Fortgeschrittenen Praktikum Rastertunnelmikroskopie (engl. Scanning Tunneling Microscopy)

Please read this guideline and consult the referenced textbook and other cited material before starting the experiment. The instrument is delicate and needs careful handling to avoid defects or malfunction / unsuccessful experiments. If in doubt consult with the assistant in charge.

Safety Rules : NO LIQUIDS into the STM scan head and electronics boxes outside the specific containers. Start the experiments by organizing the workbench. Reserve one 'clean' area for tip and sample preparation and the storage of cleaned tools (tweezers, tongues/ pliers, cutters,...) Use gloves for cleaning the tip and the sample and deposit them between the 'clean-handling' workspace and the STM workspace.

1 Tip preparation

To start reliable measurements, a good tip is primal. One gets a clean and sharp tip by the following procedure.

1. Begin by cleaning all tools with ethanol (wire cutter, flat plier, tweezers). Note that cleaning (in particular of grease from touching some of the accessories may need a number of rounds to sufficiently dilute the surface contamination.
2. Grab the wire with the flat plier at the end and cut an about 5 mm long piece. Clean the Pt wire as well with ethanol, from both sides, holding it with the tweezers to do so. (Any grease you deposit on the tip has a bad effect on measurements so be meticulous. It is also advised to clean the tweezers after each use to avoid contamination.) Hold the wire with the flat plier and hold the wire cutter with a sharp (non rectangular angle) to the wire.
3. Softly and slowly apply pressure with the wire cutter to the wire until you feel some resistance.
4. Start pulling along the axis defined by the wire with the slightly closed tweezer grabbing the wire on one end and the tweezers on the other.
5. Increase the pulling force while also increasing the pressure of the cutter onto the wire until the wire breaks. The wire should be more 'pulled' off than pinched off in order to get a sharp enough tip. From now on, the tip must never be touched again ! Grab the wire with the tweezers and place it under the golden spring on the small slit. The tip must not protrude out of the tip holder for more than 2-3 mm beyond the end of the tip holder.

2 Scanning

2.1 Sample preparation

The samples have to be completely cleaned and expose a smooth surface. The surface of Highly Oriented Pyrolytic Graphite (HOPG) can be cleaved (the top surface layer can be removed) with 'Scotch Tape'. You should never touch them directly with your fingers but always use clean tweezers. Do not touch the critical surfaces of the sample, as the grease you have on your fingers will contaminate it and it may result in malfunction or bad quality experiments. Ta ke the clean sample holder and place the sample on the magnetic sample holder. Mount the sample holder into the instrument.

2.2 Approach the sample to the tip

Start the measurement with graphite. The tip must never be mechanically touching the surface as you will blunt it in this case. The LED on the measuring head will give you information on the state of the tunneling junction :

— Orange : no current flows, you are too far away.
— Red : too much current flows, the tip is in contact with the sample –> consider to replace the tip.
— Green : the tip is in tunneling mode and the instrument and table should not be touched.

Rough manual approach :

1. Move carefully the sample holder down to about 1 mm distance to the tip.
2. The tip must be above a good-looking place of the sample. If it is not the case, rotate the sample holder. Place the protective cover.

Step-by-step approach with the piezo motor :

1. Open the 'approach panel'. Observe the tip-sample distance with the loupe and click on the 'down' arrow in approach panel to get closer. Go as close as possible, but you should still be able to clearly see a spacing between the tip and the sample.
2. Once you think you are close enough, open the 'feedback panel'. Check that the 'setpoint' is around 1 nA, 'gap voltage' around 0.05 V, P-Gain is 12 and I-Gain is 13.
3. Click then on 'approach' in the approach panel of the controller to start an automatic approach. If it was successful, the LED will be green and you will get a message stating 'approach done'.
4. It could happen that the tip crashes onto the sample, in this case the LED will be red and you will have to make a new tip. If you crash your tip, think about which parameters could be changed in order to avoid tip crashes.

2.3 Start measuring

Click in 'scan panel' on 'full' to have the maximal measurement range. To begin a scan, click on 'start'. The images of the actual measurement appear as lines in 'line view' and as surfaces in 'top view'. If the lines are bumpy, this means that the measurement contact is bad. Usually, the cause is that the tip quality is bad. In this case, you can stop the measurement clicking on 'stop' and make a new tip. If the line looks like almost a straight line, you can do good measurements.

2.4 Adjust measurement coordinates to the tip

The ideal raster range is on the (x, y) plane of the piezo scanner. The sample is usually a bit inclined with respect to it. As you cannot correct the sample position directly, you change the orientation of the scanning motion by adding a tilted plane i.e. by adding a component to the z voltage of the piezo which is a linear combination of the x and y coordinates like $\Delta z = a * x + b * y$. Change the value for 'x-slope' with the arrow buttons till the measured line is parallel to the x axis. Change 'rotation' to $90^{\leq}$. Change now 'y-slope' until the acquired data is parallel to the horizontal axis on the screen. Change rotation' back to 0 to see the data in the same way as you had started$\circ$.

2.5 Reach atomic resolution for graphite

In the middle of 'line view', you should have now a straight and not too bumpy line. Now you have to reduce the measurement range and amplify the signals to distinguish atoms.

1. Reduce in 'scan panel' the value of 'z-range' to 50 nm. Click in 'top view' display anywhere to be sure that it is activated. Click on zoom.
2. Look in 'top view' for a planar surface and click and drag the mouse to choose your measurement range. In 'tool info panel' you can read the size of the range. Get a range between 30 and 50 nm. Double click on the left mouse button to set this range.

3. You now have to reduce step-by-step the ‘scan range’ down to 4 nm and ‘z-range’ to about 1.5 nm in order to distinguish atomic structures for graphite.
4. You can obviously try other values as well in order to get a feeling of what works best. But always measure and save images noting down in your lab book or in the file name which parameters you are using.
5. Watch out that the height of the signal in ‘line view’ must not be bigger than about one third of the display, otherwise this means that the z-range is too small.
6. As stated before, the parts of the microscope react to really small temperature changes, therefore the scan should but taken as fast as possible. For atomic resolved measurements, set ‘time/line’ to 0.06 s.
7. In ‘top view’ you can control the imaging process. If you observe values that are out of range, open the ‘view panel’ and click in ‘visible input range’ on ‘optimize’.

2.6 Save measured images

To save an image, click during the measurement on ‘photo’. As soon as the scan is finished, a copy of the image is made. Click on the image you want to save to activate it, open the menu ‘file, ‘save as’ and give a nice name to your image. S

2.7 Stop a measurement

Click on ‘stop’. If you want to change the sample, click on ‘withdraw’ in ‘approach panel’ and then move for a few thousand steps with the up arrow to withdraw the tip far enough. If you do not do that, the tip might crash into the sample due to thermal expansion or somebody not careful enough bumping onto the table. Be aware to increase the spacing sufficiently before sliding the sample out.

3 Spectroscopy

With the ‘spectroscopy panel’ you can measure current voltage (I-V) characteristic curves.

— Click on ‘spec’ in ‘scan panel’ while taking an image. What was measured is then copied into the ‘spectroscopy panel’. With ‘point’ or ‘line’ you can pick the coordinates for the spectroscopy measurements with the mouse. When you double click, you confirm what you have picked.
— In the field ‘output’, you can choose between ‘gap voltage’ and ‘z- axis’ depending on which characteristic curve you would like to measure.
— You can start the measurement by clicking on ‘start’. It goes on as following :

1. The tip moves to the chosen start point.
2. The PI controller is switched off.
3. The characteristic curve is measured.
4. The PI controller is switched on again.

— You can change the measurement range in ‘input level’. If the tunnel current leaves the range, the measurement is stopped for safety.
— In case it is interrupted, in ‘ModAborted’ appears in ‘Data info panel’ and in ‘spectroscopy panel’ a sign “ !” appears.

4 Lattice Constant measurement

There are two different atomic positions for the atoms in the first layer of the crystal lattice. One part of the atoms has an underlying atom, the other has not. At the high currents (1 nA) used in STM for easily visualizing the atomic corrugation, the tip is exerting a force. We will detect a high current (large overlap of electronic wave functions) for the atoms sitting on top of another atom and a low current (less overlap of electronic wave functions) for the other atoms. This means that the 'conductivity' of the atoms at the surface varies slightly with respect to the exact site. Therefore, one finds for the lattice constant of graphite a too high value of 0.25 nm if one measures the distance between two spheres instead of the actual one of 0.14 nm. Search the literature and find a good figure to explain this phenomen to be included (with proper citation) in your report. How long did it take scientist to realize that they had initially not been looking at the atomic corrugation ?

Determine the lattice constant as follows :

— In the menu 'tools' choose 'measure length'.

— Click, drag and let go the left mouse button to draw a double arrow.

— The length of this arrow is shown in 'tool info panel'. The value you will measure will probably not agree with the one you expect. This is due to the fact that the STM is not calibrated. Use the calibration sample for that purpose. Scan it exactly as you have done for graphite and measure the lattice constant of the calibration sample. Compare the value you get with the value it actually is (160 nm) to get the calibrated value of your measurement of the graphite lattice constant.

Questions

1. Acquire STM overview image
2. Get atomic resolution and determine the lattice constant
3. Measure a IV and IZ characteristic and explain the results

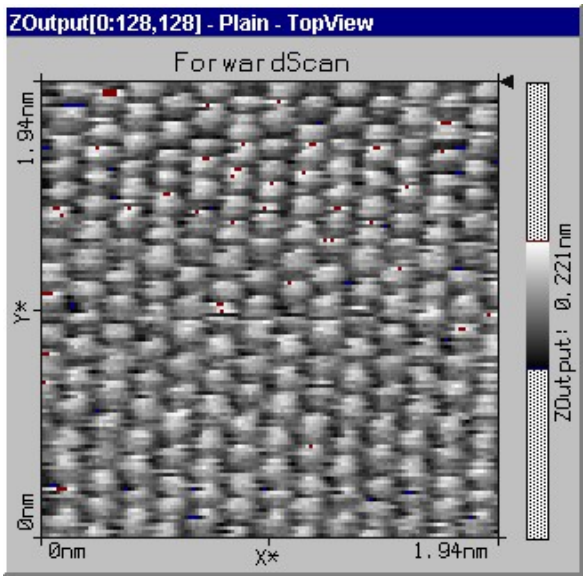

FIGURE 1 – Graphite.

Références

[1] H. Rohrer, R.J. Behm, N. Garcia, "Scanning Tunneling Microscopy and Related Methods", 1989.

[2] ETH STM Operation Manual

[3] K. Oura, V.G. Lifschitz, et al., Chapter 7 "Surface Science An Introduction" 2003