Ba10: Scanning tunneling microscope

1 Overview of the topic

The scanning tunneling microscope (STM) is an imaging technique for non-destructive examination of surface topographies down to a resolution of atomic distances (a few $\mathring{A}$).

It uses a metallic pointed probe hovering in a distance of $< 10\mathring{A}$ over the surface. An external difference in potential V_t between probe and surface leads to a net tunneling current I_t. This current directly depends on the electron density distribution of both surfaces and is an exponential function of distance s between probe and surface:

$$I_t \sim V_t * \exp(-c * \sqrt{\Phi} * s) \quad (\phi\text{: work function in } eV,\ s \text{ in } \mathring{A},\ c \approx 1.02\mathring{A}^{-1}eV^{-1/2})$$

Because the work function has a magnitude of multiple eV, the tunneling current is changing about one magnitude per $1\mathring{A}$ in change in distance between probe and surface. This extreme dependency on distance allows for the observation of atomic structures.

One application of this method is the examination of structure and morphology of technical surfaces (roughness, steps, grain-borders etc.). Additionally, it is possible to determine the geometric arrangement of individual atoms on the surface. This however often requires the use of ultrahigh vacuum equipment to prepare defined surfaces.

With this experiment you are to be introduced to into the application of the scanning tunneling microscope under normal atmospheric conditions.

2 Theoretical basis

Crystalline solids are characterized by a three-dimensional periodic arrangement of their building blocks (atoms or molecules) [Kit]. This symmetry which occurs in the volume is disturbed at the surface interface.

The potential threshold originating from the interface between solid and its surroundings poses certain boundary conditions on the propagation of the electronic wave function in the solid and the surrounding space. Therefore, the amplitude of the wave function Ψ has to drop off exponentially when leaving the solid:

$$\Psi = \Psi_0 * \exp(-\chi z)$$

The damping constant χ depends on the binding energy of state Ψ and is smallest for electrons near the Fermi energy. The wave function of these electrons therefore reach the farthest out of the solid and can be "seen" the brightest by the probe head.

The change in behavior of the wave function at the surface compared to space within the volume leads to a change in the electronic charge density distribution [Zan]. This process can be associated to a rearrangement of surface atoms (reconstruction), if this leads to a lower energy state of the system.

Using the scanning tunneling microscope, the electron density distribution at the surface of conductive samples can be visualized in real space with atomic resolution. The basis of this sort of microscopy is quantum tunneling [Dav]. If the distance between probe head and sample is less than $10\mathring{A}$, the overlap of electronic wave functions from the surface with functions from the probe is sufficiently high.

Tunneling currents of up to $100nA$ can be reached for a differential potential of $10mV$ to $1V$. The probe head is scanning the surface (X, Y-Signal) using piezo-electric positioning elements. During this process, the tunneling current is held at a constant value by changing the distance between probe and

sample using an electronic measurement and control circuit. This circuit feeds a Z-signal back to the Z-piezo in order to achieve the desired current value.

X, Y and Z values combined can be translated into a three-dimensional image of the surface topography. In this mode, the probe head is following the contour of the constant overlap of the wave functions from probe and sample. The measuring range of a STM is limited by the used piezo elements. The maximum scanning surface can be up to 20µm x 20µm. Therefore this method is also suitable for the examination of technical surfaces, where a judgment of quality, type and roughness is key.

3 Preparation

The basics of the following topics should be known at the start of the experiment:

- Quantum tunneling
- Piezo-electric effect
- Feedback and control circuits
- Tunneling spectroscopy
- Spatial structure of solids
- Electronic structure of solid surfaces
- Fourier transform

4 Experimental setup and tips

4.1 Technical setup of the scanning tunneling microscope

The STM in use is a custom design of AG Franke dedicated for use under atmospheric conditions. Control and imaging is done through the GUI of the Nanotec electronics. This interface consists of Digital/Analog converters for controlling the X- and Y- piezo elements as well as an Analog/Digital converter for measuring the Z-voltage generated by the control loop. Communication with the electronics proceeds via a PC with the freeware WSxM supplied by Nanotec.

The probe head of the STM consists mainly of a piezoelectric tube scanner which can be approached to the sample surface using a micrometer screw. The probe head approach towards the surface is started by hand while carefully observing the approach with an optic microscope until the given tunneling current is reached.

The entire unit is mounted on a vibration-damping table to decouple the microscope from mechanic vibrations.

Both data collection as well as post-analysis using an image processing system are done with the special freeware package WSxM which can be downloaded from http://www.wsxmsolutions.com for data evaluation and analysis purposes at home.

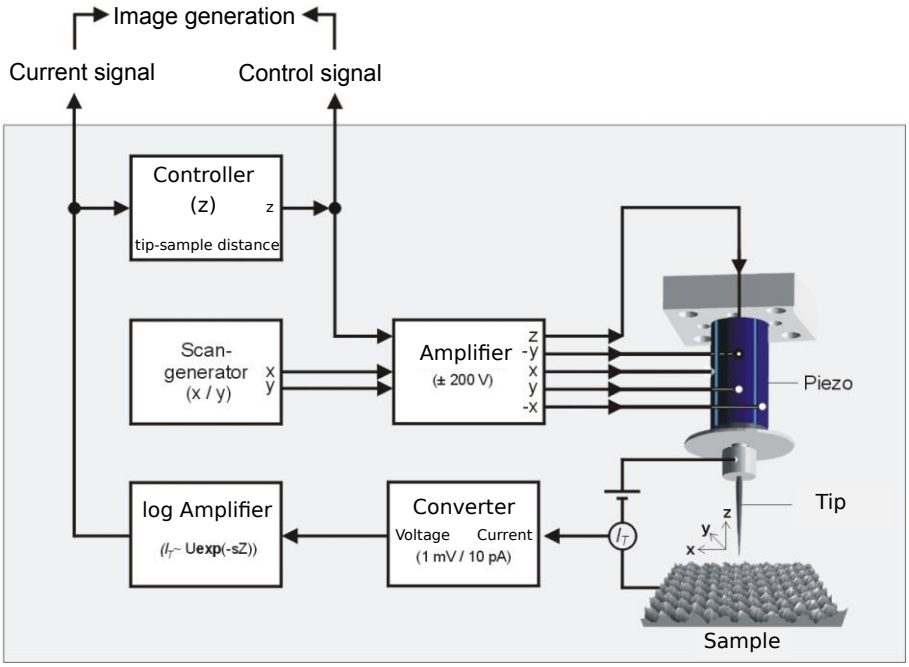
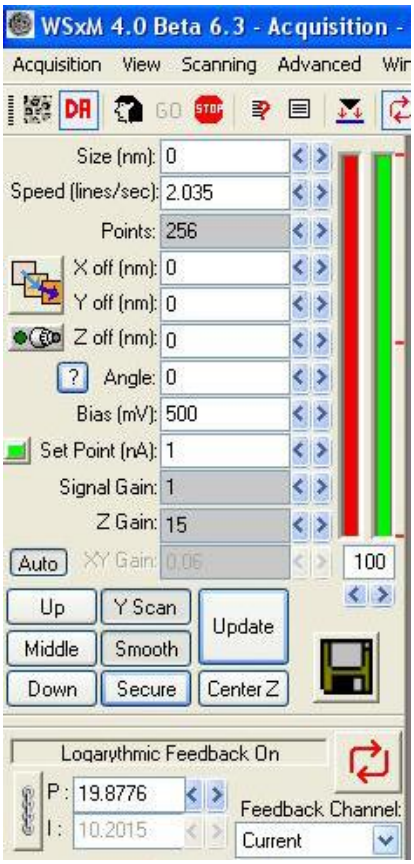


Figure 1: Block diagram of the STM (by Frank Trixler, Ludwig-Maximilians-Universität München).

4.2 Data collection software



The software WSxM offers two different modes of operation: Generating and plotting tunneling images in real time and processing of previously saved images. A couple of parameters have to be set before image generation. They can be accessed via the menu *DA* (data acquisition). The parameters have the following meaning :

STM menu			
Parameter	Description	Manual approach	Imaging
Size (nm)	Size of the square scanning area	[0 nm	1 - 50 nm]
Speed (lines/sec)	Scan speed	2	$\propto$4 to 1
Points	data points per line	256	256
X / Y off (nm)	offsets of scan area	0	...
Z off (nm)	offset of zero point along Z	adopt!	
Angle	Rotation of image	0	0
Bias (mV)	voltage applied to sample	+ 1000 mV	$\pm$30 - 1000 mV
Set point	tunneling current	1 nA	1 nA - 30 nA
Signal Gain	amplification factor	1	1
Z Gain	HV amplification for Z scan (10 - max.)	10/5/3/1	
XY Gain	HV amplification for X,Y scan		
P	proportional gain	0.12 - 0.06	
I	integral gain	P/2: 0.06 - 0.03	

Table 1: Parameters of WSxM control software

During the approach the window *Approach* has to be open and the approach control has to be activated. Upon detecting a tunneling current the proportional gain P has to be set to 1.5. The screw driver is removed and the protection cover put in place. Then the window *Approach* can be closed.

A measurement is startet by pressing **Secure**. Thereby the STM tip is further approached to the sample by the control electronic until the preset tunneling current (Set Point) is reached. Then, the real time generation of the scanning image will begin. Individual parameters can also be reconfigured during a scan allowing for changing the scanning area or improving the imaging quality. To cancel the scan press **Secure**, which will withdraw the probe head from the sample and turn off the high voltage on the piezo elements. The scan image is constantly refreshed onscreen. To save it on the PC press the "save" icon. The data is stored in a predefined folder. The stored images can be edited via the menu *Representation*.

Prior to tip or sample exchange: Completely retract the tip and set the X, Y and Z offsets to zero! This procedure must be completed before switching samples in order to protect the integrity of the probe head and prevent electrical arcing.

5 Tasks

Highly oriented Graphite (0001)

a) Use the Graphite sample. Use the parameters from Table 1 as defaults. Especially familiarize yourself with the parameters "Integral gain" and "Proportional gain". How do these parameters have to be changed if you want to switch to higher scan rates or areas?

b) Change the size and offsets of the scanning area to find areas with step edges or grooves and acquire appropriate images. Determine the height of many different steps (>20). Use thereby the filter *local plan correction* for determining the step height. Sort the observed heights by value and plot the height vs number of the measurement from your series. Compare the obtained values of step heights and groove depths with the lattice constant of graphite. Which sources of uncertainty have to be considered in the comparison? Make sure that the calibration of the Z piezo (1 nm/V) is correct. If not, determine the appropriate value.

c) Look for an area on the graphite surface that is completely flat and try to scan the "atomic" structure of graphite.

d) Interpret the occurring structures and determine the height of "atomic" elevations, as well as the distance between two neighboring elevations using the menu **Representation**. What distances in the graphite surface can you identify these structures with? Are the piezo x-y calibration values correct? Which symmetries occur? Determine the angle between atomic directions. Are they the same? Discuss possible sources of distortion. Which is the direction of greatest distortion? How can you explain the height of the elevations?

e) Perform a two-dimensional Fourier transform on the image and interpret the resulting pattern. What structure information can be obtained from the FT?

f) Discuss the results from b) to e)

6 Numbers and constants

Graphite:	planar distance between closest neighbors	$1.42\,\mathring{A}$
	Distance between levels	$3.35\,A$

7 Literature

8.1 Textbooks

[Kit] Kittel, Einführung in die Festkörperphysik

[Dav] Davydov, Quantenmechanik

[Zan] A. Zangwil, Physics at Surfaces

8.2 Manuals

[**WSxM**] **The manual is available after installing the WSxM software (Windows or Wine emulator) in the Help menu.**

8.3 Primary literature

8.3.1 G. Binnig, H. Rohrer, Scanning tunneling microscopy, Helv. Phys. Acta 55, 726-735 (1982)

8.3.2 P.K. Hansma, J. Tersoff, Scanning tunneling microscopy, J. Appl. Phys. 61, R1 (1987)

8.3.3 J. Tersoff, D.R. Hamann, Theory of the scanning tunneling microscope, Phys. Rev. B31, 805 (1985)

8.3.4 P.K. Hansma, V.B. Elings, O. Marti, C.E. Bracker, Scanning tunneling microscopy and atomic force microscopy: application to biology and technology, Science 242, 209-216 (1988)

8.3.5 Y. Kuk, P.J. Silverman, Scanning tunneling microscopy instrumentation, Rev. Sci. Instr. 60, 165-188 (1989)

8.3.6 H. Fuchs, Strukturen - Farben - Kräfte: Wanderjahre der Raster-Tunnel-Mikroskopie, Phys. Bl. 45, 105-145 (1989)

8.4 Scanning tunneling microscope on graphite surfaces

8.4.1 J. Tersoff, Anomalous corrugation in scanning tunneling microscopy: Imaging of individual states, Phys. Rev. Lett. 57, 440 (1986)

8.4.2 H.A. Mizes, Sang-il Park, W.A: Harrison, Multiple-tip interpretation of anomalous scanning tunneling microscopy images in layered materials, Phys. Rev. B 36, 4491 (1987)

8.4.3 J. Tersoff, N.D. Lang, Tip-dependent corrugation of graphite in scanning tunneling microscopy, Phys. Rev. Lett. 65 1132 (1990)

8.4.4 Sang-II park and C.F. Quate, Tunneling microscopy of graphite in air, Appl. Phys. Lett. 48, 112 (1985)

8.4.5 A. Selloni, P. Carnevali, E. Tossati, and C. D. Chen, Voltage-dependent scanning-tunneling microscopy of a crystal surface: Graphite, Phys. Rev. B 31, 2602 (1985) Erratum to Phys. Rev. B 31: A. Selloni et al, Phys. Rev. B 34, 7406 (1986)

8.4.6 F. Atamny, O. Spillecke, and R. Schlögl, On the STM imaging contrast of graphite: towards a "true" atomic resolution, Phys. Chem. Chem. Phys. 1, 4113 (1999)

8.5 Academic literature

8.5.1 Tunnelspektroskopie R.M. Feenstra, Scanning Tunneling Spectroscopy, Surf. Sci. 299/300 965 (1994)

8.5.2 Vibrationsspektroskopie mit dem Rastertunnelmikroskop W. Ho, Single molecule chemistry, J. Chem. Phys. 117, 11033 (2002)

8.5.3 Manipulation einzelner Atome J.A. Stroscio, D.M. Eigler, Atomic and molecular manipulation with the scanning tunneling microscope, Science 254, 1319 (1991) M.F. Crommie, C.P. Lutz, D.M. Eigler, Confinement of electrons to quantum corrals on a metal surface, Science 262, 218-220 (1993). G. Meyer, K.H. Rieder, Bauen mit einzelnen Atomen, Physik in unserer Zeit, 31, 8 (2000)

8.5.4 Rastertunnelmikroskopie mit magnetischer Spitze M. Getzlaff, M. Bode, R. Wiesendanger, Nanomagnetische Domänen mit dem Rastertunnelmikroskop, Physik in unserer Zeit, 31, 110 (2000)

8.5.5 Rastertunnelmikroskopie mit supraleitender Spitze S.H. Pan, E.W. Hudson, J.C. Davis, Vacuum Tunneling of superconducting quasiparticles form atomically sharp scanning tunneling microscope tips, Appl. Phys. Lett. 73, 2992 (1998)