metal films). This pre amplifier provides an output voltage of 100 mV/1nA. However, this output is further amplified by a JFET operational amplifier (TL074) as shown in Figure 9.

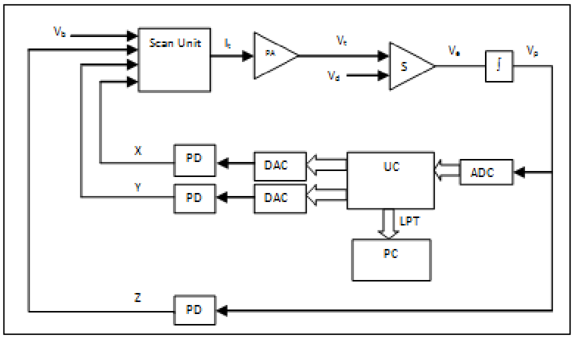

Figure 8: – Block diagram of the STM electronics. PA, Pre-Amplifier; S, summing amplifier; ∫, Integrator; ADC, analog to digital convertor (AD1674); UC, micro controller (Atmega128); DAC, digital to analog convertor (AD767); PD, piezo driver; PC, personal computer; Vb, bias voltage; Vd, datum voltage; It, tunneling current; LPT, Parallel port

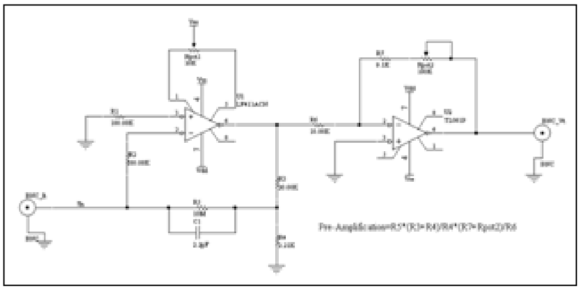

Figure 9: Current amplifier

The bias voltage (Figure 10) is applied to the sample from the power supply through a buffer and a low pass filter.

All wires connecting the current amplifier to the rest of the circuit are well shielded and secured at several places to minimize any vibration.

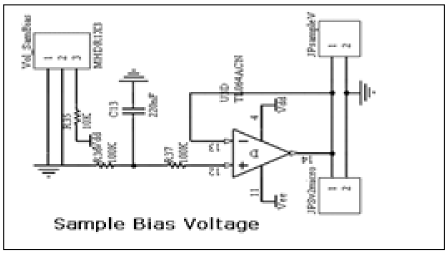

Figure 10: Sample biasing voltage circuit

FEEDBACK CONTROL CIRCUIT

The feedback unit compares the actual tunneling current with a user specified reference current. If the measured tunneling current is too large, the feedback control circuit generates a voltage which is applied to the Z piezo scanner to pull the tip back and vice versa. The piezo stack expands linearly with the applied voltage which is in fact directly proportional to the changes in the vertical tip position. This error voltage therefore represents the surface height. Figure 11 shows the electronic circuit used for feedback purpose.

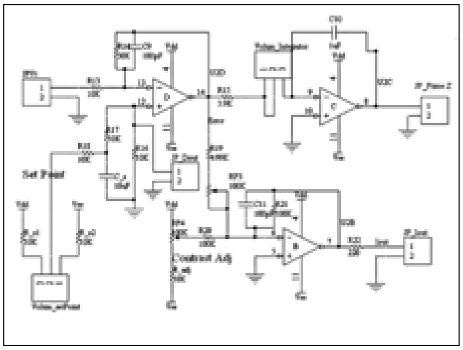

Figure 11: Feedback control circuit

COMPUTER CONTROL AND DATA ACQUISION

A simple and low cost computer interface board was designed and built. It essentially uses a microcontroller (ATMEGA128), an analog to digital converter (AD1674) and two digital to analog converters (AD767) with parallel port interface as shown in Figure 12.

The software for the micro was written in C language and the compiler to develop the code was CodevisionAVR from HP Info Tech. The Z Piezo voltage is fed to the ADC and after digitization is sent to computer control software through parallel port interface. X and Y position of scanner is sent from computer control software (application program) to the microcontroller and then sent to the DAC's. The STM line scan image is displayed in real time on PC control software.

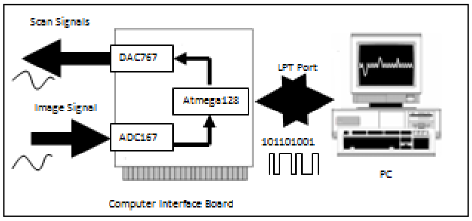

Figure 12: – Computer interface board

PIEZO DRIVERS

To scan the surface of a sample in a STM in constant current mode, the probe has to move in x and y direction while the distance between the probe's tip and sample's surface (Z direction) is measured and compensated continuously. The computer control software calculates the size of the step (X) and the sweep (Y) signals based on the image size and resolution required. The information is then sent to the interface board to be passed on to the DACs. The outputs from the DACs are fed into two low pass fourth order filters to make sure no digital noise or unwanted harmonics are present (Figure 13).

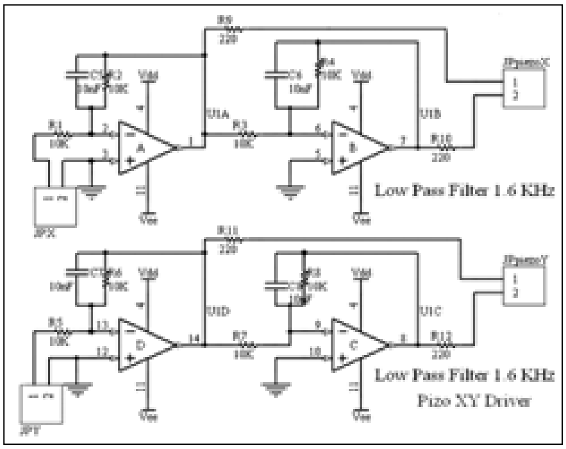

Figure 13: Low pass 4th order filters used with scan signals

The corner frequencies for the filters are set at 1.6 KHz. However, the filtered signals are then applied to the x and y piezos directly. Typical scan signals at the DACs outputs on an oscilloscope are shown in Figure 14.

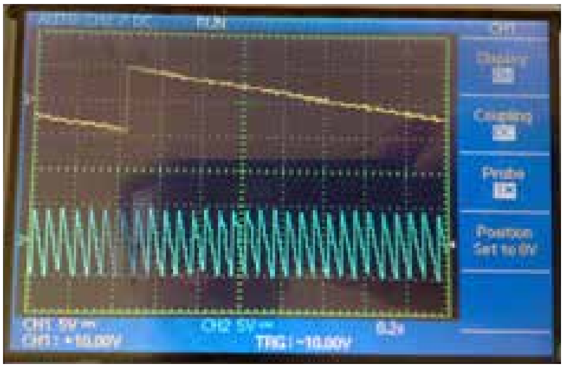

Figure 14: Typical y, x scan signal generated by the DACs

STM SOFTWARE

The software developed for the control of the STM comes into 2 parts. One is the application software that runs on the computer and has been written in Delphi language [5]. The other is the code developed in C language which is programmed into the microcontroller on the interface board. The application program basically does 3 tasks. 1) It produces appropriate scan signals and sends them to the interface board, 2) the program receives the signal from the STM via the interface board that represents sample's surface information and stores it. 3) Using the data from the STM, it builds up an image of the sample surface being scanned.

The code within the microcontroller takes command from the computer and essentially handles the signals regarding

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the ADC and the DACs. Figure 15 shows an overview of the firmware.

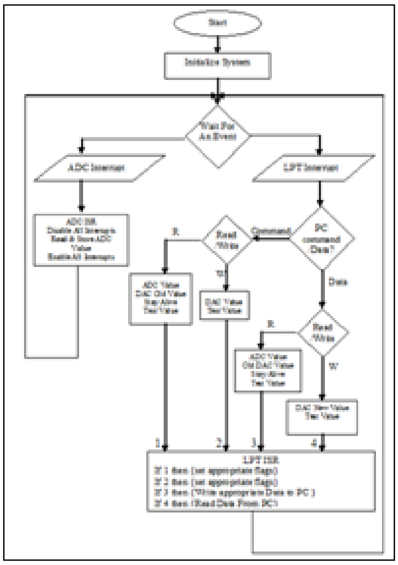

Figure 15: An overview of micro's code

Figure 16 shows the graphic user interface (GUI) developed for the control of the STM. It has deliberately been designed to be user friendly and simple to operate. The GUI allows adjustments of the parameters for the image to be acquired. Sampling frequency, image raster size, pixel size and scan step size can all be controlled by the user. The GUI can also display sample's image in real time, store new and or reload saved images. To obtain an insight into the frequency content of the image signal at testing phase of the system a Fast Fourier Transfer (FFT) function routine can display this information at the bottom of the GUI. This tool is very effective to detect noise and other unwanted signals that may contaminate the image signal. However once the system got working this part of GUI is used to look at the probe current shape to make sure that current tunneling is actually occurring and probe tip is at the right place. This is very helpful because it eliminates the possibility of receiving any probe current due to arcing or other possible artifacts.

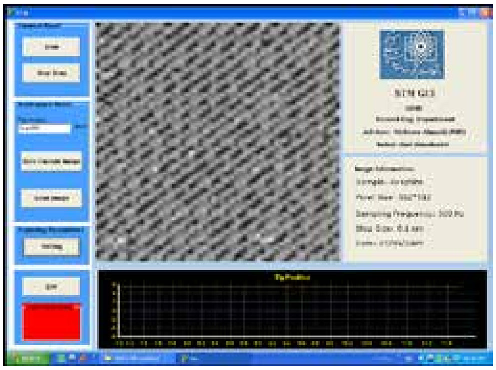

Figure 16: STM's graphic user interface

OPERATION OF THE STM

To obtain a surface image of a conductive sample by the STM, it is placed and fixed on the lower plate under the probe as shown in Figure 17 (b). The bias voltage cable is then attached to the sample usually by the use of some conductive glue. A set point voltage (tunneling current) is now selected and a bias voltage is applied to the sample. The probe is moved down towards the sample to within 1 mm of the surface using two coarse positioning screws. Naturally, visual inspection with a magnifier can help this process. To avoid a tip crash on the sample when the probe is sufficiently close to the sample's surface, one uses the fine positioning screw to direct the probe tip into current tunneling position. During fine approach, tunneling current is observed. At this point a voltage signal representing the tunneling current can be seen on the GUI on tip position display area (Figure 16). However, it can be shown that if the output of the integrator in the feedback circuitry (Figure 11) is buffered and fed into two light emitting diodes (LED) that are connected in a back to back configuration, when there is no tunneling current, Z piezo is elongated and is in its maximum length due to integrator saturation. As soon as tunneling establishes, feedback loop is closed and Z piezo withdraw itself so both threshold LEDs are switched off. However, if there is no tunneling current, one or the other LED will be on, depending whether the probe has touched the sample or has not reached the tunneling position. In our design we have used this technique for rapid tip positioning without crashing it. Figure 17(a) shows the actual electronic hardware controls of the STM while Figure 17(c) gives a block diagram of the feedback control that has been employed in the STM and corresponds to the design circuit of Figure 11.

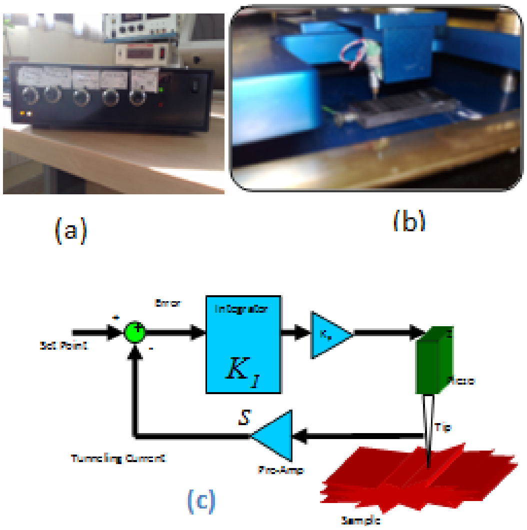

Figure 17: a) STM electronic hardware controls, b) Sample placed under the probe, c) block diagram of the feedback controller

A further tool we have devised to make sure that tunneling current is established is to apply a 100 Hz sine wave signal to the feedback circuit when two LEDs are off, so if tunneling is established and tip apex is good enough, we could see this sine wave at the output. Finally, when the probe tip is in its tunneling current position, the Start Scan Button on the GUI is pressed to commence scanning of the sample surface. As the probe scans the surface, the changes in probe position (Z piezo) is measured (Figure 11), digitized and read by the computer software to display the surface image.

RESULTS AND DISCUSSIONS

In every STM microscope, sample preparation is very important and choosing a good sample for imaging is somehow an art. We have used the system to analyze different samples. Here is an image which is obtained from a freshly cleaned graphite surface with a fairly sharp tip (Figure 18).

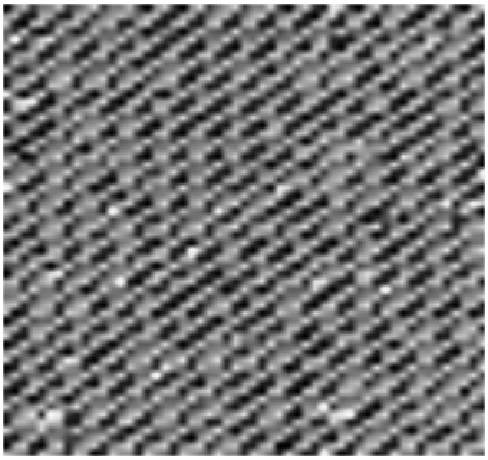

Figure 18: Atomic structure of graphite Picture size 5x5 nanometer

Several factors influence the apparent shapes of atomic lattice, like tip sharpness, bias voltage, and vibration isolation performance (14) (15) (16). However, this image was produced with a bias voltage of 100 mV and scan rate of 500 Hz in constant current mode in ambient temperature. The atomic structure of graphite is clearly observed with excellent resolution. We used a tungsten tip which was produced with our mentioned method. Figure 19, is due to a sample of gold (AU) sputtered on a polycarbonate disk (80 nanometer in thickness).

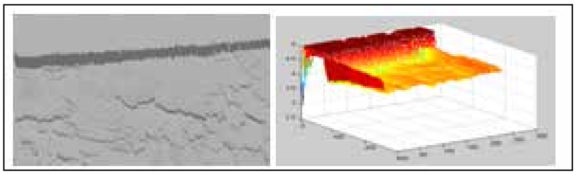

Figure 19: STM image of Polycarbonate surface covered with an 80 nanometer layer of gold. Image size is 1000x1000 nanometer

In Figure 20 we used the STM to obtain the surface image of a coin. The signal due to line scan of the surface clearly indicates the surface roughness.

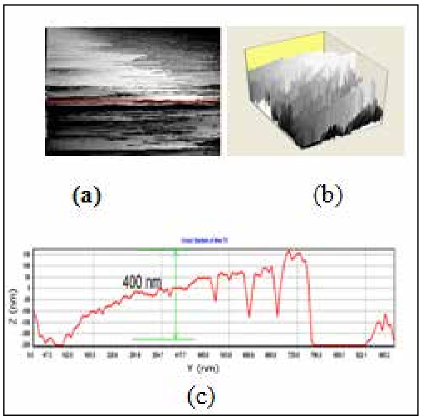

Figure 20: STM images of a coin surface, a) Actual surface, b) reconstructed 3D image of the surface, c) cross sectional (line scan) image signal

In this research, a very robust and economical scanning tunneling microscope was designed and constructed which could be used as a basic instrument for nanotechnology research labs. This prototype microscope was built by simple, low cost and easily available components which could be found in every basic lab and was configured as a constant current mode STM for surface analysis. A customized Data Acquisition Board with parallel port interface was used for computer interfacing and data acquisition and finally customized computer control software was used for image reconstruction and analysis.

During system development, design errors and challenges were solved step by step. The software codes for the computer control and the microcontroller were made to work properly and flawlessly. The electronic control tune up, vibration insulator optimization and ultimately optimization of tip fabricating method were dealt with along the research work. Different samples were analyzed by the system. Reconstructed images showed that system is working with high reliability and could acquire atomic images of sample surfaces.

CONCLUSIONS

The most important characteristics of this prototype microscope can be defined as: a) its portability, the system is designed such that its transportation is very easy and reassembling of the system needs minimum effort, b) simple but functional design, c) very low cost, d) using techniques that make probe tip approach into current tunneling position a routine task. Finally, the method offered for probe production with precise cutting off timing circuit yields a complete STM system.

We have shown in this work that a good robust cost effective STM and probe making system can be designed and built that is used for routine nano scale surface analysis. It has powerful features that make the system a valuable tool for researchers, individuals and institutions, yet, it costs less than a fraction of a commercially available system

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