PC2232 Physics for Electrical Engineers
Scanning Tunnelling Microscope

"Imagine a microscope that creates three-dimensional (3D) images down to the atomic scale, that works in air and in liquid as well as in vacuum, that uses a technique for which biological specimens need no staining, and that can map electronic, mechanical, and optical properties, and, moreover, that can manipulate a surface to the level of moving atoms one by one. These are the remarkable capabilities of scanning probe microscopy (SPM)"

— Mervyn Miles

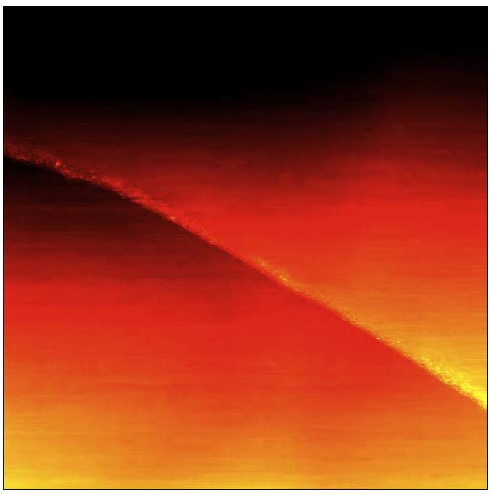


Figure 1: Global imaging with an STM: Graphite Terraces.

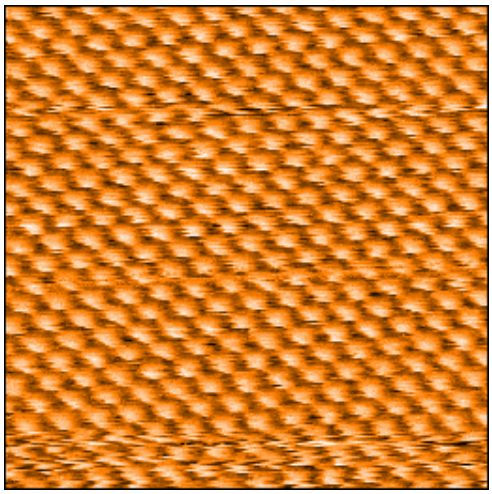


Figure 2: Atomic imaging with a STM: 3 nm x 3 nm scan of HOPG.

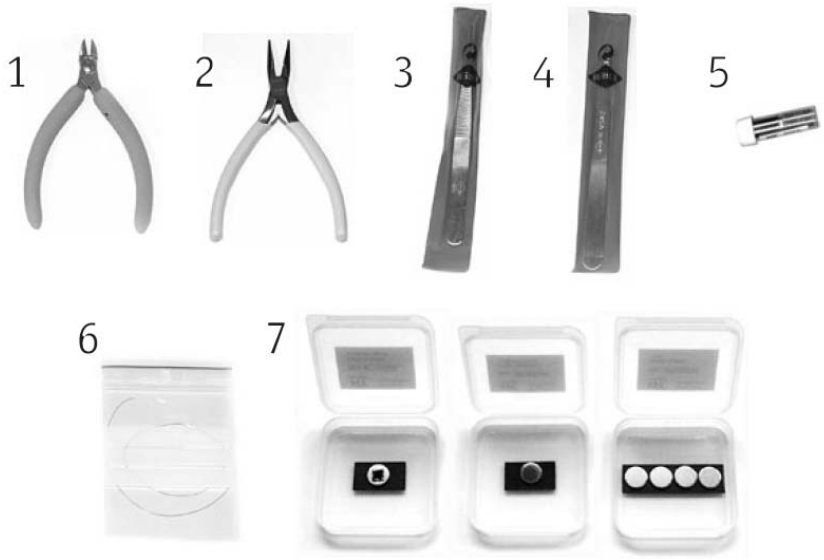
Objectives

- To familiarize oneself with the operation of the STM and associated software.
- To determine the spacing between different layers of atoms in graphite from relevant STM scans.
- To determine the distance between atoms in each layer of atoms in graphite from relevant STM scans.

Instruments & Setup

Figure 3 shows the various components of the *easyScan 2* setup. These are already assembled and ready for your use.

(1) *easyScan 2* controller, **(2)** *easyScan 2* STM Scan Head, **(3)** Magnifying cover with 10× magnifier, **(4)** USB cable (to connect controller to PC), **(5)** Mains cable, **(6)** STM Tool set (see below) and **(10)** Vibration isolation platform



(1) Wire cutters, **(2)** Flat Nose Pliers, **(3)** Pointed tweezers (00D SA), **(4)** Rounded tweezers (2A SA-SL), **(5)** Sample holder, **(6)** Pt/Ir wire: 0.25mm/30cm for making STM tips, **(7)** STM Basic Sample Kit with HOPG, gold thin film and four spare sample supports.

Figure 3: The various components of the *easyScan 2* setup.

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Sample Preparation (if necessary)

The HOPG (graphite) sample may be cleaved so as to obtain a clean surface as shown in Figure 4 and described below:

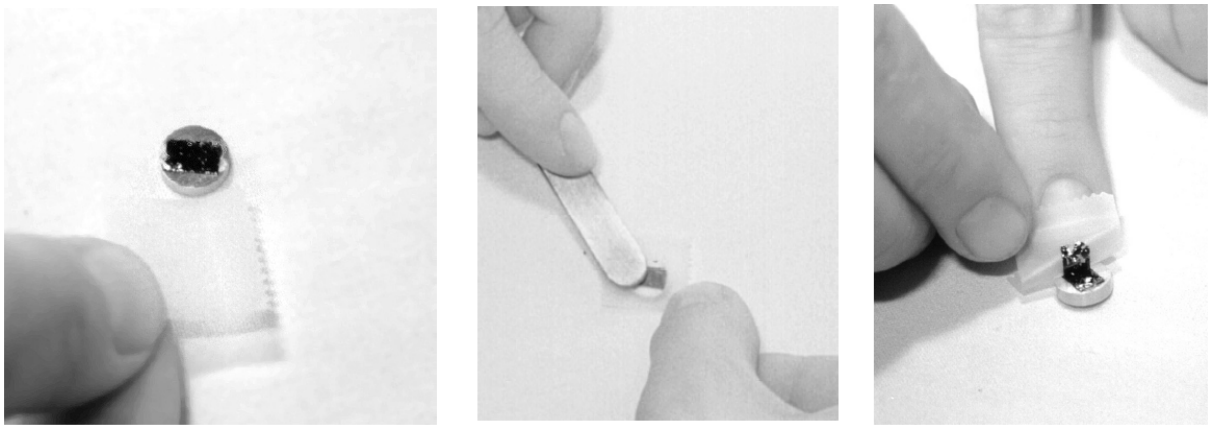

Figure 4: Cleaving the HOPG sample

1. Stick a piece of scotch tape gently to the graphite and then gently press with the back, flat part of the tweezers.
2. Pull the tape off. The topmost layer of the sample should stick to the tape, leaving a freshly exposed graphite surface.
3. Remove any loose flakes with the part of tweezers.

Install Sample (if necessary)

The steps to install the sample is shown (Figure 5)/described below:

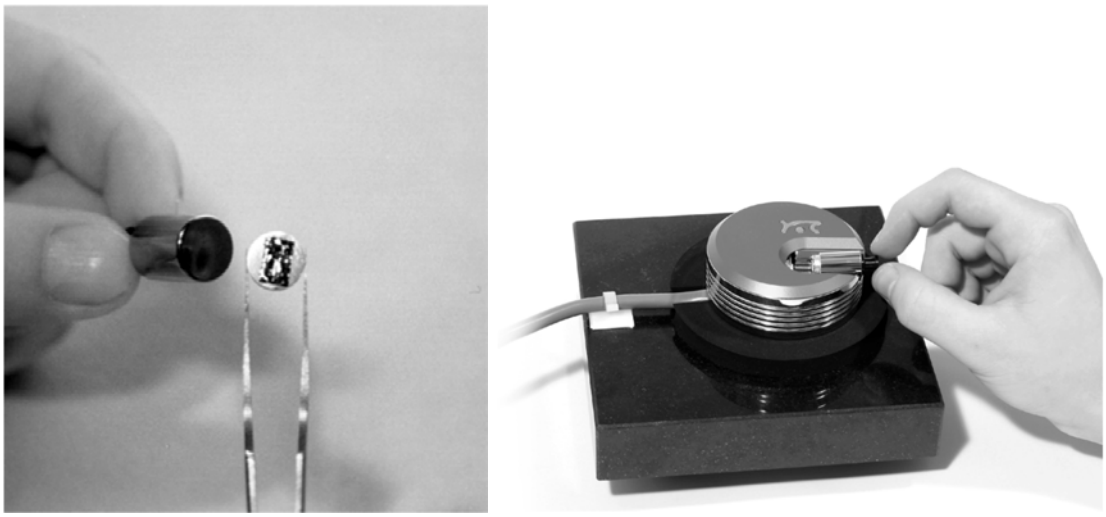

Figure 5: Installing the sample

1. Remove the sample holder from the storage container by holding the black plastic part. DO NOT TOUCH the metal part.

2. Check for any contamination (dust, fingerprint) on the metal part. If cleaning is necessary, follow the cleaning procedure.
   (a) Moisten a cotton swab with ethanol and gently clean the surface.
   (b) Allow the alcohol to completely dry.
3. First place it onto the little rails, then slide it onto the piezo motor. Make sure it does not touch the tip.
4. Using a tweezers, hold the graphite sample at the magnetic part.
5. Take the sample holder (handle at the black plastic part), and place the graphite sample on the magnet.
6. Push the sample holder very carefully to within a few mm of the tip.

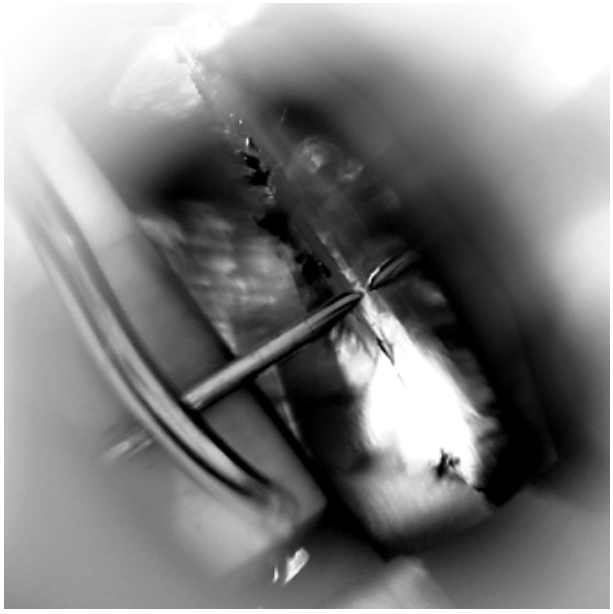

Figure 6: Reflection of the tip off the graphite

Approach & Imaging

1. **Important: The tip must not touch the sample or the tip may be damaged and would need to be replaced and the sample may need to be cleaved again.**
2. Through the magnifier, watch the distance between the tip and sample as you click '**Advance**' in the approach panel. The tip should be within a fraction of a millimetre to the surface. There should be a very small gap between the very end of the tip and the reflection of the end of the tip as can be seen in figure 6.

3. Set control parameters in Z-control panel (if necessary):
    - Set point 1 nA
    - P-gain 1000,
    - I-gain 2000,
    - Tip voltage 50 mV
4. Click '**Approach**'.
5. If the approach was completed successfully, the probe status light (see below) changed from blinking to green and automatically start to scan. This step might take some time depending of how close the the tip was positioned during coarse approach. If the approach was unsuccessful, retract and try again.
6. Once the scanning has begun, you will need to optimise the tip-sample orientation as described next.

The Probe Status Light

The probe status light indicates the status of the Z-feedback loop that is responsible for controlling the tip-sample gap.

RED	Scanner at it's upper limit. Very strong tip-sample interaction. Possibility of a damaged tip.
ORANGE/YELLOW	Scanner at it's lower limit position. Weak tip-sample interaction Tip possibly not in contact with the sample
GREEN	Scanner not at either limit. Conditions good for imaging
BLINKING GREEN	Feedback loop inactive.

Optimising the Image Plane (if necessary)

It is important that the plane of the lateral movement (i.e. the raster plane) of the tip be parallel to the sample (measurement, image) plane. Figure 7 shows this idea. The following are the steps to optimising the image plane.

1. Set the rotation to zero.
2. Observe the line scan. (An unoptimised plane will appear as shown in the figure on the left 8).
3. Change 'x-slope' until the linescan becomes horizontal (figure on the right 8).

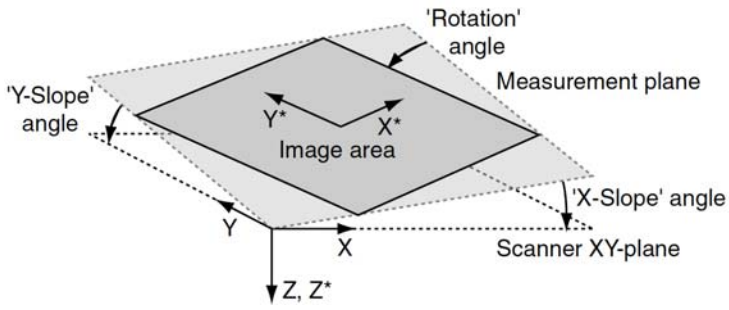

Figure 7: The important planes to keep in mind when using the *easyScan 2*

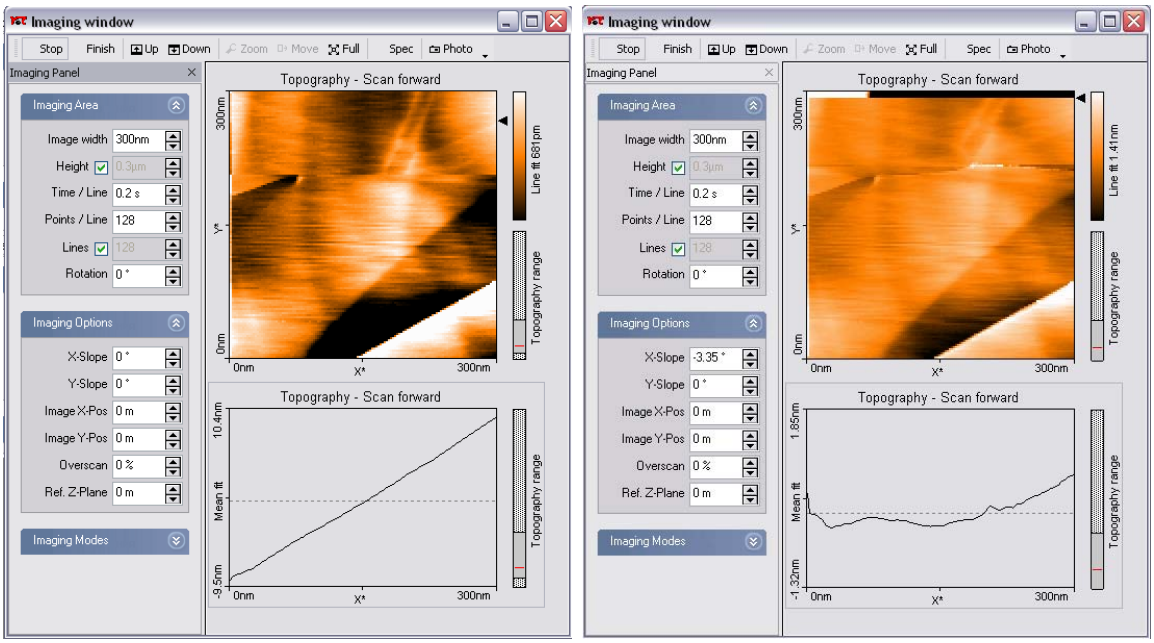

Figure 8: The effect of changing the x-slope. Before (left) and after (right).

4. Now change the rotation to 90° and optimise again, as above, but by changing the 'y-slope'.

Task 1 : Global Imaging - Looking for HOPG Terraces

It is not uncommon for the HOPG sample to be multi-layered so that scans over large areas show 'terraces' as seen in figure 9.

Experiment

1. Obtain an image of a scan area of 500 nm x 500 nm using 256 x 256 pixels.
2. Suggested setting for Time / line is 0.3 s. (However, you may use other settings if that gives you better scans.)

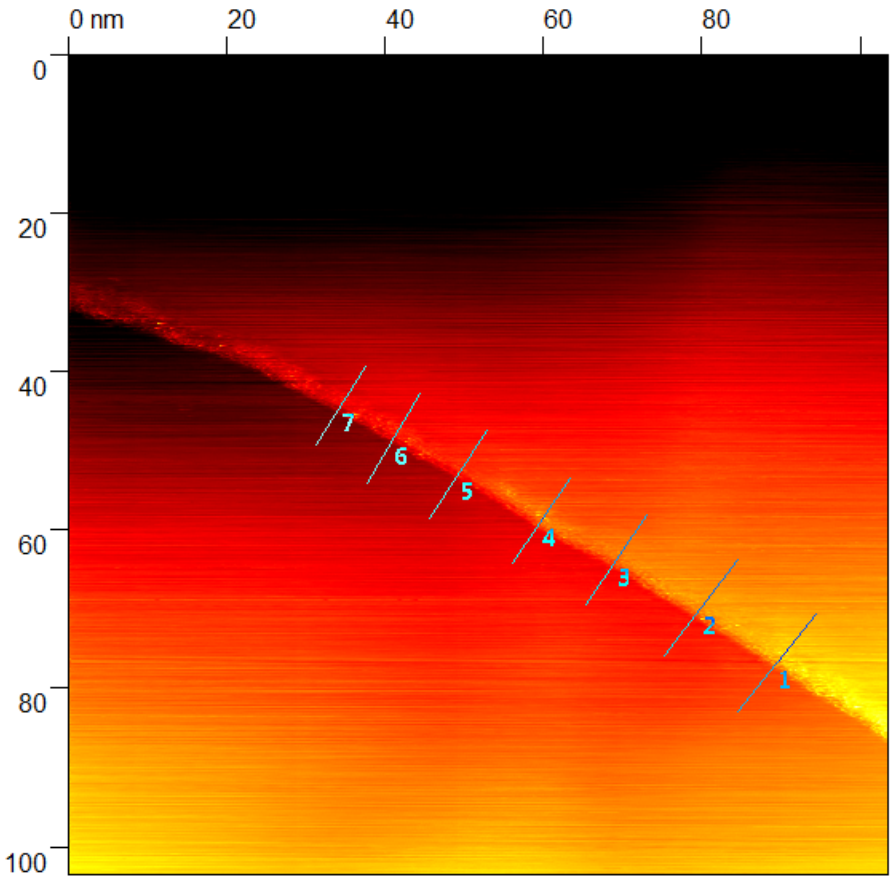

Figure 9: HOPG terrace with lines draw using `Gwyddion` for the extraction of line profiles.

3. Locate a region with a terrace (see figure 9).
4. Reposition and zoom in (to around 100 nm x 100 nm) and obtain a good image.
5. Save as a .nid file.
6. Obtain one more good image (of about 100 nm x 100 nm) of another region of this terrace and save the respective .nid files.

Analysis

1. Open the relevant .nid file in `Gwyddion` . (You may download the software from http://gwyddion.net/)
2. Draw five (5) lines (similar to the one shown in figure 9) at right angles to the terrace step and obtain the profiles. You might want to increase the 'thickness' of the lines to obtain an averaged measurement.
3. Use 'Edge Height' function to extract the step heights for your lines profiles. Figure 10 shows this feature in use.

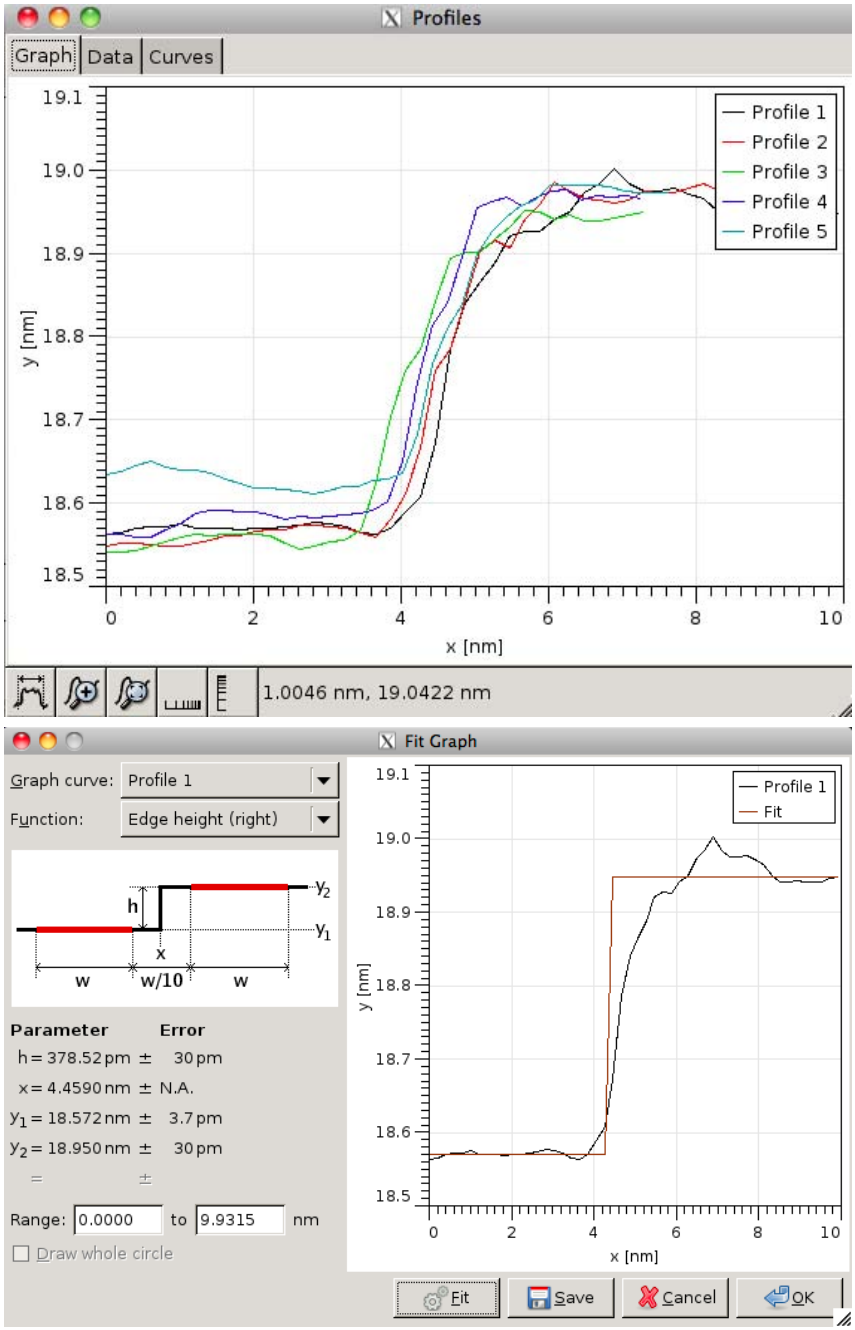

Figure 10: Screenshots of extracting terrace information using `Gwyddion`

4. Save this image to include in your report.

5. Present the data for this measurements in the form of a table similar to the one below.

6. Repeat the above **Experiment and Analysis** for the other images of the terrace and produce a table of the form shown below.

measurement	Terrace Region	
	region 1	region 2
profile 1 profile 2 profile 3 profile 4 profile 5		
average std. dev.		

7. Now add another row to the table that shows values for $\frac{\text{average}}{\text{HOPG layer spacing}}$.

8. Comment on the values of the heights of the terraces obtained with reference to the data given for the HOPG in the theory section.

Task 2: Atomic Imaging

Experiment

1. Obtain an image at a scan area of 500 nm x 500 nm at 256 x 256 pixels.

2. Optimise and image for the scan sizes (bear in mind the pointers in the Hints Section below).

   (a) 250 nm x 250 nm

   (b) 50 nm x 50 nm

   (c) 10 nm x 10 nm

   (d) 4 nm x 4 nm (should be similar to figure 11)

3. Save a *.nid file for each of the above.

Measurement

1. Extract the surface roughness from the screen for each of the images obtained. Present this data in the form of a table as shown below.

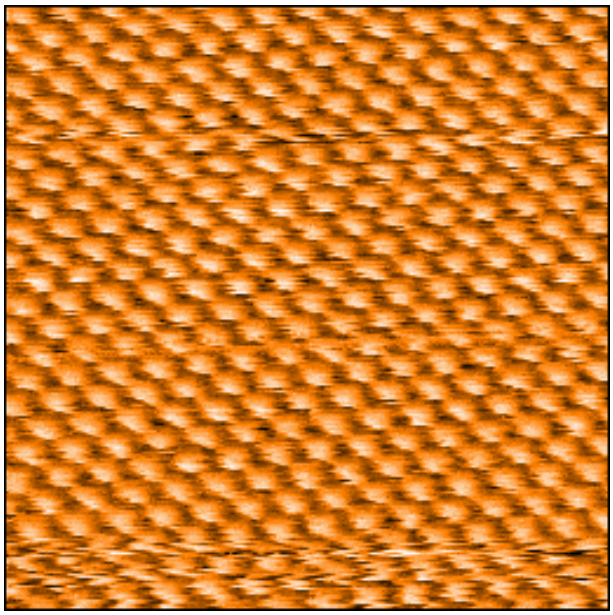

Figure 11: 3 nm x 3 nm scan of HOPG using *easyScan 2* . (Tip voltage: 50 mV, Set point: 1 nA, Time per line: 0.2 s)

Scan size (nm x nm)	Roughness
250 x 250	
50 x 50	
10 x 10	
4 x 4	

2. For the 250 nm x 250 nm and 50 nm x 50 nm scans, it is suggested that you continue to use 0.3 s scan per line. For 10 x 10 nm, reduce to 128 x 128 pixels at 0.03 s / line until the system stabilises. Then increase back to 256 nm x 256 nm. Do the same for 4 nm x 4 nm.

3. Apply appropriate filter, if necessary, to improve the contrast of your images.

4. Use your images to extract:

   (a) lattice constant

   (b) atom-to-atom distance

   Make at least five measurements at different sites and present them in the form of a table as shown below.

	atom-to-atom distance
measurement 1	
measurement 2	
measurement 3	
measurement 4	
measurement 5	
average	
std. dev.	

5. Describe the symmetry that your images of HOPG exhibit? Is this consistent with our understanding of the HOPG? Explain

Shutdown Procedures

1. Wear gloves.
2. Retract the STM tip as far as possible by auto-positioning.
3. Close the *easyScan 2* software window.
4. Leave the STM tip on the head.
5. Take the sample off of the sample holder. Place the sample in its case.
6. Clean the sample holder with ethanol and a cotton swab. Let it dry.
7. Place the sample holder in the case. Close the cap tightly.
8. Place the STM cover over the STM head.
9. Turn off the controller power switch.

Hints & Tricks for better images

Optimize the "I-gain" and "P-gain" These values control error correction in the feedback loop maintaining constant current. A useful strategy is to increase one parameter slowly and watch for spikes to occur in the linescan, indicating that unwanted oscillations are occurring in the feedback loop. Then back off one or two steps until the linescan becomes smooth again.

Speed things up It is essential to apply short scanning times to minimise thermal drift effects. So set **time/line** to as small as possible.

Give it a few scans The piezoelectric drivers can sometimes takes a finite time to settle down when it is started/restarted. So it is best to allow for a couple of scan before acquiring the final image.

Trouble with debris There can be instances where some debris might be attached to the tip which will result in poor results. In such cases you may:

- Retract the tip and press the Cleaning Pulse button in the Tip Properties section of the 'Z-Controller' panel. (This momentarily increases the current and 'burns' the debris off)
- Perform a new approach.

'Rotate' the tip The pull/tear method of producing a tip does not result in a rotationally symmetrical tip. This leads to a variation in the quality of images depending on how the tip is oriented with respect to the raster pattern. This can be understood by the schematic depicted in figure 12.

Overscan When imaging the tip is moved in a left-right raster pattern. This requires the tip to change its direction abruptly at the left and right edges. This sudden change can cause instability in the smooth motion of the tip that can lead to deterioration in the image quality. To avoid this the *easyScan 2* offers the 'overscan' option that allows space on the left and right sides of the scan for the tip to stabilize. These areas are not included in the final image (so your imaged area will appear smaller than that indicated in scan area setting).

Change the value of the overscan to about 10%.

Figure 12: Schematic showing the effect of an asymmetrical tip. Notice that the conditions of (a) will result in a more faithful reproduction of the rectangular 'feature' than those of (b).

Minimise Interference Bright light and movement near the STM setup can interfere with the measurement. Avoid abrupt movements and walking around in the room.

Finetune Image Plane When decreasing the scan range, look at the slope of the individual line scans. Sometimes, they have to be re-adjusted (remember the 90° rotation).

Theory Supplement

Scanning Tunnelling Microscopy

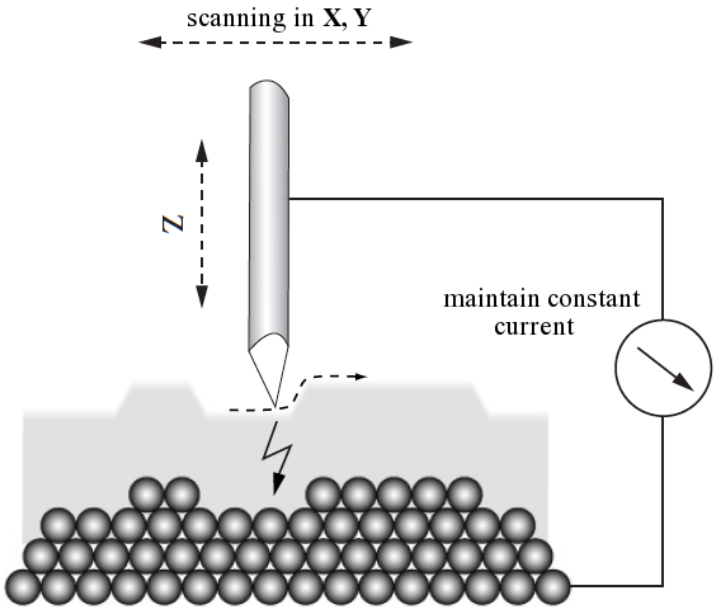

Figure 13: The basic idea involved in scanning tunnelling microscopy. The tip is moved horizontally in a raster pattern while moving the tip vertically so as to maintain a constant tunnelling current.

- The basic idea of scanning tunnelling microscopy, as depicted in figure 13, involves scanning a sharp tip over a surface so as to maintain a given tunnelling current. This is achieved by moving the tip in the horizontal plane in a raster pattern while simultaneously adjusting the vertical tip distance. Owing to the exponential dependance of the tunnelling current on the sample-tip distance this is able to reproduce features of the surface with sub-nanometer resolution.
- In spite of the phenomenon of quantum mechanical tunnelling been known since the 1920s, its use in microscopy was implemented only more recently in 1982, by Binnig *et al* for which the 1986 Nobel Prize in Physics was awarded.
- The most significant feature of STM is the ability for real-space visualisation of surfaces at atomic scales.
- Since STM depends on a tunnelling current its applicability requires a conducting sample. A similar but more universal technique is atomic force microscopy (AFM) which does not have this restrictions.
- STM constant current images provide information about the variations in the electron density and do not necessarily correspond to the location of atomic nuclei. This is further illustrated in figure 14.

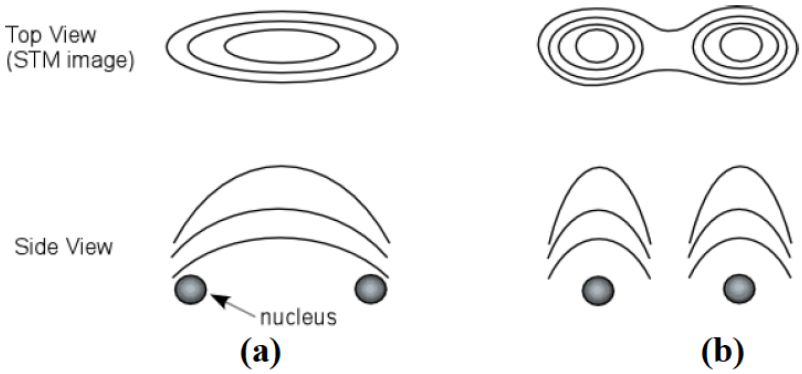

Figure 14: Sketch of possible STM images relative to the nucleus locations. Top view is the contour lines of constant intensity for the STM images that corresponds to the side view at the bottom. The STM image shows high tunnelling location at centre of two nuclei in (a) but at the top of each nucleus in (b).

- Varying STM parameters actually allows us to probe different aspects of certain structures. For instance we may obtain two different images of the same sample by merely changing the polarity of the tip. An example is shown in figure 15 which uses the Si $< 011 >$ surface.

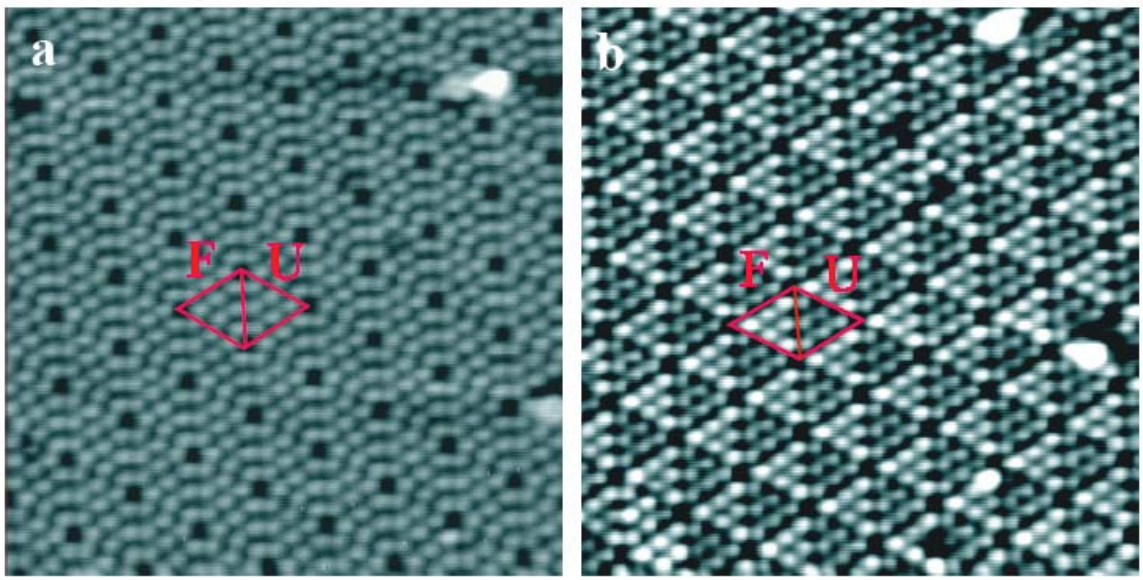

Figure 15: STM images of a Si surface with (a) +1.8 V bias and (b) -1.8 V bias. Source: Lucia Vitali's (l.vitali@fkf.mpg.de) PhD thesis.

Highly Ordered Pyroletic Graphite (HOPG)

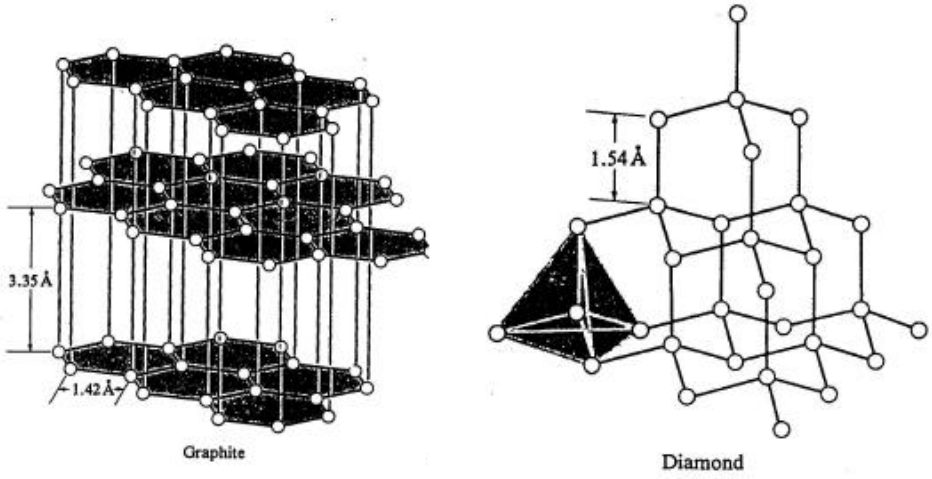

Figure 16: Structure of Graphite (left) and Diamond (right), the two stable allotropes of Carbon

- Carbon may be found in two allotropes: diamond and graphite.The relevant lattices for these two allotropes is shown in figure 16.
- Diamond is an insulator and one of the hardest known materials.
- Graphite, in contrast to diamond, is a electrical conductor and softer and easily flaked.
- Naturally found graphite exhibits an imperfect structure due to plenty of defects and inclusions.
- **Highly Ordered Pyroletic Graphite (HOPG)** is a synthetic form of graphite that exhibits a very high degree of three-dimensional ordering, especially in the alignment of adjacent layers.
- HOPG is an excellent tool for using in SPM as a substrate or calibration standard at atomic levels of resolution. This is an easily renewable material with an extremely smooth surface. It has an ideal atomically flat surface and provides a background with only carbon in the elemental signature thus making results in a featureless background. This is vital for SPM measurements that require uniform, flat, and clean substrates, for samples where elemental analysis is to be done.
- Graphite exhibits (figures 16 and 17) a layered structures with the atoms in a given layer being covalently bonded in a hexagonal lattice. Adjacent layers are held together by weak van der Waals bonds. This is why graphite can work as a good lubricant.
- Graphite has its adjacent layers displaced relative to each other leading to the atoms of a layer either having a neighbour directly below (A atoms) or not (B atoms) as can be observed in figure 17.

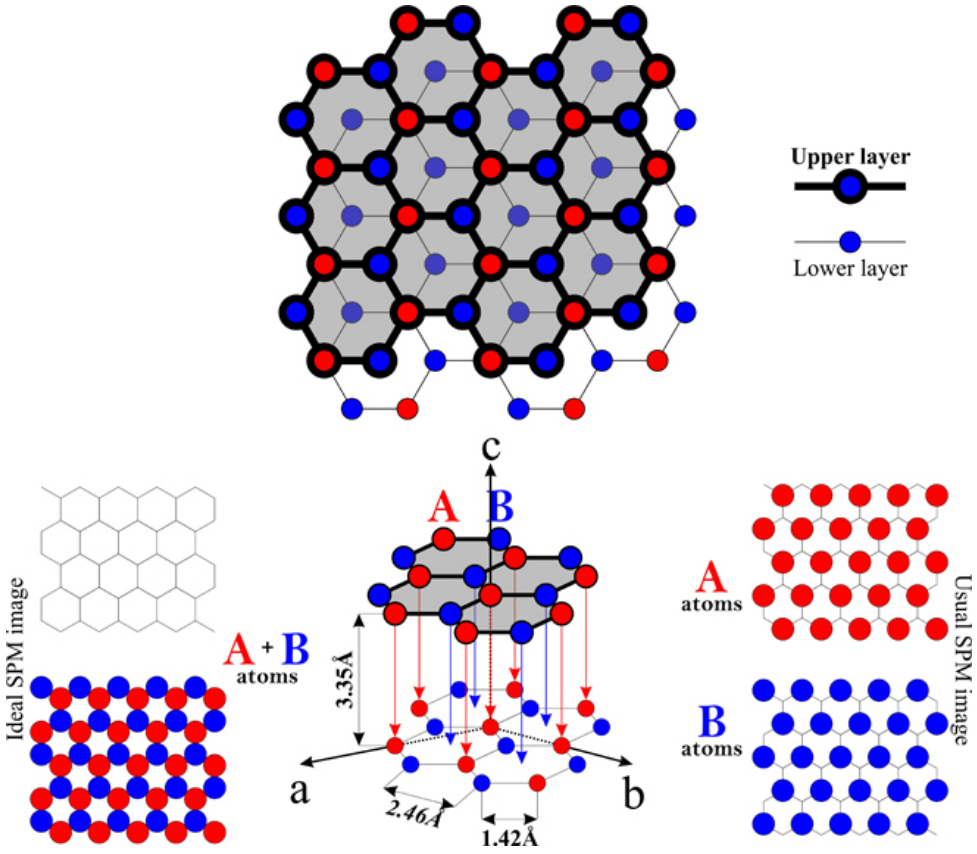

Figure 17: Schematic representation of the structure of the bulk hexagonal graphite crystal. The dashed lines show the axes of bulk unit cell. Side insets: top view of the basal plane of graphite and schematic representation of the surface structure (carbon atoms) of graphite most viewed by SPM. **Right:** Every other atom is enhanced. **Left:** viewed under ideal conditions, where every single atom is seen. (**Source:** http://nanoprobes.aist-nt.com/apps/HOPG%20info.htm)

- This results in the in-plane layout to be such that any A atom lies at the centre of a triangle formed by three B atoms and vice versa.
- Some important lattice lengths:

In-plane A - B separation	2.46 Å
In-plane A - A (or B - B) separation	1.42 Å
Adjacent layer separation	3.35 Å
Layer lattice constant	6.70 Å

STM of HOPG A consequence of the difference between A and B atoms is that SPM of HOPG can result in two different type of images.

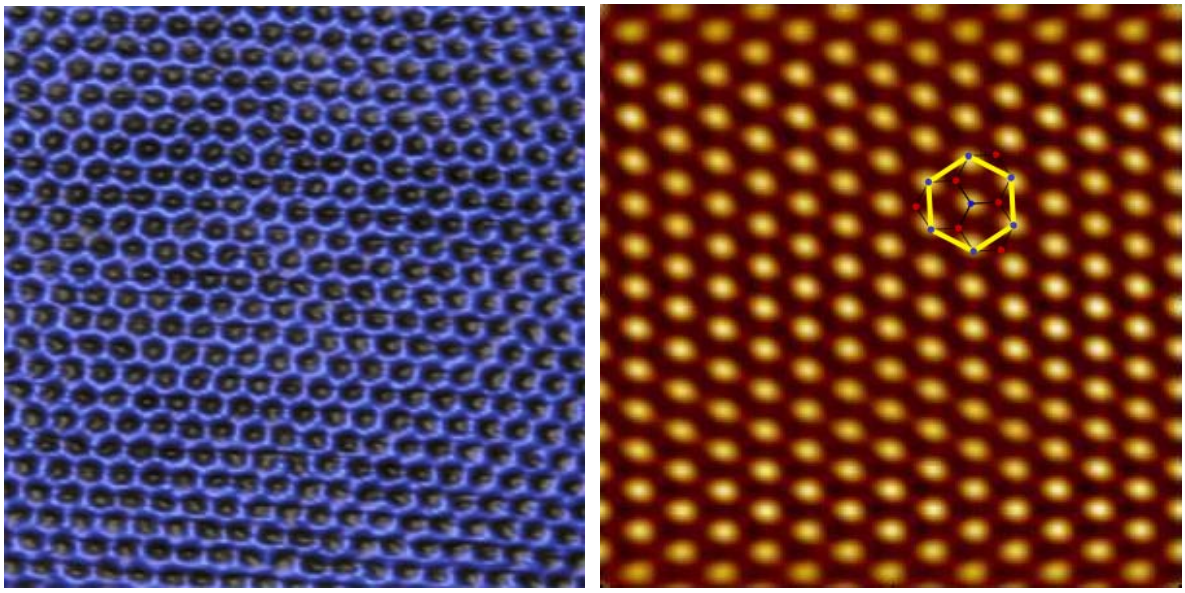

Figure 18: STM of HOPG. **Left:** Symmetric contrast. **Right:** "three-for-six". Compare these images with the schematic of figure 16

Symmetric contrast

- Under ideal conditions, SPM images of HOPG surface reveal a lattice of dark spots with a lattice parameter of 2.46 Å. (Figure 18)
- The six carbon atoms composed in hexagonal ring surrounding each spot give a bright signal, which leads to a true honeycomb atomic pattern (symmetric contrast).
- The center to center atomic distance is 1.42 Å.

"three-for-six"

- The atomic pattern normally observed in most SPM images under usual conditions shows an asymmetric positive contrast in that bright spots originate from only three (the B atoms) of the set of six atoms that form a graphene hexagon unit cell of the graphite lattice.
- Each apparent atom is surrounded by six nearest neighbors. These form the vertices of the yellow hexagon of figure 18 . The distance between any two of these atoms is 2.46 Å.
- This asymmetry in the surface atom environment results in a threefold symmetry ("three-for-six") pattern as observed in the picture on the right of figure 18.