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Manual number:

Physikpraktikum für Vorgerückte (VP)
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Rastertunnelmikroskop (RTM)
Scanning Tunneling Microscope (STM)

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November 19, 2019

Contents

1 Theory

As you already know, matter is made of atoms. There are nowadays some ways of detecting single atoms and investigating their behavior. TEM (transmission electron microscopy), FIM (field ion microscope), optical near-filed microscopy and SPM (scanning probe microscopy) that has many different applications. The historical origin of SPM is the STM (scanning tunneling microscope) that was developed by Gerd Binnig and Heinrich Rohrer in the research lab of IBM in Rüschlikon and for which they got the Nobel price in 1986.

1.1 Operating mode

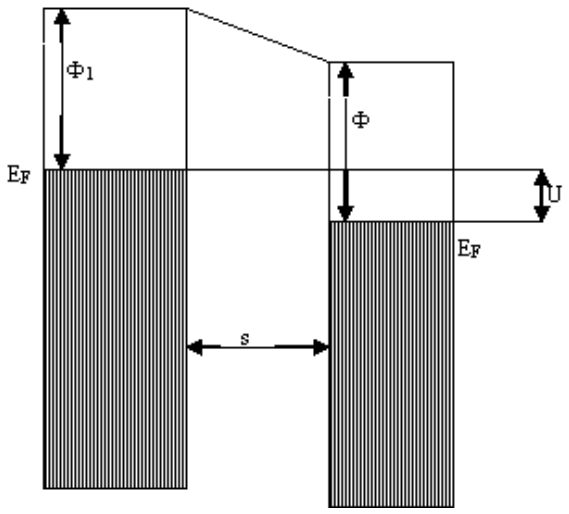

Figure 1: Schematic representation of a potential barrier.

STM operates by approaching a sharp tip really close to the surface of interest. The tip then records signals that are sent to a computer which treats it and produces an image on the computer screen. The principle of STM

is based on the tunnel effect, a quantum mechanical effect which happens for example between two conductors separated by an isolating layer. While classical physics would not allow it for all energies, there is actually a finite probability that an electron goes from on conductor to the other even if its energy is smaller than the energy gap. In a metal, the conducting electrons fill the conducting band up to a limit, defined by means of the Fermi energy E_F. The electrons having the energy E_F possess the lowest binding energy to the metal.
The system formed by the two conductors and the insulating gap forms a potential barrier. From quantum mechanics it is known that electrons having an energy about E_F can tunnel through the insulating gap with a probability proportional to $\exp^{-\alpha \cdot s}$ where s is the separation between both metals and α is a quantity depending on the height of the barrier (in the one-dimension case).

In an STM measurement, a metallic sharp tip is approached really close to the sample of interest and a small tension U is set between both. The spacing between the tip and the sample (air in our case but it could be vacuum or even other media) acts as the insulator. Thanks to the tension set, a potential barrier described above is formed and tunnel processes happen between the metallic probe and the tip. This electron movement corresponds to a current, the tunnel current I, which is proportional to the tunneling probability. It writes

$$I \propto U \cdot \exp(-A \cdot \sqrt{\Phi} \cdot s) \tag{1}$$

with Φ being the effective height of the barrier, s is the tip-sample distance, U the tension and A a constant which for a vacuum gap writes

$$A \approx 1.025(\mathrm{eV})^{(-1/2)}(\mathrm{\AA})^{(-1)}. \tag{2}$$

The sensitive dependence of the current on the distance is the foundation of an STM measurement. One method of measuring is to keep the tunnel current constant as a feedback mechanism. In addition, the tunnel current depends as well on the density of state of the sample. For example, electrons in a metal having an energy close to the Fermi energy will give the biggest contribution to the tunnel current. They "feel" the smallest barrier height and cross over to states in the metal on the other side of the potential barrier with energy $E_{F,2} + V$. This dependence is the foundation of STS, scanning tunneling spectroscopy which studies the electronic properties of a sample.

The STM needs sharp tips to scan with a good resolution. There are some methods to produce sharp tips but as you will learn in this experiment, a really simple process can already be enough to get a single atom at the tip. As written before, the insulating medium between the sample and the tip can be air, vacuum or even a liquid. Any change in height of the surface of the sample translate to a different tip-sample distance which corresponds to a different tunnel current. As this current depends exponentially on this distance, even changes in the range of less than 1 pm can be measured. Therefore, a topographical image of the surface in the range of atomic resolution of sample can be recorded in principle.

To displace the tip in order to get topographical images, piezo crystals are used. They can move the tip in three axes, commonly denominated as (x, y) for the movement over the samples surface (scanning) and z which is perpendicular to the samples surface which is used to control the distance tip-sample.

1.2 Different scan methods

There are different methods to scan, for which different sample properties can be studied.

a) Topographical methods

Constant Current Mode: in this case, the current is set to be constant. Either the tension U or the tip-sample distance s are varied. One gets then a map $U(x, y)$ or $s(x, y)$ which directly gives the topography of the sample studied.
The feedback loop works as following: whenever the tip goes above an atom, the tunnel current will change. In order to get the current back to its set value, U or s will be changed, i.e. a different tension will be set or the tip will be approached or withdrawn. Note that in reality, this measurement method does not give in all case the exact topography of the sample as the tunnel

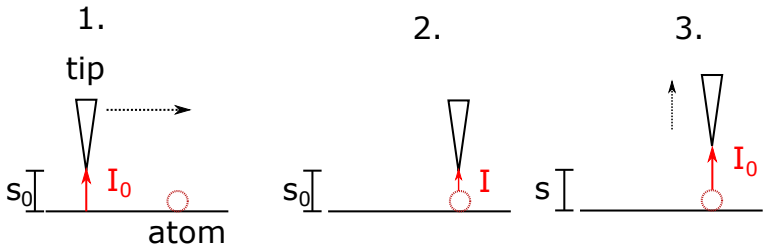

Figure 2: Principle of constant current mode. 1. The tip is at an initial distance s_0 of the surface and the current reads I_0. This current is set as the constant current value. 2. The tip moves above an atom, the tunnel current will change. 3. The feedback loop adjust the tip-sample distance in order to recover the tunnel current set to be I_0.

current depends as well on the density of state of the electrons.

Constant Height Mode: in this case the tip-sample distance and the tension are not varied, the tunnel current variation is directly measured when moving the tip above the surface. One gets this time a map I(x,y) that can be then translated into the topographical map of the sample.

b) Spectroscopical methods

In this case, one is interested about $U(x, y)$ from which electronic properties of the sample can be derived.

Constant Resistance Mode: a slow sweep of U over time ($U(t)$) is superposed to a fast alternating voltage with a frequency above the cut-off frequency of the lowpass of the feedback loop. The current$I(t)$ will be change, so that the resistance $R(t) = U(t)/I(t)$ and therefore for small $U(t)$ the tip-sample distance s stays constant. What is measured is dI/dU as function of the tension U. This gives information about the density of state, as only electrons between E_F and $E_F - U$ can tunnel. With this method, it is possible to observed absorbed molecules as well. In the curve dI/dU

typical peaks appear corresponding to defined bonds corresponding to a certain tension value U. If this certain U is set between the tip and the sample and the curve dI/dU is measured, the obtained peaks yield the adsorption species. The spectroscopic methods using fast tension sweeps have the advantage that the information is not deformed by the tip-sample distance.

Work function profil: in this case the tip-sample distance s is measured as function of the tension U for a constant current I or the current I is measured as function of the tip-sample distance s keeping the tension constant. From this measurement, information is obtained about the effective barrier height.

1.3 Resolution capability

The resolution capability of the STM depends in general on how sharp the tip is because it has an influence on how the surface can be scanned. It is important as well, how stable the tip-sample distance can be kept. More reference can be found for example reading [Stoll, "Resolution of the STM", Surf. Sci. 143, L411 (1984))] or [Tersoff, "Resolution in Scanning Tunneling Microscopy", Springer (1988)].

1.4 Application of the STM

- Investigation of the surface of metals and semiconductors: regular structure, defects, favored sites for the adsorption of molecules, etc.
- Atomic Force Microscope (AFM). In this case, the tip is moved above the surface and the atomic force between the tip and the surface atoms cause it to bend. Measuring this tip displacement gives direct information on the surface topography. This has the advantage that non-conducting samples can be measured as well.
- Spectroscopy: electronic band structure, surface states.
- Investigation of biological samples (viruses, DNA) after depositing a thin conducting film that does have an untextured surface.
- Chemical applications: investigation of the adsorped molecules of a surface.

- Modification of the surface at defined sites with help of high electrical fields at the tip or directly with help of the tip. It is possible even possible to move around single atoms on the surface in this case.
- Observation of the growing of structures on the surface, metal film deposition for example.

1.5 Tip fabrication

The method used in this experiment to produce tip is not used in research. It is roughly speaking cutting a metallic wire in a certain way to obtain a sharp tip but is obviously not completely optimal for daily use and does not gives best tips qualities. A rather simple but efficient way is the tip etching, which can be made on a tungsten wire. The etching take place in a 5% natron base with an alternating current at about 4 V.

2 Setup of the STM

2.1 easyScan STM system

This STM is a comercially available product. Measurements can be made in air and at room temperature. The microscope is operated with help of a computer software. The tip is held in place on a platform that can be move in three axes by piezo crystals in the nanometer range. The samples are placed on a sample holder that can be approached down to 1 nm to the tip. The tip scans then over the surface, which means that parallel lines are taken line-by-line.

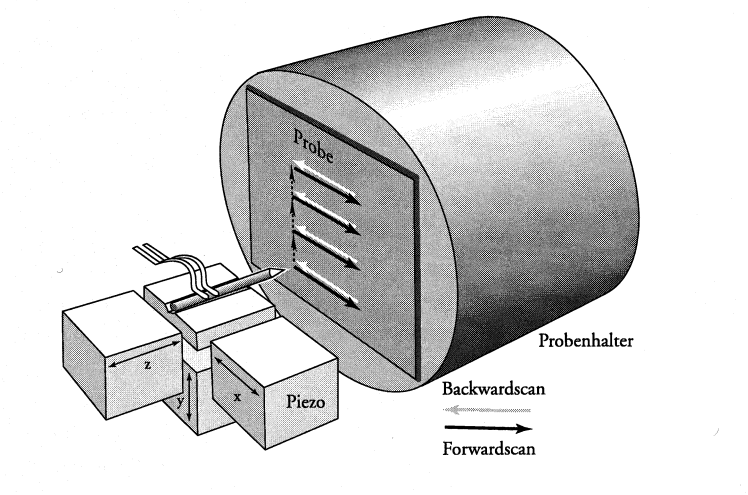

Figure 3: Schematics of the easyScan STM. (Probe=sample, Probenhalter=sample holder)

2.2 Wiring diagram

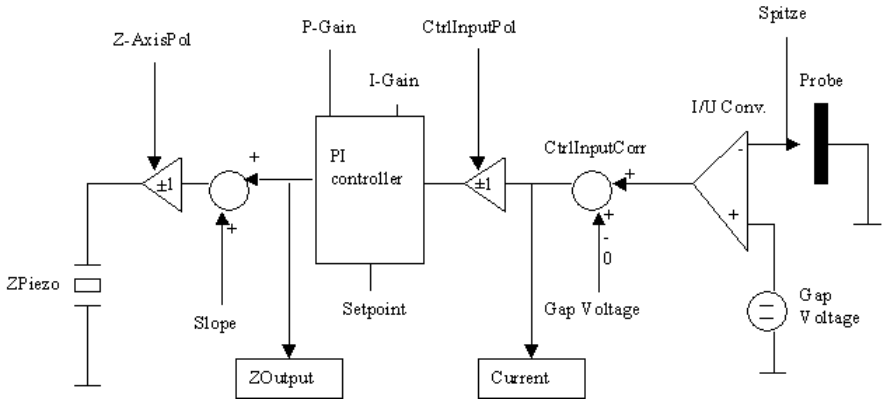

Figure 4: Wiring diagram of the easyScan

2.3 Operating mode of the PI controller

The task of a PI controller consists in bringing a physical value X to a setpoint S and then in keeping it at this set value. Therefore, the controller must work against the different perturbations. The controller changes X with help of the correcting variable Y, so that the deviation $S - X$ is as small as possible. The perturbations are formally denominated by Z, which are additionally added to Y.
Such a controller is in its simplest form an amplifier, which amplifies the deviation $S - X$. If $X > S$ then $S - X < 0$ and Y has to decrease. This decrease acts again the increase of X. This is therefore a negative feedback. In steady state, the bigger the amplification, the smaller residual deviation is.
Our device uses a PI controller to regulate the extension of the z-piezo. A PI controller consists of a proportional amplifier and an integrator, connected in parallel. The proportional amplifier is a linear amplifier. If this amplification is increase, to get a smaller deviation $S-X$, then the transient response gets worse. Therefore, an integrator is connected in parallel. This PI controller behaves like an integrator for low frequencies and like a proportional amplifier for high frequencies.

2.4 Error sources

The following causes act negatively on the quality of a measurement:

Tip shape: the blunter the tip, the more limited is the resolution capability. In this case, it is even possible that the tip actually consists in few small tips that obviously reduce the resolution.
The tip length plays a role as well. It should be as short as possible because it can start to vibrate when moved by the piezo crystals. It could be compensated by moving the tip, waiting for it to be at rest and then measuring but if you would like to get a fast scan of 512×512 points, this waiting time can increase the scanning time to an unreasonable way.
If an oxide layer forms on the tip, it acts as a so-called tunnel resistance connected in series. The PI controller must approach the tip more and more to decrease the resistance. This leads eventually to the tip crashing onto the sample surface.

Mechanical resonances: when moving around the tip that is only a few nanometers away from the surface, externally induced vibrations have a strong influence on the quality of the image. The STM must therefore be isolated as much as possible from those external vibrations. The table where it stays should for example not be touched and the cover must be at all-time placed while measuring, but loud talking or closing the lab door not carefully can be seen as well on the images. Depending on the location on the lab and the quality of the device, trucks passing on a street nearby can be observed as well!

Thermal perturbation: if the STM is not in thermal equilibrium, some expansion or contraction of the metallic parts occurs. The temperature variations have a quite strong effect on the atomic scale and variations of 0.1 °C induce already an expansion of the sample holder of a few nanometers for example. Therefore, sun light exposition should be avoided for example.

3 Assignment

3.1 Scan of graphite, gold a semiconductor

Scan the surface of the samples of graphite and gold. Try to obtain a picture with atomic resolution for graphite. For gold, atomic layer can be visualized. Finally, try to scan the semiconductor sample.

3.2 Influence of the parameters on the images

The feedback panel enables to modify the quality of images. Investigate the effect of varying the following parameters: tunnel current (setpoint), applied tension (gap voltage) and the inputs of the PI controller (P-Gain, I-Gain).

3.3 I-V- and I-s- characteristic curves of graphite, gold and semiconductor

Measure the current-tension- and current-distance characteristic curves of the graphite, gold and semiconductor probes.

4 Instructions

4.1 Tip preparation

In order to get good measurements, a good tip is primordial. The tip preparation needs some exercise and patience, before one gets the first usable tip.

1. Begin by cleaning all tools with ethanol or IPA. (wire cutter, flat plier, tweezers)

2. Rule of the thumb for tools is that every time you use them, you should always assume they are not clean unless you did it yourself. This holds as well if you have left the lab for longer time (overnight etc.).

3. Grab the wire with the flat plier at the end and cut an about 5 mm long piece.

4. Clean the Pt wire as well with IPA, from both sides, holding it with the tweezer to do so. (Any grease you deposit on the tip has a bad effect on measurements so try to get the cleanest possible wire. Cleaning the tweezer after having held one side of the wire could be a good precaution to take as well.)

5. Hold the wire with the flat plier and place the wire cutter at the end as diagonally as you can.

6. Apply slowly pressure with the wire cutter till you feel the wire in between.

7. Apply more pressure and pull the wire from both sides equally till it breaks. It must be more broken than pinched off in order to get a sharp enough tip. From now on, the tip must not be touched again!

8. Grab the wire now with the tweezer and place it under the golden spring on the small slit. The tip must not go longer than 2-3 mm after the tip holder end.

4.2 Scanning

4.2.1 Sample preparation

The sample have to be completely clean and have a smooth surface. Your samples cannot be cleaned with what you have. Therefore, you should never touch them directly with your fingers but always use clean tweezers.
Take now the sample holder (not on the metallic part as the grease you have on your fingers will contaminate it and it will not be possible anymore to move it with the piezo) and place the sample on the magnetic surface of the sample holder. Place now carefully the sample holder on its place.

4.2.2 Approach the sample to the tip

Start the measurement with graphite. The tip must never be in direct contact with the surface as you will blunt it in this case. But it must be really close as well in order to do a successful approach and so that a tunnel current can flow. The LED on the measuring head will give you information on that:

- Orange: not current flows, you are too far away.
- Red: too much current flows, the tip is in contact with the sample.
- Green: the tip is in the measurement range.

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.
3. Place the protective cover.

Step-by-step approach with the piezo motor:

1. Open the "approach panel".
2. Observe the tip-sample distance with the loupe and click on the "down" arrow in approach panel to get closer.

3. Go as close as possible, but you should still be able to clearly see a spacing between the tip and the sample.

4. Once you think you are close enough (rule of the thumb: it is always better to do an unsuccessful approach starting from too far away than crashing your tip onto the sample. Think about that if you do so, you would have to make a new tip.), open the "feedback panel".

5. Check that the "setpoint" is around 1 nA, "gap voltage" around 0.05 V, P-Gain is 12 and I-Gain is 13.

6. Click then on "approach" in the approach panel to start an automatic approach.

7. If it was successful, the LED will be green and you will get a message stating "approach done".

8. 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.

9. If you crash your tip, think about which parameters could be change in order to avoid that.

4.2.3 Start measuring

1. Click in "scan panel" on "full" to have the maximal measurement range.

2. To begin a scan, click on "start".

3. The images of the actual measurement appear as lines in "line view" and as surfaces in "top view".

4. 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.

5. If the line looks like almost a straight line, you can do good measurements.

6. If you have a bad tip but can still resolve some patterns on the surface, try to make a full scan to compare afterwards with a nice tip.

4.2.4 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 through the software the coordinate system so that it seems that the sample is on this (x, y) plane:

1. Change the value for "x-slope" with the arrow buttons till the measured line is parallel to the x axis.
2. Change "rotation" to 90°.
3. Change now "y-slope" to it is parallel to the axis.
4. Change "rotation" back to 0°.

4.2.5 Reach atomic resolution for graphite

In the middle of "line view", you should have now a straight and not 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.
2. Click in "top view" display anywhere to be sure that it is activated.
3. Click on "zoom".
4. 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.
5. 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.
6. 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. (Rule of the thumb: the more parameters you write the better it is. It happens often that two weeks after measuring something, you realize you

forgot to note down one crucial value, which makes your measurements useless.)

7. 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.

8. As stated before, the parts of the microscope react to really small temperature change, the scan should but taken as fast as possible. For atomic resolved measurements, set "time/line" to 0.06 s.

9. 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".

4.2.6 Save measured images

When you want 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. (Rule of the thumb: again, the more parameters you write the better it is.)

4.2.7 Stop a measurement

Click on "stop". If you want to leave the lab for a while or 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, you tip might crash into the sample due to thermal expansion or somebody not careful enough bumping onto the table. (It should not be the case but if you have a nice tip or a nice sample, it is better to stay on the safe side.)

4.2.8 Interpret images and determine the lattice constant

The software offers different tools to interpret the images. In figure 5 you can see a pattern that looks like a three dimensional image of spheres; this is not single atoms. To interpret the image, think about the lattice of graphite: it consists on regular hexagons in one plane. Such planes are stacked to give an hcp structure. There are as well two different carbon sites in the

crystal lattice, one with a neighbor and one without. This means that the "conductivity" of the atoms at the surface varies slightly with respect to the site and the atoms without a neighbor look like they are slightly above the rest. 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.

Determine the lattice constant:

1. In the menu "tools" choose "measure length".
2. Click, drag and let go the left mouse button to draw a double arrow.
3. 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.

4.2.9 Measure gold

It is more difficult to measure gold. The free electron on the surface are really evenly distributed, therefore the measurement signals are not really distinguishable. It is quite rare that you can see atomic structures but you can distinguish atomic layers of gold.

1. Compared to the measurement process for graphite, choose a gap voltage between 0.3 and 0.5 V.
2. Change the time/line to0.3 s.
3. Move around the sample to a cleaner site if your tip is good but you get bumpy lines.
4. When lines are not bumpy anymore, reduce the z-range to 50 nm and choose a scan range between 200 and 300 nm.
5. Reduce then the z-range down to 12 nm or even lower.

4.2.10 Measure the semiconductor

It is basically the same procedure, but you might have to play a bit more with the feedback parameters.

4.3 Spectroscopy

With the "spectroscopy panel" you can measure I-U and I-s characteristic curves.

1. Click on "spec" in "scan panel" while taking an image. What was measured is then copied in the "spectroscopy panel".
2. 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.
3. In the field "output", you can choose between "gap voltage" and "z-axis" depending on which characteristic curve you would like to measure.
4. You can start the measurement clicking on "start". It goes on as following:
5. The tip moves to the chosen start point.
6. The PI controller is switched off.
7. The characteristic curve is measured.
8. The PI controller is switched on again.
9. You can change the measurement range in "input level". If the tunnel current leaves the range, the measurement is stopped for safety.
10. In case it is interrupted, in "ModAborted" appears in "Data info panel" and in "spectroscopy panel" a sign "!" appears.
11. With the I-V characteristic curve, you can observe for example for gold the Ohm law.
12. With the I-s curve, you can observe the exponential dependence of the current on the distance.

5 Examples of good images

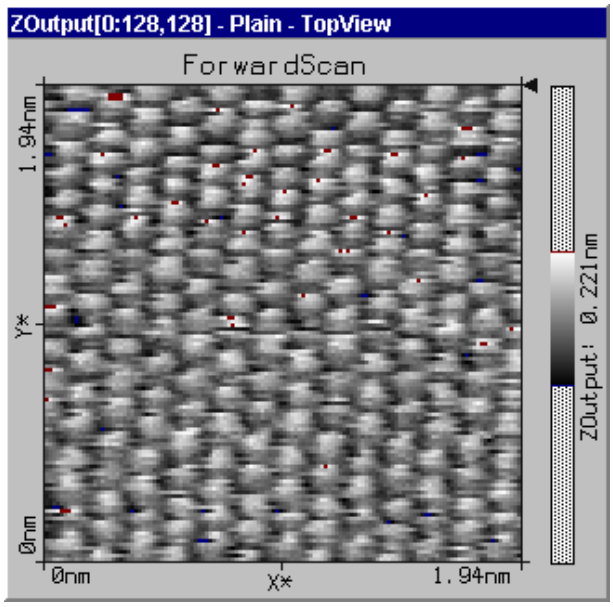

Figure 5: Graphite

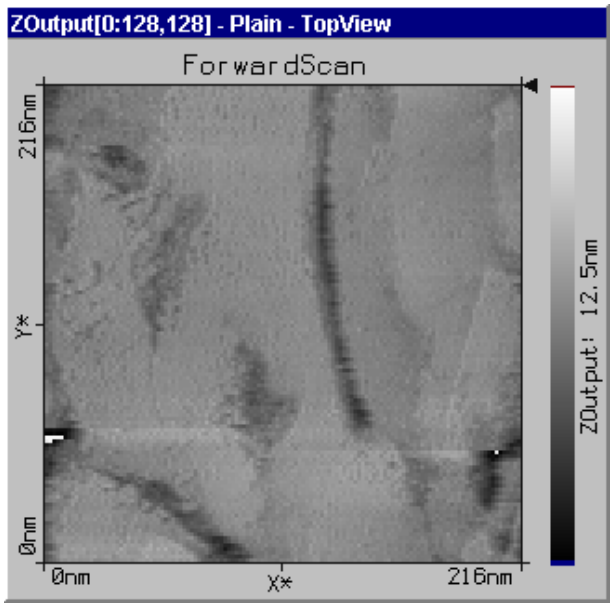

Figure 6: Gold

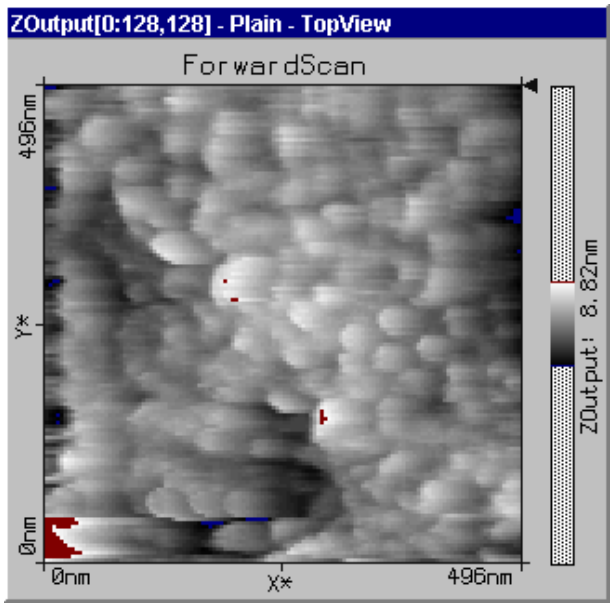

Figure 7: Semiconductor

6 Literature

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There is of course a lot of books about STM. All books cited here are available in the Physikbibliothek. The last book can be found as well in the Praktikumsbibliothek.