Scanning Tunneling Microscopy (STM)

Measurement Exercise for Physicist-Engineer Students

Contents

Practical Information

Energiatudományi Kutatóközpont

HUN-REN Magyar Kutatási Hálózat

Host Institution of the Lab Practice: Nanostructures Department, Institute of Technical Physics and Materials Science (MFA), HUN-REN Centre for Energy Research (EK), Hungarian Research Network (HUN-REN)

Location: KFKI Campus (Csillebérc), Budapest XII., Konkoly-Thege Miklós út 29-33., Building 26, Ground Floor, Lab No. 25.

How to get there: From the northern part of the university: tram 56 or 56A to Széll Kálmán Square, then bus 221 to the final stop. From the southern part of the university: bus 212 to Svábhegy, then bus 221 to the final stop; or bus 212B directly from the southern part of the university.

Lab Instructors: *Kandrai Konrád* (kandrai.konrad@ek.hun-ren.hu, phone: +36-20-5149779)
Kun Péter (kun.peter@ek.hun-ren.hu)

Introduction

During the lab practice, students will get acquainted with one of the most exciting surface analysis tools — the Scanning Tunneling Microscope (STM). They will have the opportunity to perform STM measurements and evaluate the results. The invention of the STM in 1981 (by Gerd Binnig and Heinrich Rohrer at IBM) opened the path to atomic-scale investigations. Their work was awarded the Nobel Prize already in 1986. STM enables atomic-scale investigation and modification of electrically conductive (e.g., metal, semiconductor) surfaces. In the STM, a particularly sharp tip — ideally terminating in a single atom — is brought within sub-nanometer distance to the sample surface while applying a voltage of the order of 0.5 V between the tip and the sample. Even though the tip and the surface do not touch, their electron wave functions overlap enough to allow electrons to "tunnel" through the vacuum gap, a phenomenon predicted by quantum mechanics.

In the most commonly used STM measurement mode (constant current imaging), the tip scans across the sample surface while a feedback loop adjusts its vertical position (tip-sample distance) to maintain a constant tunneling current (typically around 100 pA). The resulting 3D motion of the tip is then used to generate the STM image. A properly shielded (mechanically and electrically) STM, when used on suitably smooth surfaces (e.g., cleaved single crystals), can routinely achieve atomic resolution. Since STM enables “seeing” at the atomic level, atoms can also be manipulated and positioned one by one. A famous example is the "quantum corral" experiment by Crommie and Eigler (IBM, 1993), where 48 iron atoms were arranged in a circle on a copper (111) surface, visualizing electron standing waves (Figure. 1). These measurements already nicely illustrate that the STM image is sensitive to both atomic corrugations, and electronic effects. By forming an ellipse and placing a cobalt atom at one focal point, they observed "quantum reflection": a mirror image of the atom appeared in the STM image at the other focal point.

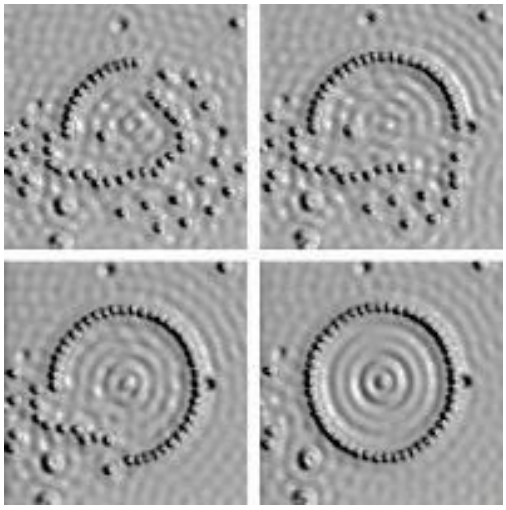

Figure 1. Atomic-resolution STM images show the formation of a “quantum coral” of 48 iron atoms on a copper (111) surface. M. Crommie et al., Nature. 363. (1993) 524–527.

Theoretical Summary

The Tunneling Effect

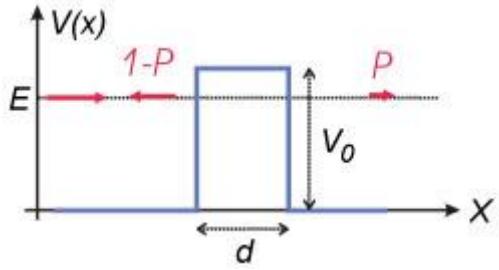

Figure 2. A potential barrier of height V_0 and width d. A particle arriving with energy E can penetrate the potential barrier with probability P and is reflected with probability (1-P).

If we apply an electric voltage between two conductive materials and bring them into contact, an electric current flows. As we separate the electrodes, the current gradually decreases — not abruptly, but exponentially with distance. At macroscopic separations, the current becomes immeasurably small and practically zero. However, if the distance between the electrodes is sufficiently small — on the order of angstroms (1 Å = 100 pm) — a measurable current still flows. This phenomenon is known as the quantum mechanical tunneling effect. A particle approaching a potential barrier of height V_0 and width d has a finite probability of passing through it, even if its energy E is less than the barrier height ($E<V_0$), therefore, in classical mechanics it would **always** be reflected, as it has not enough energy. By contrast, in the quantum mechanical case, the probability that it passes through the potential barrier (tunneling probability) is given by:

$$P=\frac{16E(V_0-E)}{V_0^2}\, e^{-2\kappa d} \qquad (1)$$

where $\kappa = \sqrt{\frac{2m(V_0-E)}{\hbar}}$

(This formula holds asymptotically when, $\kappa d >> 1$. Here $\hbar = h / (2\pi)$, with h being Planck's constant, introduced due to the quantization of energy.))

In the case of metal electrodes, the potential barrier height V_0 is on the order of electronvolts (eV). Importantly, according to this formula, increasing the electrode separation by 0.1 nm (1 Å) — a change on the scale of atomic dimensions — decreases the tunneling probability by an order of magnitude. For typical STM tip-sample separations of angstrom scale, $P \sim 10^{-5}$.

Tunneling Current

If we apply a bias voltage U_t between two electrodes, the resulting tunneling current I_t depends on this voltage, the material properties, and the tip-sample distance. Note that electron tunneling occurs even in the absence of an external bias voltage, just the probabilities to tunnel form tip to sample are roughly equal to the sample to tip tunneling, hence no net current occurs. We only apply a small voltage to break this equilibrium, and measure a net tunneling current, but the tunneling itself is an elastic process, it is not driven by the potential difference (at typical low bias voltages). In the simplest model — the free electron gas model — conduction electrons

move freely inside the metal but reflect from the surface. Given typical electron densities $n = 10^{29}\frac{1}{m^3}$ and average velocities $\mathrm{v_f} = 10^6\frac{\mathrm{m}}{\mathrm{s}}$, approximately $\frac{\mathrm{n}\cdot\mathrm{v_f}}{6} = 1.7\cdot 10^{34}$electrons per second strike a unit area collide with it. Assuming a fraction P of them tunnel through (as per the earlier formula 1), the resulting current is approximately: $\frac{\mathrm{n}\cdot\mathrm{v_f}\cdot\mathrm{A}\cdot\mathrm{P}\cdot\mathrm{e}}{6} = 2.6$ nA, where e is the electron charge, and A is the area of the tunneling channel — typically about the size of a single atom.

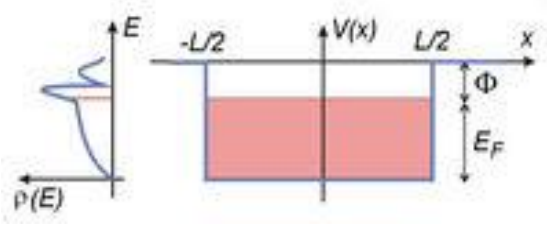

Figure 3. In the potential bath model of a metal, the electron experiences a potential of $-(\Phi + E)$ inside and zero potential in the vacuum outside the metal. The energy levels between $[-\Phi + E), -\Phi)]$ are filled, and the levels above are empty. The figure on the left shows a hypothetical density of states function (the density of states is the number of electron states at a given energy). The red dashed line indicates the location of the Fermi level. An electron at the Fermi level must be given energy Φ (work function) to escape the metal.

Although the obtained tunneling current almost corresponds to the usual tunneling current of the order of hundreds of pA of the STM, this rough estimate needs to be refined. First of all, the Pauli principle must be taken into account, since an electron with a given energy E can only pass to the other electrode if an empty energy state is available at that energy. Secondly, the tunneling current is also influenced by the energy distribution of the electrons in the electrodes. Taking these into account, we can arrive at the simplest quantum physical model of metals, which is the potential bath model (Figure 3). This assumes that the conduction electrons are enclosed in a potential box. In this model, we take into account the most essential properties of the conduction electrons of the metal:

a) the electrons are bound in the metal, work must be invested to free them, the smallest value of which is the work function Φ
b) Electrons in a solid (metal, semiconductor, or insulator) occupy energy states up to the Fermi level, filling the valence band, while the conduction band above the Fermi level remains empty.

The structure of this energy band is described by the density of states (DOS) function ρ(E). By definition ρ(E)dE is the number of electron states in the infinitesimal energy interval dE around the energy E. Knowing the density of states function, most important data of a given solid (e.g. electrical, optical, mechanical, thermodynamic properties) can be calculated, and as we will show below, the magnitude of the tunneling current is also determined by the DOS.

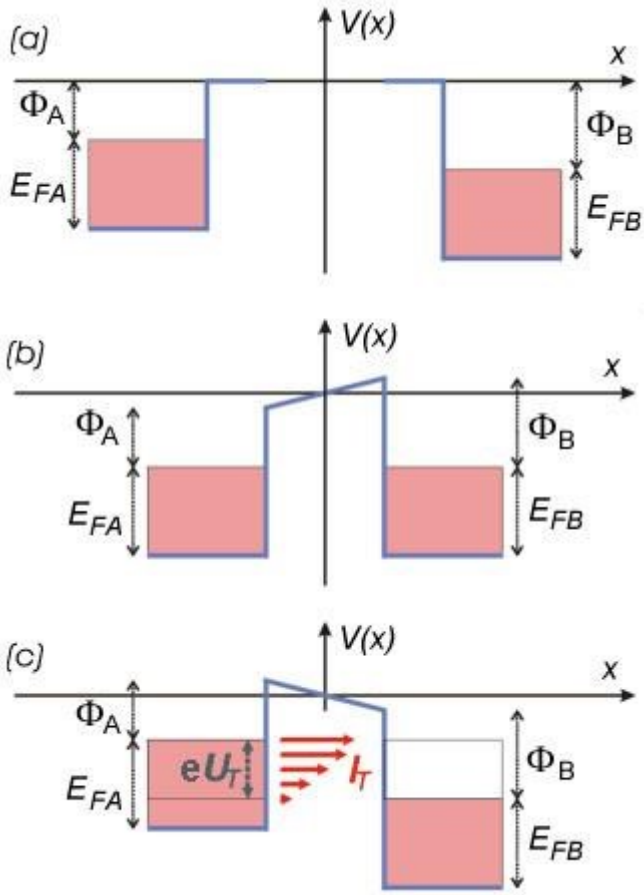

Figure 4. Band structure model of the tunneling current. (a) Electrodes A and B are far apart. (b) The electrodes are brought into sub-nm proximity, the Fermi levels are equalized and a contact potential is formed. (c) A bias U_t is applied between the electrodes. The tunneling current I_t flows in an energy interval of width eU_t, from the filled states of electrode A to the empty states of electrode B. The current contribution of higher energy components is larger, due to their higher transition probability.

In Figure 4/a, the band structure of two electrodes, initially at a macroscopic distance from each other is depicted. In general, the two electrodes (A and B) are made of different materials, therefore their Fermi energy and work function are different. In the figure the Fermi level of electrode A is higher than the Fermi level of electrode B. If we bring the electrodes close enough to each other that electrons can travel from one to the other with a significant probability (by tunneling effect), then electrons start to flow from electrode A to electrode B. This means that A gains a positive charge and B a negative charge, as a result of which both electrodes gain electrostatic potential energy compared to the vacuum level. Since "-" work is required to move an electron from infinity to the "+" charged surface A, and "+" work is required to move an electron to the "-" charged surface B, the energy levels of A are shifted in the "-" direction and the energy levels of B in the "+" direction relative to the vacuum level. Consequently, a contact potential is created between the two electrodes. The charge flow continues until the Fermi levels of the two electrodes are equalized (Figure 4/b). As we can see by simple calculations, to achieve equalization (in the case of metals), it is sufficient for very few electrons to pass from A to B compared to the total number of conduction electrons, so the change in the bandwidths is completely negligible.

If we then apply a bias voltage U_t between the two electrodes, which are at a distance d, the situation depicted in Figure 4/c arises. In this case, only electrons falling within the energy band of width U_t indicated in the figure can pass from electrode A to electrode B, because only at these energies are there filled states in electrode A and empty states in electrode B. Since the number of electrons passing through in an interval dE around a given energy E depends on the transition probability P and on how many states there are in the two electrodes in this interval dE, the tunneling current I_t flowing in the entire energy interval eU_t:

$$I \propto \int_{E_F}^{E_F+eU_t} \rho_{tip}(E - eU_t) \cdot \rho_{sample}(E) \cdot \mathrm{P(E}, U_t) dE \propto \int_{E_F}^{E_F+eU_t} \rho_{sample}(E)\, dE \qquad (2)$$

The latter approximation is true if we assume that the P probability and the density of states of the tip depend only negligibly on the energy.

The scanning tunneling microscope

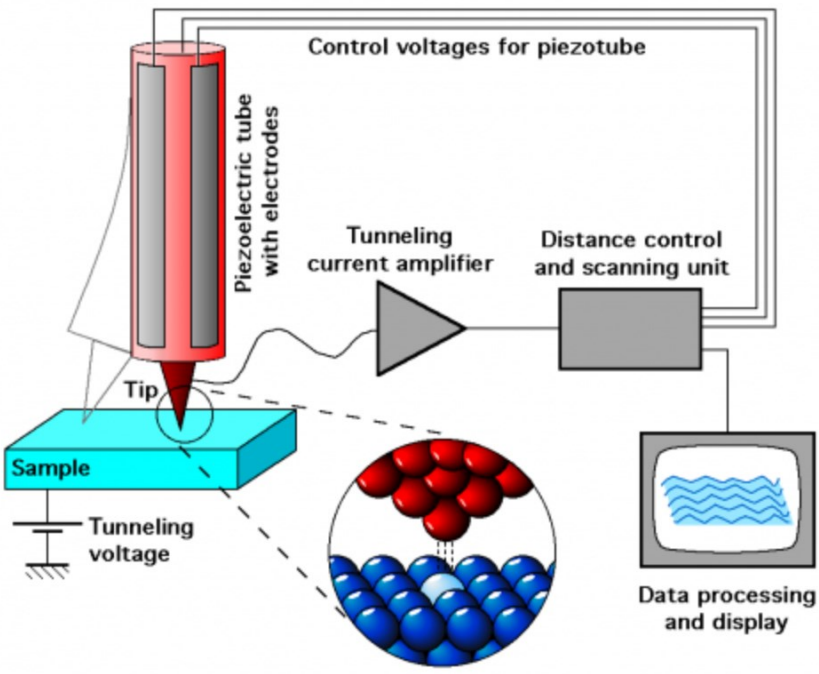

Figure 5. The principle of operation of the scanning tunneling microscope (STM).

The STM as a tool (Figure 5) works in the following way:

- One of the electrodes (e.g. platinum-iridium or tungsten) is made into a very sharp tip. In this case, the tunneling current will flow only in a narrow channel around the apex of the tip, since the tunneling probability depends exponentially on the tip-sample distance, so the current between the more distant parts of the tip and the sample is negligibly small. The width of this tunnel channel is approximately the same as the radius of curvature of the tip. Thus, the resolution of our instrument is primarily determined by the sharpness of the tip used. If we use a tip that is sharp on an atomic scale, the resolution can also be atomic.

- The tip is mounted on a three-dimensional mechanical actuator system that can move the needle over the sample with very fine (sub-angstrom, picometers) precision. In practice, piezoelectric actuators are generally used for this purpose.
- A bias voltage U_t of the order of half a volt is applied between the tip and the sample.
- The tip is then carefully approached to the surface until a tunneling current can be measured and reaches a preset I_{SP} reference value (current set point). The higher the set I_{SP}, the closer the needle gets to the surface, in practice its typical magnitude is around a few hundred pA.
- The tip is scanned in the *xy* plane of the sample, similar to the image generation in old cathode-ray tube TV sets (Figure 5). Of course, since we are not moving an electron beam here, but macroscopic masses, the applicable frame or line frequency is lower than that used in TV due to the inertia and resonances of the mechanical system. Therefore, the acquisition of an STM image usually takes, minutes or even tens of minutes.
- During scanning, the height of the tip above the sample (Z position) is controlled with a feedback loop so that the measured tunneling current is constantly equal to the I_{SP} value.
- In this way, each (x,y) position of the tip will have a specific z value. These z(x,y) values are recorded by a computer.
- In STM, very careful vibration damping is required because the tip must be moved above the sample at distances on the order of angstroms (Å). If the tip hits the sample, it usually causes damage to the tip apex and the sample, and random changes in the width of the tunnel gap appear as "noise" in the recorded image.
- Although the first STM experiments were performed in ultra-high vacuum (UHV), it was later found that the STM also works in air or even in (insulating) liquids. Therefore, expensive vacuum systems are only necessary if the properties of the surface to be measured require it, i.e. if the sample under study would oxidize or become contaminated in air. Since samples resistant to oxidation (e.g. carbon, noble metals) can be examined in air, the tunneling microscope is a relatively inexpensive instrument compared to other atomic-resolution characterization instruments of modern materials science, such as the electron microscope.

STM modes

In practice, the tunneling microscope is usually used in topographic or spectroscopic mode.

In **topographic mode**, with a fixed bias U_t and tunneling current I_{SP}, we map the sample surface z(x,y). If we assume that the tunneling probability and the density of states of the sample are homogeneous, the surface will give the geometric surface, the topography of the sample, to the first approximation. However, in practice, these quantities can also depend on the location, so it is worth remembering that the measured surface z(x,y) contains both the topographic and electronic structure information. In topographic mode, a small bias is most often used. If U_t is infinitesimally small, the energy dependence of the density of states (DOS) and P in the integrand of formula (2) can be neglected. Since the location dependence of the tunneling probability plays a minor role compared to the location dependence of the DOS, the tunneling current will be proportional to the value of the sample density of states corresponding to the Fermi level:

$$I_t(r,\ U_t) \propto\ U_t \cdot \rho_{sample}(E =\ E_F,\ r) \qquad (3)$$

In the **spectroscopy mode** (Scanning Tunneling Spectroscopy, STS), we stop the tip at a selected (x,y) point and temporarily turn off the feedback loop. At this fixed tip position r_0, we record the current-voltage characteristic of the tunnel junction, i.e. by changing the bias voltage, we measure the change in the tunnel current. Since changing U_t corresponds to changing the integration limit of formula (2), by differentiating the

measured current with respect to voltage, we obtain the energy dependence of the integrand of the formula:

$$\frac{dI_t(U_t)}{dU_t} \propto \rho_{sample}(E = E_F + eU_t,\ r = r_0) \qquad (4)$$

The STM family

Brilliant ideas often have a fertile effect on the development of science. This was also the case with the STM, which led to the creation of the so-called scanning (or local) probe microscopy (SPM) method family. In each of these devices, a sharp probe is scanned over the sample and some kind of interaction between the probe and the sample is measured. The most important of these is the Atomic Force Microscope (AFM). In the AFM, similarly to the STM, a sharp tip is moved over the sample and the force applied is kept constant by means of a feedback loop. Since this instrument also allows the examination of insulating surfaces, it is a more versatile method than STM, yet its commonly used modes are not able to provide information on the electronic structure of the samples.

Samples

The size of the samples typically does not exceed 20 mm in diameter and 2-3 mm in thickness. The samples must be mounted on a sample holder. The mounting must ensure both electrical conduction and mechanical stability.

In order to measure a sample with an STM, it is necessary for its surface to be electrically conductive. As we know, metals are generally excellent electrical conductors, so we can expect that we can measure them well with an STM. Unfortunately, this is not always the case. For example, aluminum is an excellent electrical conductor, but when exposed to air, a thin oxide layer (Al_2O_3, also known as sapphire or corundum) forms on its surface in a short time, which in turn is one of the best insulators (so much so that it is the main raw material for high-voltage insulating ceramics), and on top of that, it is also very hard (abrasive and precious stone, see corundum, sapphire). Similarly, we know that iron is very prone to rusting (mixed oxide and hydroxide), and the rust can be so thick within a short time that it completely covers and contaminates the scanning tip. It is logical to think that we might have better luck with noble metals. And indeed, for example, gold and platinum can be measured quite well, under ambient conditions, with an STM, but for example, silver, which is otherwise an excellent electrical conductor, cannot! The reason for this is not oxygen, but another chemical element belonging to the oxygen group, sulfur. Sulfur is always present in the air due to industrial pollution, and silver very easily forms sulfide with it. (This also gives silver objects their dark gray color.) Copper, which can be considered a semi-precious metal, is also not without problems, as it oxidizes quite easily and forms a greenish-bluish patina on it (nitrates, hydrocarbons, etc.). However, we have the advantage that copper oxide (CuO) is semiconducting in nature (it was also used for rectification purposes at the beginning of the past century), thus it does not prevent the formation of tunnel current, provided that the oxide layer is not too thick. (A freshly vacuum-evaporated copper layer can be measured in open air for days with an STM.) Certain metals can be measured well in open air, e.g. nickel, chromium and molybdenum. The oxide layer formed on these is only thin, and they are also quite semiconducting in nature. Besides metals, semiconductors (e.g. SnO_2) can also be measured in ambient STM. However, we have a problem with the most important semiconductor, silicon, because it oxidizes very quickly (in a

fraction of a second), and its oxide, quartz (SiO_2), is one of the best insulators and hardest materials. Despite this, silicon can still be measured in open air with an STM, because the SiO_2 formed on its surface is very thin (on the order of nanometers), so electrons can tunnel through this insulating layer. Furthermore, proper STM examination of pure silicon is only possible in ultra-high vacuum, for a limited time after the in situ purification. Among the more special materials, metallic glasses can be measured well primarily if they contain a sufficiently large amount of noble metal components.

However, the most studied material in ambient STM studies is graphite. This material is electrically conductive, with slight metallic luster. Its soft nature arises from the easy sliding of its layers relative to each other (this is why we can write with a pencil), while within each layer the carbon atoms are held in place by very strong covalent bonds. Therefore, graphite is a highly stable (non-reactant) material, (it does not oxidize), it conducts electricity, and its surface is almost atomically flat, apart from atomic steps. For all this, we can relatively easily achieve even atomic resolution on it by ambient STM. In order to achieve this, we use HOPG (Highly Oriented Pyrolitic Graphite). Its crystal structure is honeycomb-like, with a lattice periodicity of 0.246 nm, and can be built up by stacking hexagonal graphene in an ABAB stacking arrangement. This means that every second graphene sheet is shifted relative to the one above/below it so that, in terms of a hexagon, 3 atoms overlap with those in the adjacent layer, and 3 are located under the center of the hexagons in the adjacent structure.

Moiré-effect

A well-known and spectacular phenomenon in optics can be witnessed if we place two periodic gratings on top of each other, slightly rotated with respect to each other. In this case, a new, larger-period superlattice appears, which is called a moiré pattern (Figure 6). For example, if we stack two graphene lattices (atomic scale gratings) characterized by a lattice constant d on top of each other, rotated with respect to each other by an angle θ, then the superperiod D of the interference pattern that appears is:

$$D = \frac{d}{2 \cdot \sin\frac{\theta}{2}} \tag{5}$$

This phenomenon can also be easily studied with STM, and is often used to determine the rotation angle of stacked 2-dimensional layers (graphene, hexagonal boron nitride, molybdenum disulfide, etc.) relative to each other.

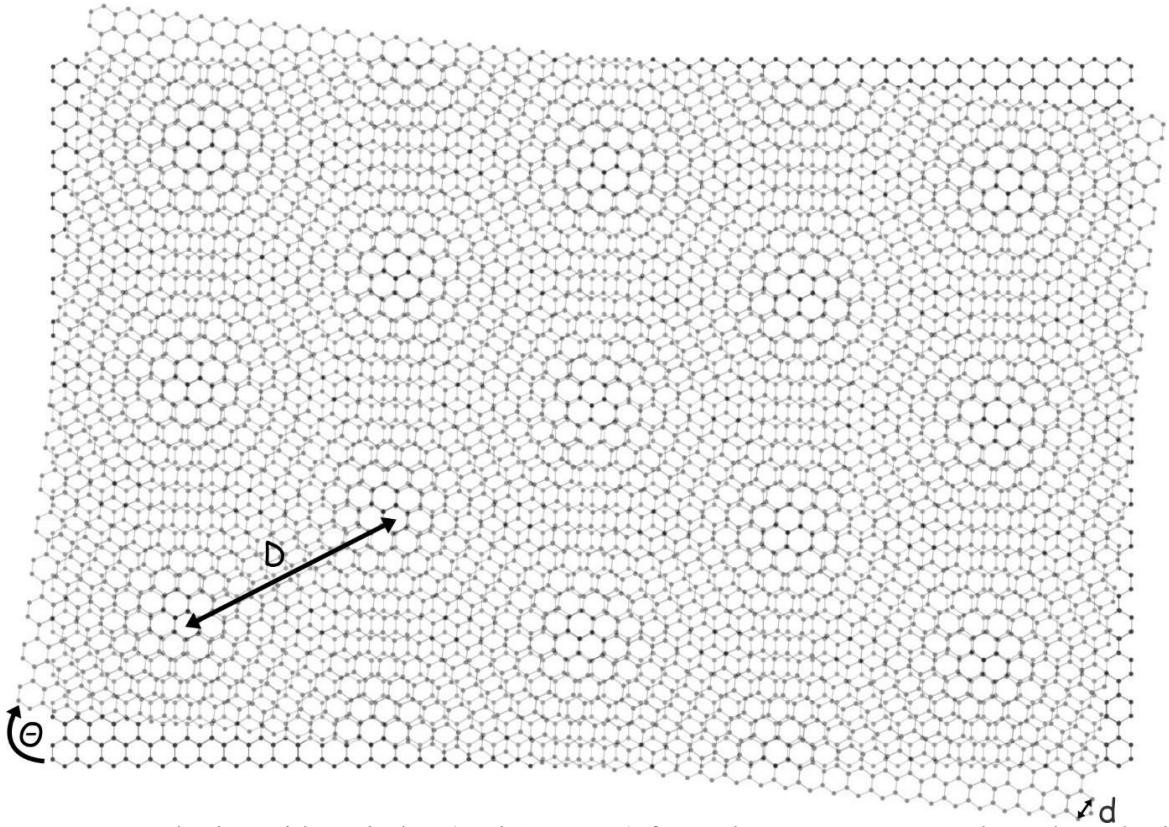

Figure 6. A superlattice with period D (moiré pattern) formed on two superposed graphene lattices with lattice constant d, rotated by an angle Θ with respect to each other.

Tasks

1.) Measure the surface topography of a graphite (HOPG) sample with different maximum vertical scanning range (z_{limit}) values, and examine the change in the achievable vertical resolution as a function of this!
2.) Look for atomic steps on the HOPG surface and determine how many layers the height difference corresponds to!
3.) Determine the distance between carbon atoms in the crystal lattice from atomic resolution measurements.
4.) Determine the angle of rotation between the two layers from the moiré pattern observed on the graphene.

Appendix

The Appendix contains technical details that will help you perform the measurement (with the help of the lab instructor) and evaluate the results.

Instruments

For the measurements, we have a Nanoscope 3 STM/AFM device manufactured by Veeco/DI (now Bruker). Its characteristic is the modular design, which allows us to combine different scanning heads, control electronics and computers. The device operates under ambient conditions. In our case, the measuring head allows scanning a 15x15 micron horizontal and 3 micron vertical range, but this can be easily replaced with a head capable of scanning a larger range.

The basic task of the **vibration damper** is to keep mechanical vibrations coming from the outside world away from the actual STM, or to dampen them to an acceptable level. This is a very important part of the device, since the needle scanning the sample has to be moved at a distance of only Å from the sample surface, so if the vibrations cause a relative displacement of the needle and the sample with a larger amplitude, the needle can hit the sample, which of course causes the needle tip to become blunt and the sample to be locally damaged. In the case of very low-frequency disturbances, the situation is not dramatic, because the current needle-sample distance is controlled by a negative feedback capable of responding with a frequency of a few kHz. So as long as the relative displacements resulting from the vibrations are not too fast and do not exceed the control range, the STM retains its functionality. However, maintaining operability does not mean optimal operation, because even in the case of successfully compensated mechanical vibrations, the interfering signal is added to the measured signal, which results in the appearance of various, non-real periodic features in the STM image. Therefore, our primary task is to attenuate the level of audio frequency vibrations by orders of magnitude, and infrasound vibrations as much as we can. The principle of attenuation is to reduce the acceleration force acting on the STM head, resulting from external vibrations, by interposing an elastic suspension (in practice, we place the STM on a large marble slab suspended on a rubber bungies).

The STM microscope head (Figure 7) is the most important part of the device, comprising the scanning tip. This is actually a thin platinum-iridium wire, sharp at one end. The STM tip is placed inside a small holding tube on the measuring head. The question arises, how can a wire be sharpened so well? There are various methods for this (e.g. electrochemical milling). We will employ mechanical sharpening, i.e. cutting the wire with a sharp pair of scissors in combination with a specific removal movement. The tips are checked under an optical microscope. Of course, there is no guarantee that an optically well looking needle will be atomically sharp, since in an optical microscope we can only see the end of the needle at a resolution of about 1 micron, i.e. about 3-4 orders of magnitude coarser than the atomic level. However, practice shows that every second or third sharpening attempt of a skilled person results in tips that can achieve atomic resolution measurements. The cross-section of the tip apex is usually not circularly symmetrical, but more irregular. A characteristic defect of needles produced by mechanical sharpening is that there may be several so-called nano-apexes next to each other, of which the tunneling current can flow. In the case of a sufficiently flat sample (e.g. HOPG), a single active nano-tip may exist throughout the measurement, while in the case of more corrugated surfaces (e.g. polished metals) the imaging nano-tip may change several times during a single measurement, or even multiple nano-tips may become active at the same time,

resulting in multiple (ghost) images superposed on the STM image. The aforementioned tip defects can be inferred from the quality of the recorded images. In other words, the quality of a tip can ultimately be judged by the images it can produce. Therefore, making the right tip is a rather time-consuming process, as preparing and performing each test measurement takes time. Moreover, the tip can be destroyed in the blink of an eye at any time, as it only takes a tiny bump into the surface of the sample. To remedy the situation, we provide a properly sharpened needle for the measurement, but of course, students can also try sharpening the needle. The needle is connected directly to a preamplifier, thus reducing the noise of the tunnel current. The device can work with a tunnel current of at least 100 pA, and the preamplifier slows down dramatically at lower currents.

Figure 7. STM microscope head

The mechanical movement (scanning) of the sample is performed by a piezoceramic scanner tube, under the driving of an appropriate (maximum ±220V) voltage. The coarse approach and retractions of the tip is performed by a micrometer screws, which is operated by a stepper motor. In case of sample or tip replacement, the tip can be removed together with the preamplifier. After tip replacement, the preamplifier is fixed with springs, the tip is brought closer to the sample with the screw to a distance of approx. 0.2 mm, and then the tip is continued to be approached with the help of the piezo actuators monitoring the tunnel current in order to soft-land the tip on the sample (a few angstroms above), avoiding to crash it into the sample.

The measurement mode (Realtime) can be started using the button  , and the recorded images (Offline) can be accessed by pressing the button  . In the measurement mode  (Figure 8), the Scan Controls window allows you to adjust the scan window size, scan location, angle, frequency, speed, and resolution. When choosing the scan speed (frequency), you must take into account the inertia of the feedback system and the roughness of the sample, as high speeds can result in damage to the sample and/or the tip. The resolution of the STM image can be 128, 256, or 512 dots/line. The tunneling current, the tip-sample (bias) voltage, and feedback strength can be adjusted in the Feedback Controls window. Higher Integral and Proportional Gain values allow for faster scanning, but if the values are too high, the feedback loop will overshoot, resulting in high noise. The maximum vertical scan range and color scale can be adjusted in the Other Controls window. In the case of an unknown sample, it is worth using the maximum vertical range, but this results in poorer vertical resolution (resolution = $z_{limit}/2^{16}$). In the Channel windows, you can select the type of recorded data (height or current), scale size, scan direction, and tilt compensation method.

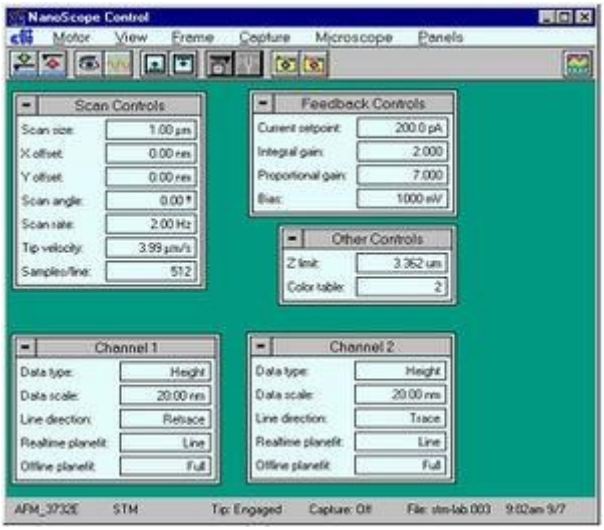

Figure 8. Measurement mode in the Nanoscope Software

Additional icons of the measurement control program: the tip is brought closer to the sample until the set current appears. causes the tip to retract from the sample. Pressing causes the image in progress, while pressing causes the last image line to be shown on the screen.

Starts the scan from bottom-to-top or from top to bottom. activates or deactivates the image capture.

The bottom of the screen shows the probe type, mode, tip status, image capture status, file name and time. The file name can be set with the **Capture → Capture Filename** command. During the measurement, the scan can be followed on the display screen (**Figure 9**). The data for channel 1 (scanning from right to left) is shown on the left side of the screen, and the data for channel 2 is shown on the right side of the screen. It is IMPORTANT to constantly monitor the Z Center Position indicator in the lower left corner of the screen, which indicates the voltage applied to the piezo ceramic. If the white vertical line indicated by the red arrow reaches the end of the green rectangle, the ceramic has maximally contracted or extended and is unable to follow the surface. The situation can usually be improved by increasing the maximum vertical range (Z limit) or reducing the scan window (Scan size). If this does not work, the tip must be retracted.

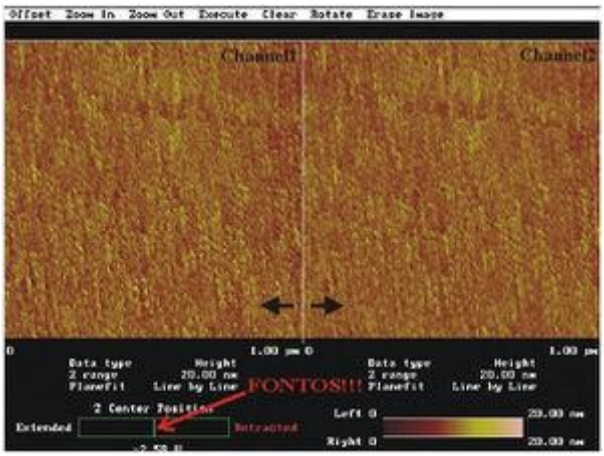

Figure 9. Display screen

Measurement

The sequential steps of the measurement are similar regardless of the specific type of sample. The general procedure is as follows:

1.) First check that the individual units are connected correctly, then turn on the control electronics and computer with the main switch (so that they reach operating temperature as soon as possible). This is done by the lab instructor before starting the measurement.

2.) We prepare the sample to be measured. Usually the instructor does this before starting the measurement.

3.) The next step is to try to produce a reasonably sharp needle. (Although the instructor will have a needle sharpened beforehand, students can also try sharpening it.)

4.) Then, using tweezers, we insert the tip into its socket, approach the tip to the sample to approximately 0.1 mm, and then place the device on the vibration-free table.

5.) We set the desired scanning parameters (window size, number of pixels per line, scanning speed, tunnel voltage and current), then activate the coarse tip approach mechanism 

6.) The measurement control software automatically starts data collection. If the image is suitable, we save it to the HDD  . If the image is not good enough, we try to improve it by adjusting the parameters. If this does not work at all, the most common reason is that the tip is dirty or blunt. In this case, we stop the measurement. 

7.) Increase the tip-sample distance to at least 2mm.

8.) We remove the tip and return to point 3.), i.e. to resharpen or replace the tip.

Image and Data analysis

The best alternative for image processing is to use the free image processing program Gwyddion. Here is a brief summary of the most important functions. After opening the file, a window can be opened with the **Info→Show Data Browser** command, in which you can select the channel of the saved multichannel file to be examined (e.g. topography, current image, etc., the program opens channel 1 by default). Frequently used commands are available by clicking on small icons. The height of individual scan lines often differs, so click: **Align rows using various methods** 
In the pop-up window, you can use different methods to align the lines. To remove horizontal lines in the image: **Correct horizontal scars** ,  but be careful with this, as it can greatly degrade the image quality of the area around the line. We can correct the tilt of the image: **Level data by fitting a plane through three points**  . After that, you need to click on three points in the image that you are sure are in plane, and to do this, you need to level the other points. Sometimes it looks good if we display the data in 3D space: **Display a 3D view of data**  . We can also crop a portion of the image for further analysis: **Crop data**.  We can set the color scale that displays the z-axis values: **Stretch color range to part of data**  The color palettes can be accessed by right-clicking on the color scale next to the image. To evaluate the measurement, we will need to filter out the periodic signal from the noise, for this: **Data Process→Correct Data→2D FFT Filtering**, and a window will pop up where we need to select the Fourier-filter shape of the expected periodic pattern (e.g., circle the transform of the triangular grid, which is a hexagon).

The Nanoscope program's own format cannot be opened using common image processing programs, but the free WSxM 3.1 or later program can open and process Nanoscope files. The operation of the WSxM program is similar to the operation of the Nanoscope program, but its appearance and the names of the commands are different. We can remove the tilt of the image by subtracting a plane fitted to the entire image (**Process→Plane→Global**) or to a part of the image (**Process→Plane→Local**). If necessary, we can extract a quadratic surface from the image with the **Process→Filter→Fit2nd** command. The heights of the individual scan lines often differ, so it is usually worth executing the **Process→Filter→Flatten** command. With this command, we fit a straight line (Offset - no tilt and Line - there is tilt) or a parabola (Parabola) to each scan line and subtract it from the scan line. **Process→Filter→Flatten→Line** also replaces the **Process→Plane→Global** command. Spikes that may appear in the image can be removed, for example, with the **Process→Filter→Matrix** convolution→Smooth_3x3 averaging matrix filter. A detail of the image can be enlarged with the **Process→Zoom** command. A line section can be created with the **Process→Profile** command. The distance between two points on the section can be measured with the **Process→Measure** distance command. The 3D display of the image can be modified with the Display→3D Settings and Preview command. Here it is possible to tilt and rotate the image, as well as change the direction of the illumination. In 2D representation (**Display→Top View**), it is important to use a color scale. The color scale can be accessed with the **Display→Info** command.