Observing graphite atom structure by Scanning Tunneling Microscopy

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Abstract

By using a commercial easyScan STM system, we have observed the atomic structure of graphite by Scanning Tunneling Microscopy (STM). Besides, we can measure the geometric property, such as bond length and bond angle, from the resulting pattern.

Contents

1. Introduction

The Scanning tunneling Microscopy (STM) technique was developed by Gerd Binnig and Heinrich Rohrer in 1981 at the IBM research laboratory in Rüschlikon, Switzerland [1]. Because of its incredible resolution and wide applicable aspect, Binnig and Rohrer were awarded the Nobel Prize in physics in 1986 along with Ruska, whose was recognized for his contribution to the development of electron microscopy. Traditional optical microscopy has a limit due to the wave length of light, but STM doesn't have this kind of limit.

An electron has finite probability to enter a classically forbidden region and may tunnel through a potential barrier which separate two classically allowed region. The smaller the barrier is, the larger probability of tunneling will be. So the probe tip should be as closer to the sample surface as possible. [2]

We usually use Pt for probe tip. Pt tip is inert and is not going to react with the sample. The tip is being driven to approach the sample surface to a very close distance to the surface of the sample, about 1 nanometer. This small scale can allow the electron tunneling effect to happen. Due to the tunneling effect, there will be a tiny current. The tunneling effect depends exponentially on the distance between the tip and the sample. Pt tip can act as a good conductor for the tunneling current. [1]

From the recording of the movements of tip during the scanning, we can know the landscape of the sample's surface. This is fabulous, since we gather the surface information by scanning over each atom on the surface,

but the size of the atom compared with the scanning tip is only a ping pong ball to a mountain!

2. easyScan system

2.1 The specialty of easyScan system

The easyScan system is a commercial product composed of a STM microscope and a computer installed data collection software.

The eazyScan system has the following advantage:

1.It’s able to observe the surface of all samples in air under room temperature. There is no need of vacuum and low temperature. This makes it really convenient.

2.All functions are carried out by the computer, except the preparation of the scanning tip and sample.

3.The system is compact and easy to be handled.

The eazyScan system has the following disadvantage:

Many surface can only be imaged under vacuum.

2.2 Brief introduction about the eazyScan system

In the system, a small sharp platinum tip is clamped between two tiny springs and a platform. Driven by piezoelectric translators, the platform carrying the tip can move freely in x, y, z direction and let the tip scan over the sample surface by any desired direction.

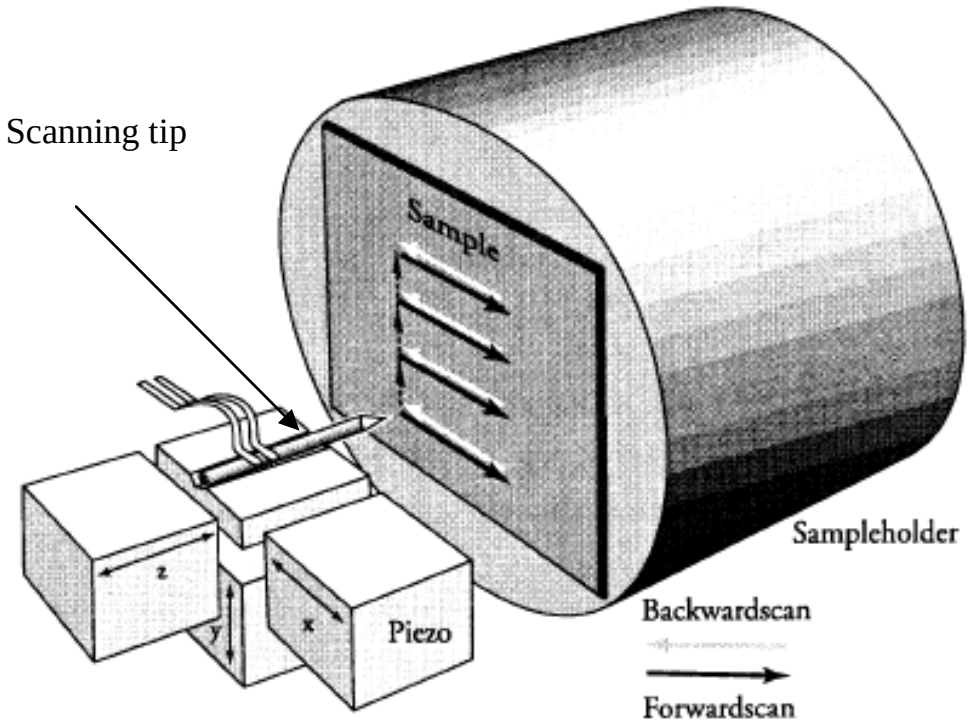


Figure 1

The tip is being driven by the computer to approach the sample surface to a very close distance to the surface of the sample, about 1 nanometer. This small scale can allow the electron tunneling effect to happen. The voltage applied in this system at the end of the tip is about 0.1V. This voltage is being set by producer and can't be altered. Due to the tunneling effect, there will be a small current about 1 nA. The tunneling effect depends exponentially on the distance between the tip and the sample. Since the tunneling current is extremely dependent on the distance between tip and sample, the tip movement can be controlled accurately. By keeping the current between tip and sample constant by a feedback loop (constant current mode), the distance between tip and surface is also kept constant and the tip follows the structure of the sample's surface. Since the distance

of the tip is kept constant to the surface of the sample, so when the surface is higher, the tip will withdraw a little in z direction; if the surface is lower, the tip will forward a little in z direction. All these movement will alter the z direction voltage of the piezo. And changing the x and y position of the tip, we can look over the different place of the sample. So the piezo ‘s z direction voltage is the actual data being plotted versus x and y movement. As a result, the shape of the surface can be simultaneously drawn on the computer screen line by line while we are recording the movement of the tip. [2] This method was first introduced by Binnig, Rohrer, and coworkers. It is called “constant current mode”. [1]

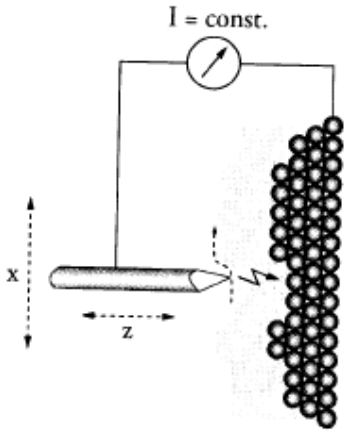


Figure 2

Another mode of operation can be done to increase the scan speed. It’s called “constant height mode”. In this mode, the tip is scanned rapidly at constant height over the sample surface while the feedback loop is slowed or turned off completely. [1] The current will vary at this time. [2]

2.3 Components of easyScan system

2. Serial cable between computer and control electronics
3. Power pack

4. Transparent cover
5. Scan head
6. Control electronics
7. Magnifying glass
8. 30 cm Pt/Ir wire for tips
9. Samples HOPG (graphite) and gold thin film
10. Software installation disc
11. Sample holder
12. Tweezers
13. Cable
14. Vibration isolation platform
15. Wire cutter and flat-nosed pliers (not shown)
16. Computer system

* Never touch the wire for tips (7), the sample (8), and open part of the scan head (4) with your finger.

* Only touch the sample holder at the black plastic end.

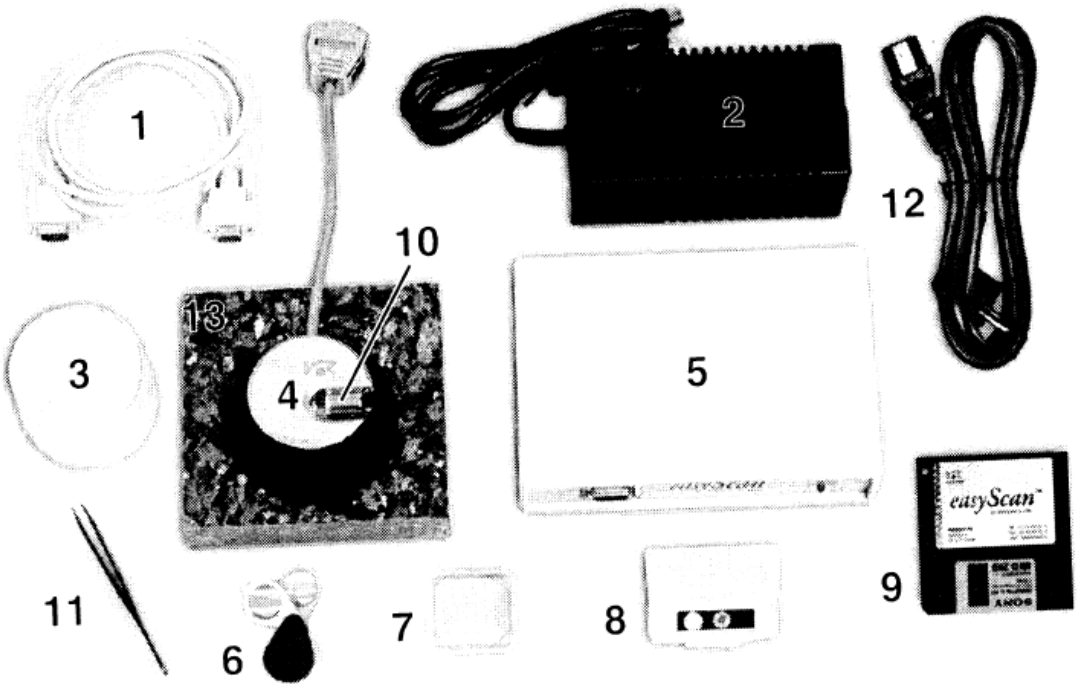


Figure 3

2.4 Computer system required

1. Pentium 133Hhz or higher recommended

2. Mb RAM or more recommended

3. graphics adapter with resolution of 1024 x 768 and 256 colors or better recommended

2.5 Preparing and installing the scanning tip

Perhaps this is the most technical and difficult part in the whole experiment. The procedure is as following:

1. Clean the wire cutters, the flat-nosed pliers, and the tweezers. Don’t touch the Pt wire with your hand.
2. Hold the end of the wire firmly with the pliers and cut a piece off approximately 5mm long.
3. Still holding this piece of wire firmly with the pliers, place the cutters at the free end, as obliquely as possible.
4. Close the cutter until you can feel the wire, then in the tilted direction shown on figure 4, pull and cut at the same time. The tip needs to be torn off rather than cleanly cut off to get a sharp end.

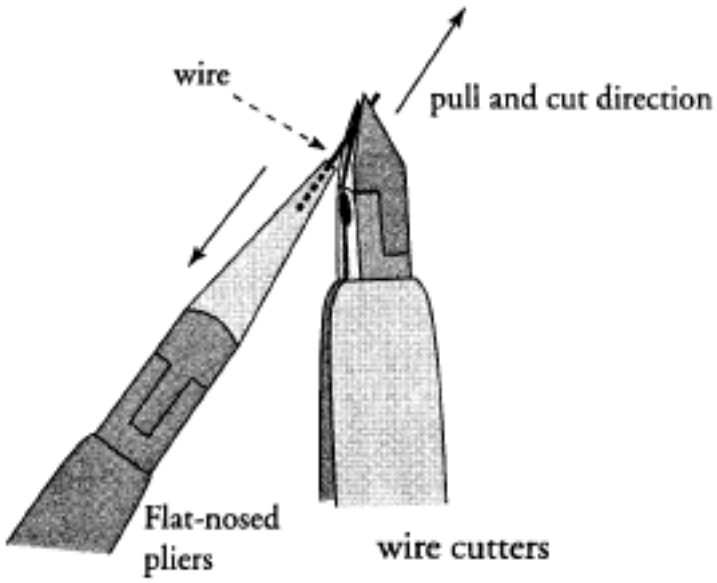


Figure 4

2.6 Preparing the graphite sample

1. Put the sample on the table with tweezers.

2. Stick a piece of adhesive tape gently to the graphite and pull the topmost layer off.

3. Remove any loose flakes with tweezers.

4. Place the sample onto the magnetic end of the sample holder by tweezers.

5. Place the sample holder in the scan head without touching the scanning tip.

3. Measuring Graphite

3.1 The reson using graphite

The reason we used graphite is a very periodical crystal only composed of one atom, carbon. The easy periodical structure can make the image be much easier to be observed.

3.2 Measuring Procedure

The procedure is as following:

1. Place the sample holder about 0.5 mm in front of the tip. Don’t touch the tip. Otherwise, you have to make a new tip again. (Figure 5)
2. Cover the system with the transparent cover without touching the sample holder.

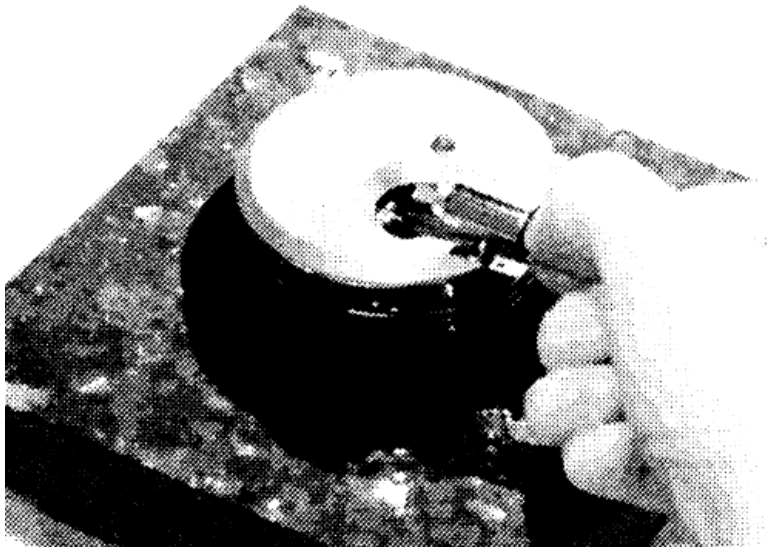


Figure 5

3. Touch the “Approach” button on the “Approach Panel” box. It might take hundreds or even more than one thousand step to reach the target Each step increase a few nanometer.
4. Start the measurement after it shows “Approach done”. There is a LED on the scan head. Orange means the distance is too big, no tunneling current is flowing. Red means the sample touch or crash into the tip, and the tunneling current is too high. Green means the sample is in measuring area, and the tunneling current is flowing.
5. Press “Full” button in the “Scan Panel” to maximize the range of the scan.
6. Start measuring by pressing “Start” in the “Scan Panel”.
7. If the “LineView” screen shows a smooth line, it means the information is good; if it is a nervous line, it indicates a bad tunneling contact. Usually this is caused by the tip being to blunt or instable. You have to stop the measurement and cut a new tip.
8. Adjust the sample’s tilt coordinates. The ideal scan range for the tip lie in the x, y-plane of the piezo-scanner. However, most time the sample is tilted with respect to that ideal plane. Since the sample’s tilt can’t be compensated directly, we have to adjust the scan coordinate. By setting suitable values foe the X- and Y-slope the scanner’s coordinate system is tilted so that the sample’s surface appears to lie in the ideal x, y-plane. Use the “X-Slope” and “Y-Slope” to make the scan line lies parallel to both the x and y axes. Figure 6 shows the relation between X- and Y-slope.

9. The menu “options” should be set to “Auto”. And the “Z-Offset” should vary automatically.

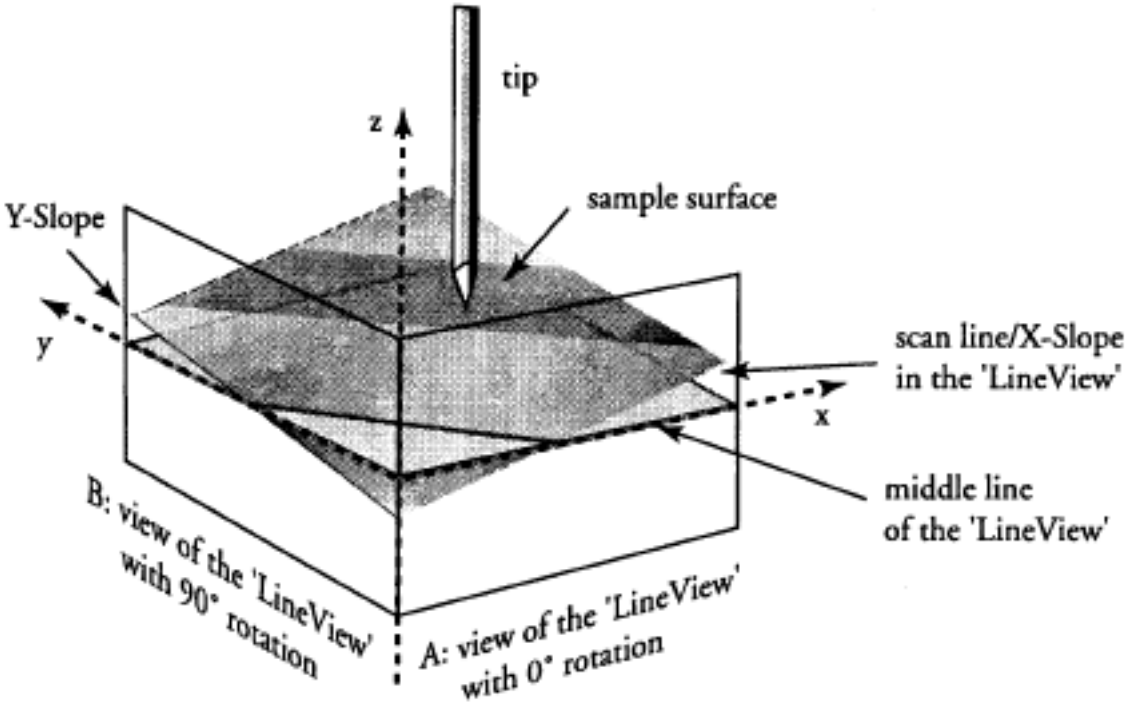


Figure 6

10. After getting a nice data, reduce the value of “Z-range” in “ScanPanel” from 200nm to 100nm and then to 50nm in order to concentrate the measurement to this range.
11. Click “Zoom” to concentrate on a small smooth region. Choose a square size to be 30-60 nm.
12. Atomic arrangements can normally be made out at a “ScanRange” of about 4 nm and at a “Z-range” of about 1.5 nm.
13. Set the “Time/Line” in the “ScanPanel” to 0.06 sec for atomic resolution.

14. Click “Photo” in “ScanPanel” to save the image.
15. Use “Stop” to stop the measurement.
16. Turn off and storing the instrument.

3.2 Graphite surface property

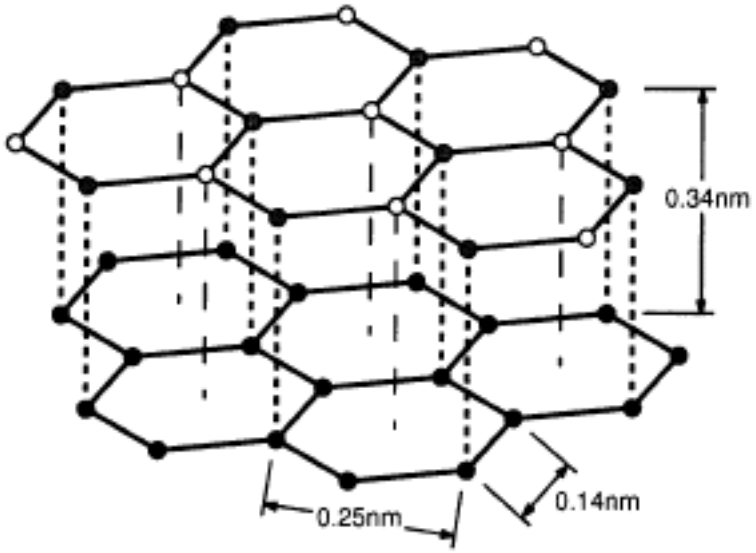


Figure 7

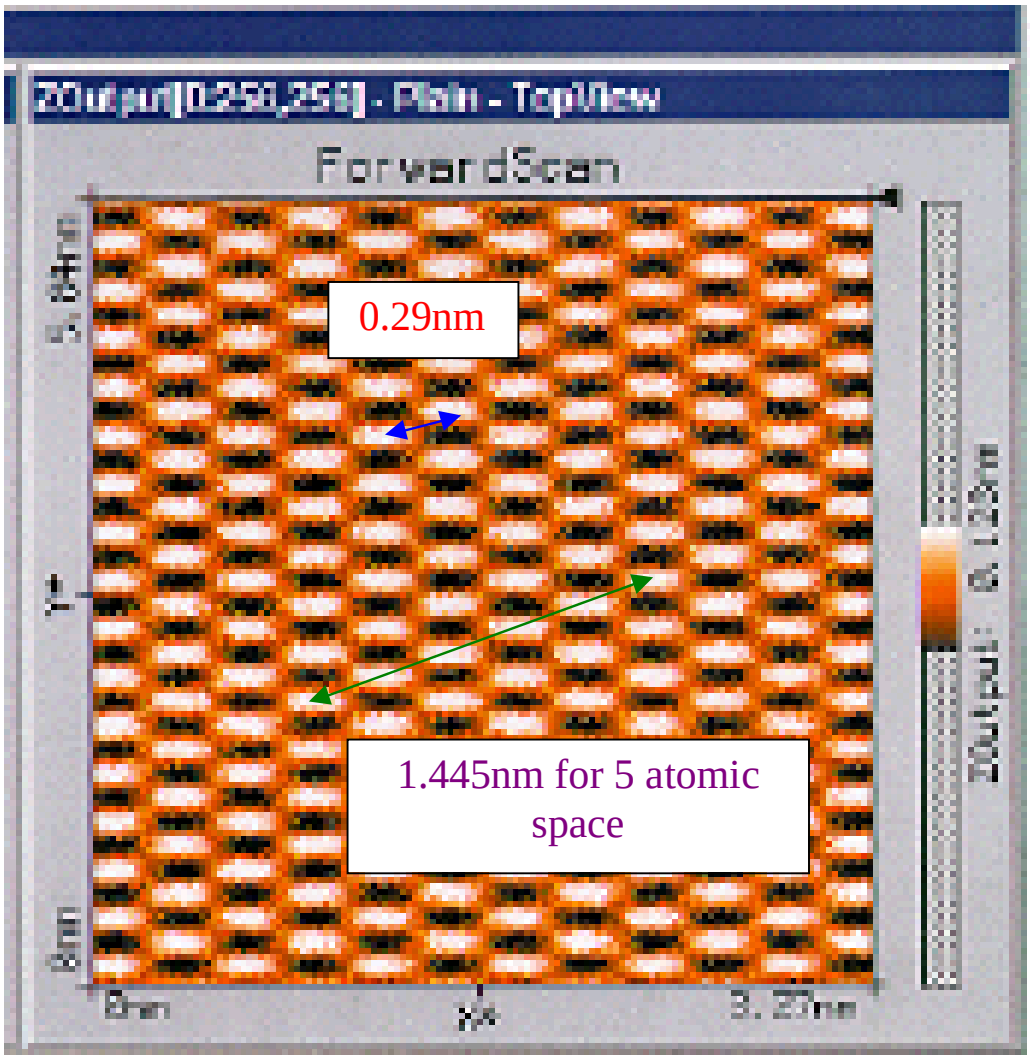
The above is a lattice structure of graphite. We can see that there are two different positions of the carbon atoms. One with a neighbouring atom in the plane below (grey) and one without a neighbor in the lattice below (white).

Consequently, the electrical conductivity of the graphite surface varies locally slightly so that the atoms without neighbors appear higher than those have neighbors. This also causes the lattice constant between the bright hills to have the higher value than normal, which is 0.25nm.

The bright spots on the diagram on computer show high point, and dark show low point.

3.3 Result

-- Scan --		Date	= 10-04-2000
ScanRange	= 3.27nm	Time	= 17:42:14
Time/Line	= 0.138s	-- Feedback --	
ZRange	= 0.781nm	GapVoltage	= 0.0501V
InputRange	= 12.5nA	SetPoint	= 1nA
XSlope	= 6.58°	IGain	= 15
YSlope	= 0°	PGain	= 14
Rotation	= 0°	LoopMode	= Run
XOffset	= 60.1nm	-- Global --	
YOffset	= 83.8nm	ScanHead	= STM
ZOffset	= 26.3nm		



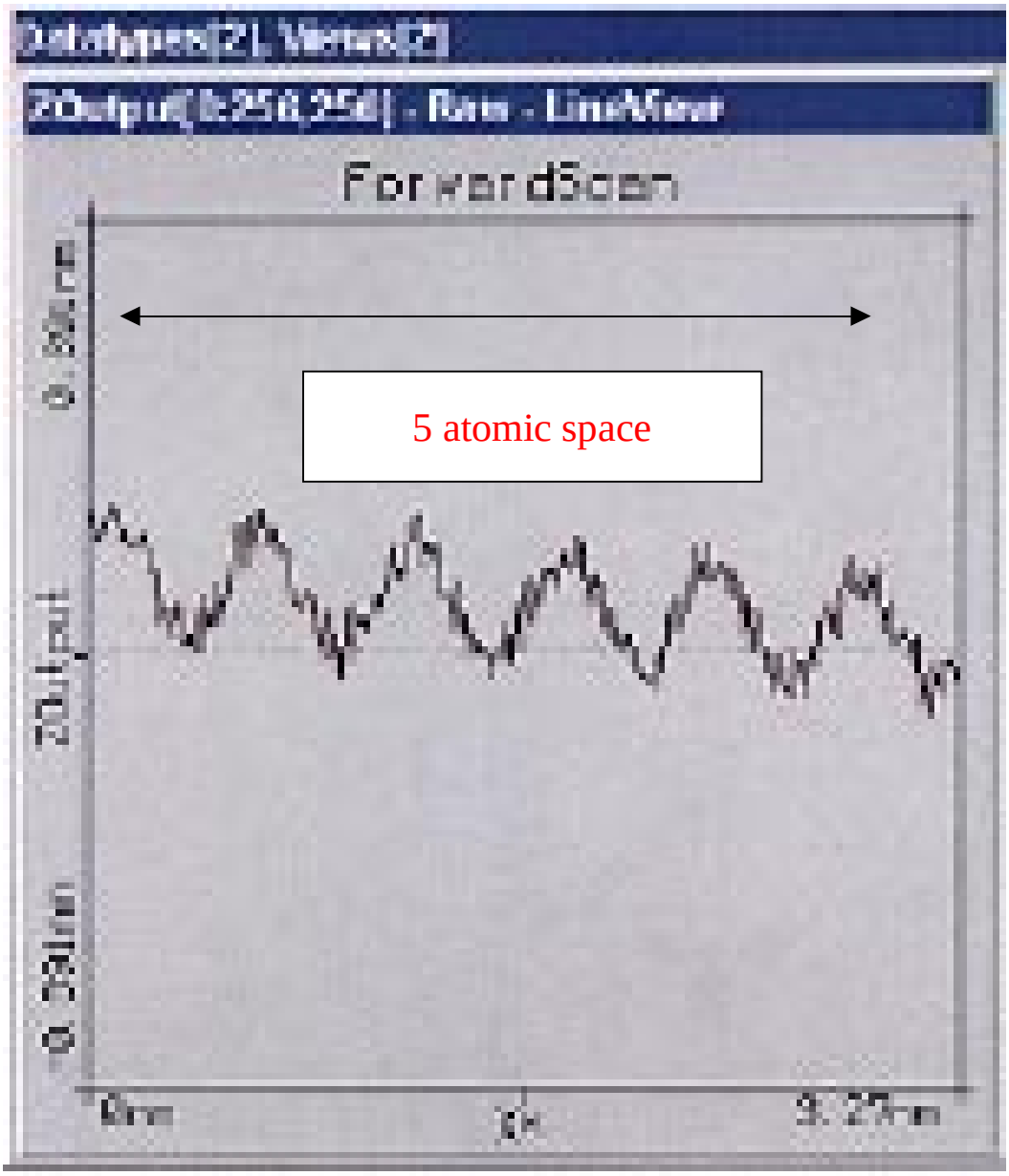


Figure 8

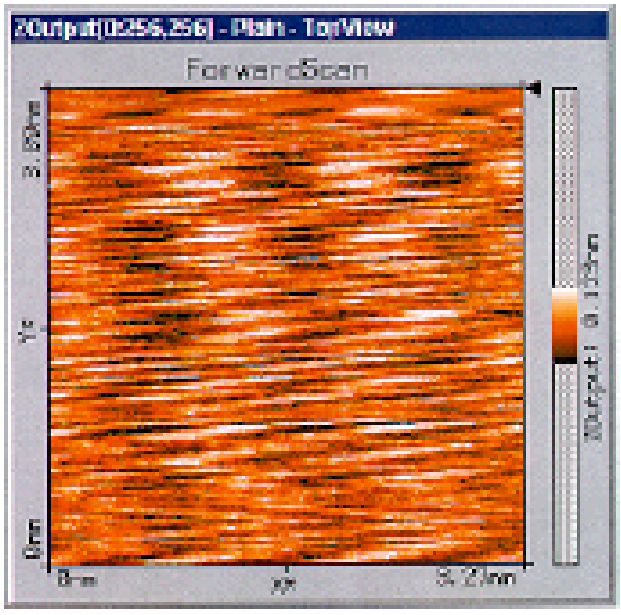
From above we can see the data, the image, and the signal. At the image graph, the blue line mark from on white dot's center to another dot's center. From the scale, we know that the distance between 2 white(black) dots is 0.29 nm. (We measure the distance across 5 atoms and divided by 5 for better precision.) Compared with the theoretical value 0.25 nm [2], the relative error is (0.29 – 0.25) / 0.25 = 16 %. This is a huge error. The error should most do with the measurement of length. Since we use ruler to

measure it, the minimum value is 0.1 mm. So we can't do very delicate measurement about the length on the graph.

However, it does show a periodic pattern about the graphite. Graphite is a structure of carbon only. It has only one atom, this make it an easy sample to be observed. Other error might due to the limit accuracy of the system and the computer screen's resolution.

Other failure graph has been shown below.

-- Scan --		Date	= 22-04-200(
ScanRange	= 3.27nm	Time	= 06:17:10
Time/Line	= 0.138s	-- Feedback --	
ZRange	= 0.781nm	GapVoltage	= 0.0501V
InputRange	= 12.5nA	SetPoint	= 1nA
XSlope	= -0.92°	IGain	= 13
YSlope	= 0.98°	PGain	= 12
Rotation	= 0°	LoopMode	= Run
XOffset	= -15.4nm	-- Global --	
YOffset	= 0.183nm	ScanHead	= STM
ZOffset	= 43.9nm		



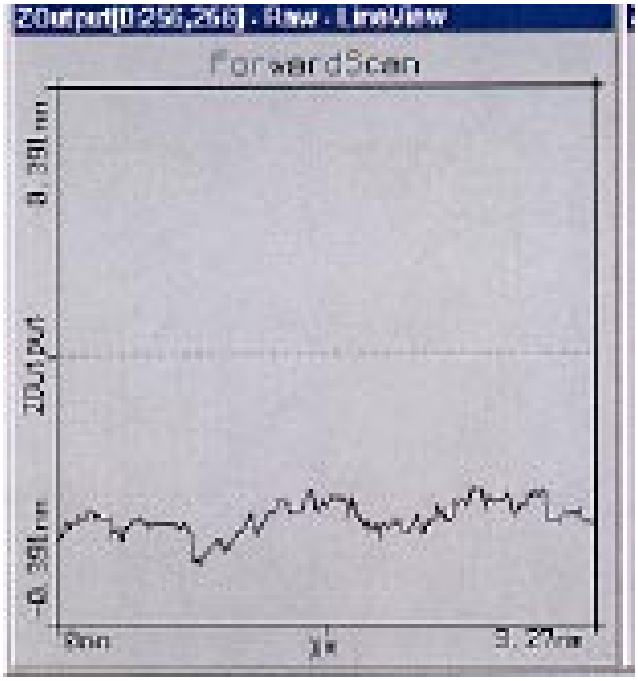
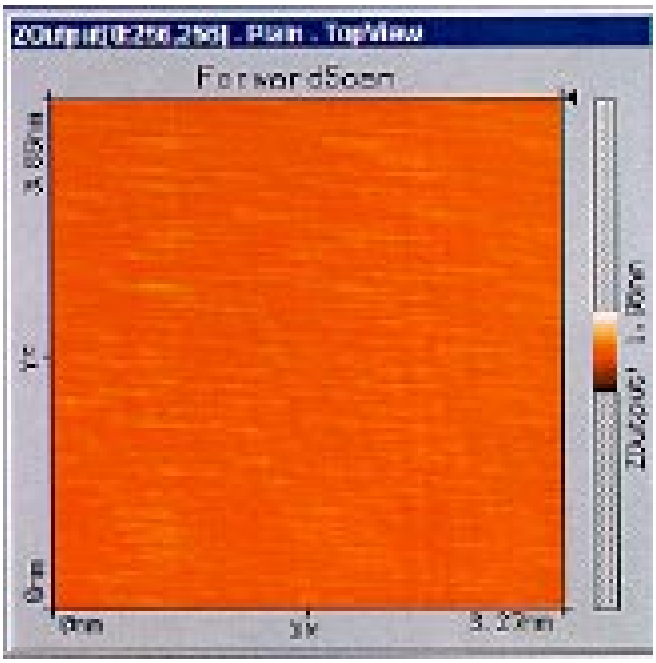


Figure 9

This seems to be only the noise. Also, the surface doesn't seem to be smooth. So no periodic pattern can be seen.

-- Scan --		Date	= 22-04-2000
ScanRange	= 3.27nm	Time	= 09:44:06
Time/Line	= 0.138s	-- Feedback --	
ZRange	= 12.5nm	GapVoltage	= 0.0501V
InputRange	= 12.5nA	SetPoint	= 1nA
XSlope	= -1.92°	IGain	= 13
YSlope	= 1.99°	PGain	= 12
Rotation	= 0°	LoopMode	= Run
XOffset	= -77.5nm	-- Global --	
YOffset	= -37.8nm	ScanHead	= STM
ZOffset	= 9.91nm		


ForwardScan
0nm
3.22nm

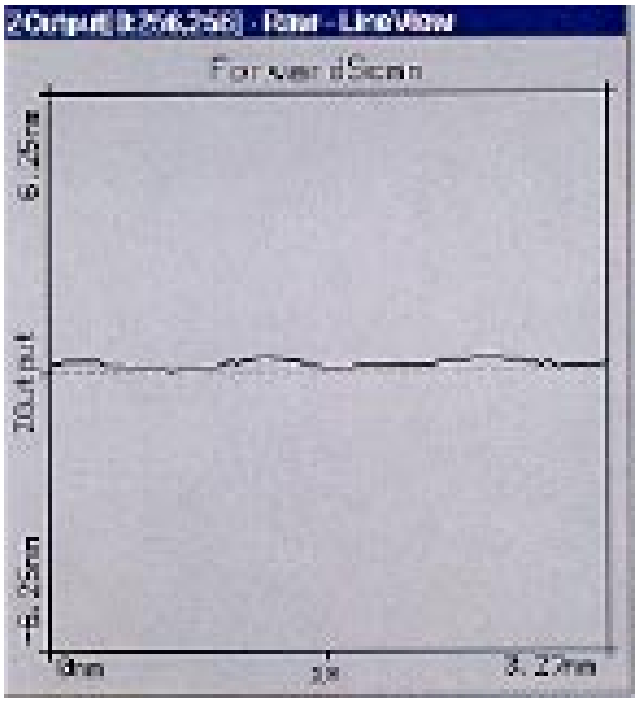

ForwardScan
6.25nm
0.0nput
-6.25nm
0nm
3.22nm

Figure 10

This figure seems that the signal is too smooth to have any information. The possible reason is that the end of the tip is too blunt, so there is no tunneling current at all. The curve might only be the back ground noise.

4. Conclusion

By using the easyScan STM system, we can observe the surface property of graphite in air under room temperature. All the data are collected on the computer and the software can convert the shape of the sample surface simultaneously to image by using the constant current mode.

We got the periodic pattern of graphite, which shows that the side of the hexahedral crystal unit is 0.29 nm. Compared with the theoretical value 0.25 nm, the error is 16%. We believe the error is most due to the limit of the measuring tool (ruler) and the limit of the device. Since we can only measure to mm by ruler, so there might be error on translating the paper distance to the atomic distance. The limit of the resolution of the computer will effect the precision as well.

Measurement on the nanometer scale are very sensitive. Direct light, fast movements causing air flow and temperature variations near the scan head can all influence and disturb the measurement. It is best to let a promising measurement run for some time to let it stabilize thermally.

If temperature variations are present they cause so called “thermal drift”. Thermal drifts are very clearly perceptible on an atomic scale. Variations of 0.1°C in temperature may cause variations in the length of the sample holder to be several nanometers!

If during a good measurement the image quality diminished dramatically the tip is very possible to pick up some particles.

5 Reference

[1] Scanning Tunneling Microscopy I and II. H.-J.Güntherodt & R. Wiesendanger. Springer-Verlag, 1992.
[2] easyScan STM system operation menu, Introduction & Software Reference.