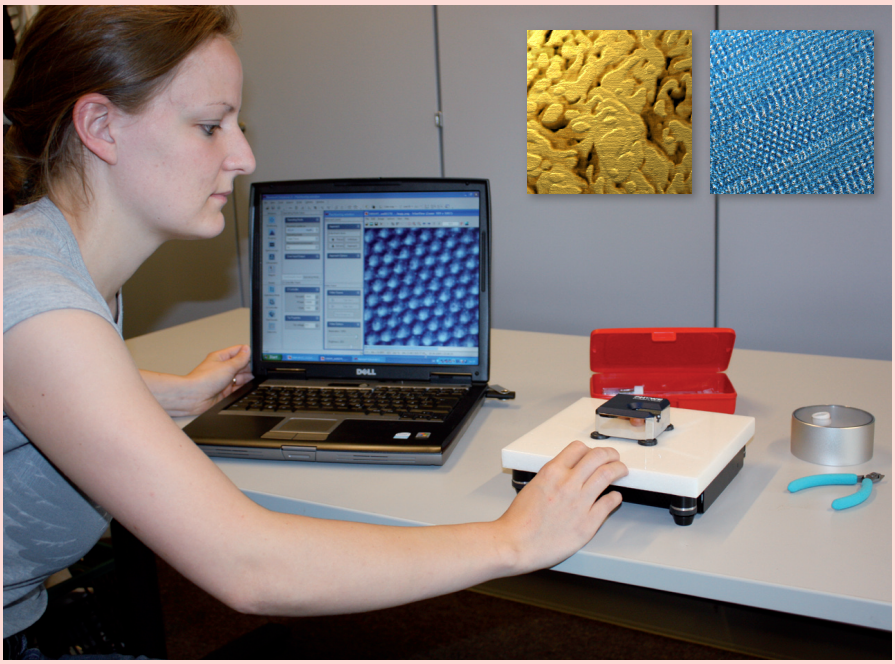
PHYWE

Operating Instructions and Experiments

TESS expert PHYWE

Scanning Tunneling Microscope (STM)



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PHYWE

Scanning Tunneling Microscopy (STM)
Operating Instructions and Experiments

Order No. 01192-02

PHYWE

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Scanning Tunneling Microscopy (STM) – Operating Instructions and Experiments
Order No. 01192-02

Author of experimental literature: Jannick Langfahl-Klabes

2nd edition

Table of Contents

CHAPTER 1:

Components of the System

The Scanning Tunneling Microscope was developed by Gerd Binnig and Heinrich Rohrer in the early 80's at the IBM research laboratory in Rüschlikon, Switzerland. For this revolutionary innovation Binnig and Rohrer were awarded the Nobel Prize in Physics in 1986. The STM uses a very sharp metal tip to scan the conducting sample surface. It measures the tunneling current which flows between the tip and the sample without being in direct contact. With the help of this current the tip-surface distance can be controlled very precisely using piezoelectric devices. Another piezo-electric device drives the tip movement line-by-line on the nanometer scale, whereas the tip surface-distance is recorded. Therefore structures on the sub nanometer scale, especially atoms and molecules, can be imaged easily.

1.1 Materials and Tools

PHYWE Compact STM System Product No.: 09600-99
consisting of:

- 1 X Control unit with mounted scan head (1)
- 1 X Magnifying cover glass (10X Magnification) (2)
- 1 X USB cable (3)
- 1 X Power cord and adapter (4)
- 1 X MeasureNano Software (5)

PHYWE STM Tool Box
consisting of:

- 1 X Wire cutter (6)
- 1 X Flat nose pliers (7)
- 1 X Pointed tweezers (8)
- 1 X Rounded tweezers (9)
- 1 X Pt/Ir-wire, d = 0.25mm, l = 30 cm (10)
- 1 X Sample Holder (11)
- 1 X Graphite sample (HOPG) (12)
- 1 X Gold sample (13)
- 4 X Spare sample support (14)

Caution!

- Keep your microscope and its components free of grease and dust
- Set up your system on a very stable table
- Do your experiments in a calm, vibrational free environment

Additionally needed equipment

- 1 X PC/Laptop with USB port, Windows XP or higher
- 1 X Ethanol and soft cleaning cloth
- 1 X Adhesive tape

Fig. 1-1 Components of the PHYWE Compact STM.

CHAPTER 2 :

Installing the System

2.1 Installing the PHYWE Measure Nano Software

Before the installation, the following steps need to be performed:

1. Make sure the computer to be used meets the minimal computer requirements (Computer requirements page 116).
2. When the PHYWE STM controller is connected to the computer via the USB cable, disconnect it by unplugging the USB cable from the computer. The PHYWE STM controller should only be connected to the computer when the software and driver installation is complete.
3. Turn on the computer and start Windows.
4. Log on to your computer with Administrator privileges.

IMPORTANT

The PHYWE STM Installation CD contains calibration information (.hed files) specific to your instrument! Therefore a backup copy of the delivered CD is recommended. Always store the CD in a safe place.

To initiate the installation procedure:

1. Insert the PHYWE STM Installation CD into the CD drive of your computer. In most cases, the Autorun CD Menu program opens automatically.
   Depending on your Autoplay settings, it is also possible that the Autoplay window opens, or that nothing happens at all. In these cases:
   - Click “Run CD_Start.exe” in the Autoplay window, or manually open the PHYWE STM Installation CD and start the program “CD_Start.exe”.
2. Click the “Install PHYWE Measure Nano Software” button. The CD Menu program now launches the software setup program, which starts the necessary drivers and the PHYWE STM software. In Windows Vista, the User Account Control (UAC) dialog may pop up after clicking the “Install PHYWE Measure Nano Software” button, displaying the text “An unidentified program wants access to your computer”. If the name of the displayed program is “Setup.exe”: Click the “Allow” button.

After the software setup program has started:

1. Click “Next” in the Welcome Screen.
2. Set the Destination Folder, either accept the default choice or adapt the folder path to your needs. Proceed with the “Next” button.
3. Set the Start Menu Folder, either accept the default choice or adapt the folder path to your needs. Proceed with the “Next” button.
4. When the “Ready to install” window appears, click the “Install” button.

The setup program now performs its tasks without any further user interaction. Depending on the configuration of your computer, a reboot may be required at the end of the software installation process. If this is the case, the setup program will inform you and will provide you with the opportunity to accomplish. Make sure any other data is saved before rebooting your computer.
This completes the software installation procedure. Proceed with the next section.

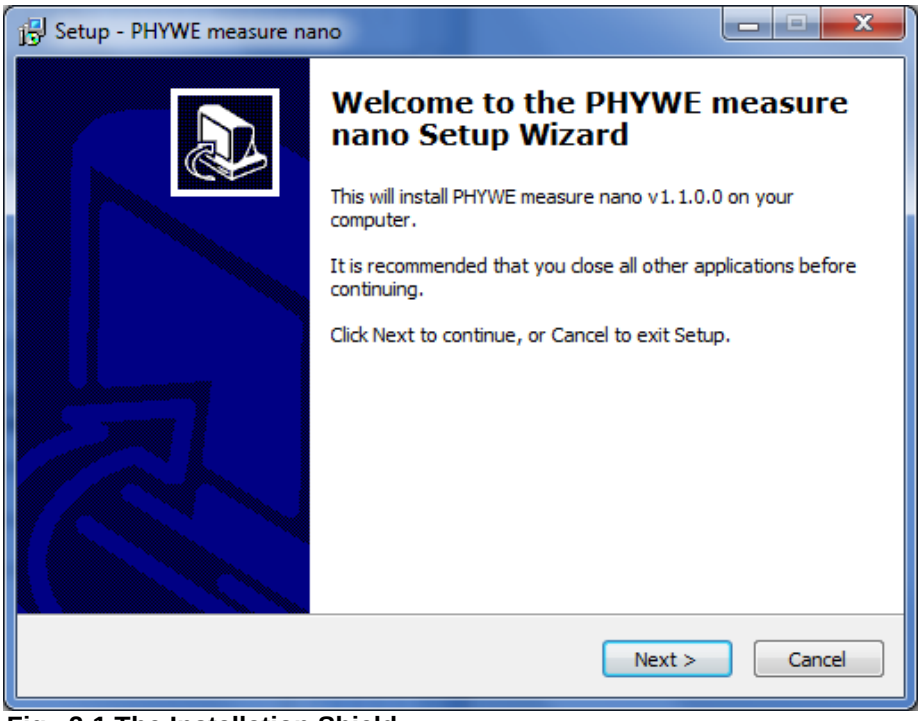


Fig. 2-1 The Installation Shield.

2.2 Setting up the Microscope

Place the instrument on a stable support (a very steady table or bench) in a location that has a low level of building vibrations (perhaps in the basement), acoustic noise (you should close the door and inform everybody around you to be as quiet as possible when they pass your set-up), electrical fields (set up your system several meters away from power switches or high powered machines), and air currents (don't set up under your air conditioning or near radiators).
Make sure that the mains power connection is protected against excess voltage surges (a power connection with built in security circuits is recommended). If you notice very high noise signals in your measurements later on you can try to protect your microscope by placing it under a Plexiglas or cardboard box.

2.3 Connecting the Microscope (Hardware recognition)

To initiate the automatic hardware recognition, follow these steps:

1. Log on to your computer with Administrator privileges.
2. Connect your computer with the supplied USB cable to the microscope control unit.
3. Press the power button on the side of your microscope next to the USB connection. A blue LED will light up.

A popup balloon appears in the Windows notification area, stating that a new hardware device has been found and drivers are being installed.

CHAPTER 3 :

The Software

Start the Measure Nano Software on your computer.

- Start you software by clicking on the program icon on your desktop.

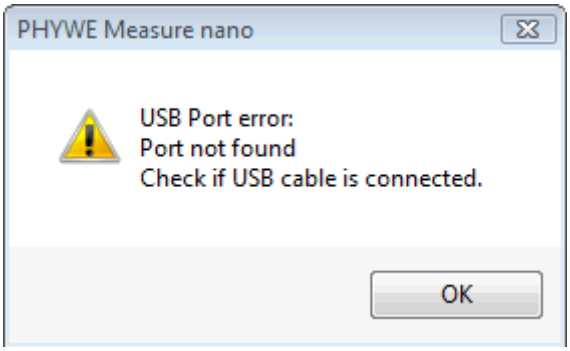
Or:

- Click the Windows “Start” button, then click “Program”, then open the “PHYWE Systeme” Menu.
- Open the “Measure Nano” menu and select the program “Measure Nano”

Now your software starts up.

3.1 Simulate microscope

The Measure Nano software can be started without having the microscope connected to your computer in order to explore the Measure Nano system (measurements and software) without danger of damaging the instrument or the STM tip. In simulation mode, most functions of the real microscope are emulated. The sample is replaced by a mathematical description of a surface. When the Measure Nano software is started without a microscope connected to your computer, the dialog on the right appears:



> Click “OK”.

The status bar will now display the text “Simulation”.

You can also switch to the simulation mode with the microscope connected:
> Select the menu entry “Settings” >> “Simulation”.
The menu entry be highlighted if simulation mode is active.

To exit the Microscope simulation mode:
> Select the menu entry “Settings” >> “Simulation” again.
The menu entry will no longer be highlighted, and the status bar will now display the text “Online”.

If you have the microscope connected to your computer:
A firmware update dialog could appear the first time you start your system. Please wait until this process is finished.

When a dialog “No connection to Microscope” appears, please check if your controller is switched on and the USB cable is connected between controller and computer properly.

3.2 Software interface

This chapter explains the general concept of the user interface of the STM Control Software. The software provides all functions to operate the microscope during imaging of surfaces and more advanced operating modes. It also provides data analysis functions for post-processing of measurement data.

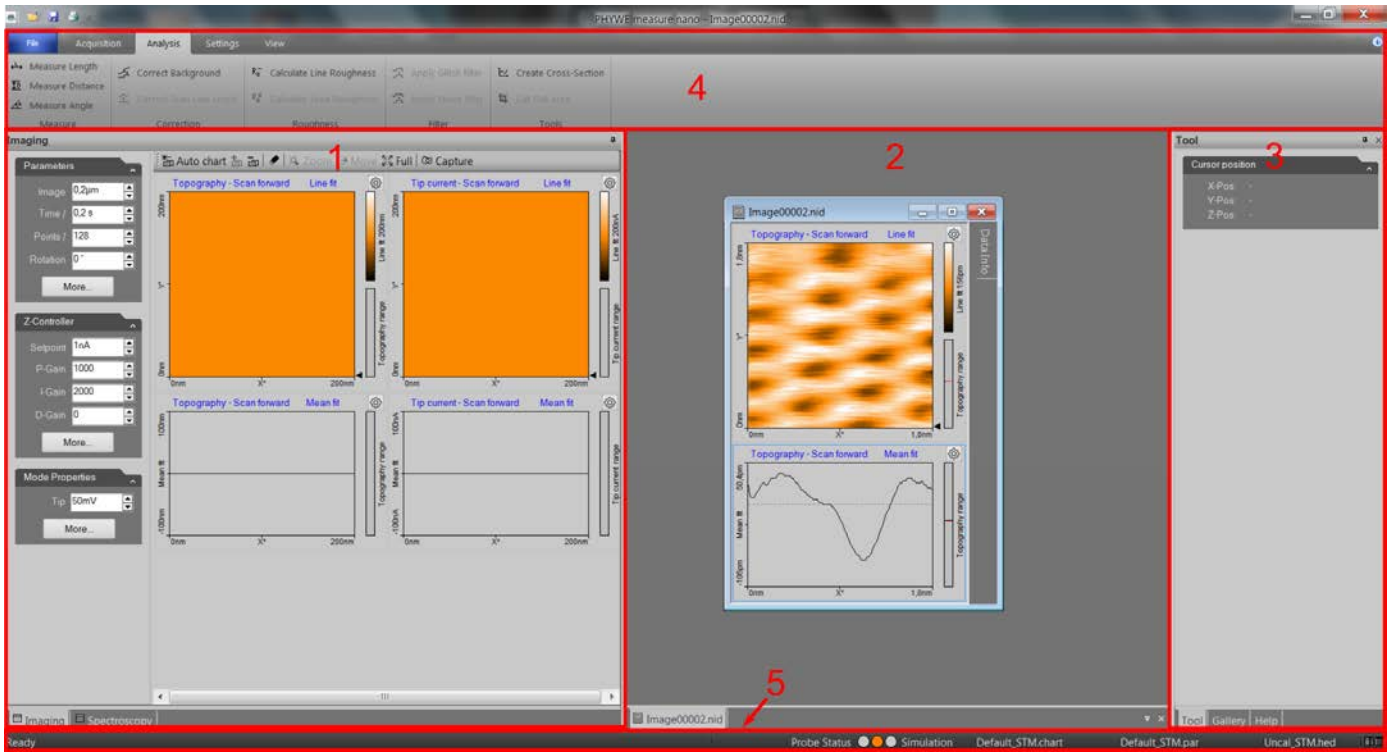


Fig. 3-1 The Software Interface in "Normal" workspace mode.

The main STM Control Software window (also referred to as workspace) consists of five major areas:

(1) The Measurement pane on the left. This area contains the so-called *Operating windows*, which are used to acquire and display ongoing measurement data
(2) The *Document space* in the middle. This area is used for displaying and analyzing previously stored measurement documents.
(3) The Info pane on the right. This area contains several stacked *Panels* and is used to group a diverse array of functionality and information.
(4) The *Ribbon* at the top. This area is used to access all action functions.
(5) The *Status bar* at the bottom. This area is used to display additional information.

3.2.1 Changing parameters in any panel

- Activate the parameter by clicking it with the mouse pointer, or by selecting it with the "Tab" key.

In case of a drop-down menu selection, change the selection with the mouse, or the "Up" and "Down" arrows on the keyboard. In case of a numerical value, use one of the following methods:

- Use the "Up" and "Down" arrow keys of your keyboard to increase or decrease the value of a parameter. The new value is automatically used after one second.
- Click the arrow buttons next to the parameter's value with the mouse pointer. The new value is automatically used after one second.

Enter the new value using the keyboard. The entered value is applied by pressing the “Enter”/“Return” key, or by activating another input.
The entered value is discarded by pressing the “Esc” key. The unit prefix can be changed by typing one of the following keyboard keys:

f = femto	k	= kilo
p = pico	M (shift-m)	= mega
n = nano	G (shift-g)	= giga
u = micro	T (shift-t)	= tera
m = milli	space bar	= no prefix

Example: If the basic unit is volts, type “m” to change to millivolts, or type “u” for microvolts. Sometimes the program changes an entered parameter value to a slightly different value. This happens when the desired value is outside the digitization range of the Measure Nano Controller, for example due to resolution or timing limits. In such cases, the desired value is automatically changed to the nearest possible value.

3.2.2 The workspace

With the PHYWE Measure Nano Control Software, measurement of newly acquired data and analysis of already stored data (in multiple documents) can be performed in parallel, since these tasks are partly performed in different areas of the workspace. It may however require a high resolution monitor (or multiple monitors) to make this process efficient. To offer the same functionality on systems with a limited resolution, the user can switch between a “Normal” (*Figure 3-1: The main window in “Normal” workspace mode*) and a “Document” mode (*Figure 3-2: The main window in “Document” workspace mode*). See also *Section 3.2.8.1: Workspace group* (page 17).

In “Normal” mode, the emphasis lies on the Measurement and Info panes. The inside border of the two panes can be dragged by the mouse to adjust their individual widths to your needs. Document space on the other hand is rather limited (see *Figure 3-1: The main window in “Normal” workspace mode*). This mode is most suited for measurements.

In “Document” mode, the Document space is maximized while the Measurement and Info panes are minimized to the left and right side of the main window, respectively (see *Figure 3-2: The main window in “Document” workspace mode*). The various window and panel titles are shown in tabs so that you can still open them when needed. A click or a mouse-over on one of these tabs will cause the respective window or panel to slide out automatically, so that you can work on it. It will automatically minimize again when you are done. This mode is most suited for analyzing stored measurement data.

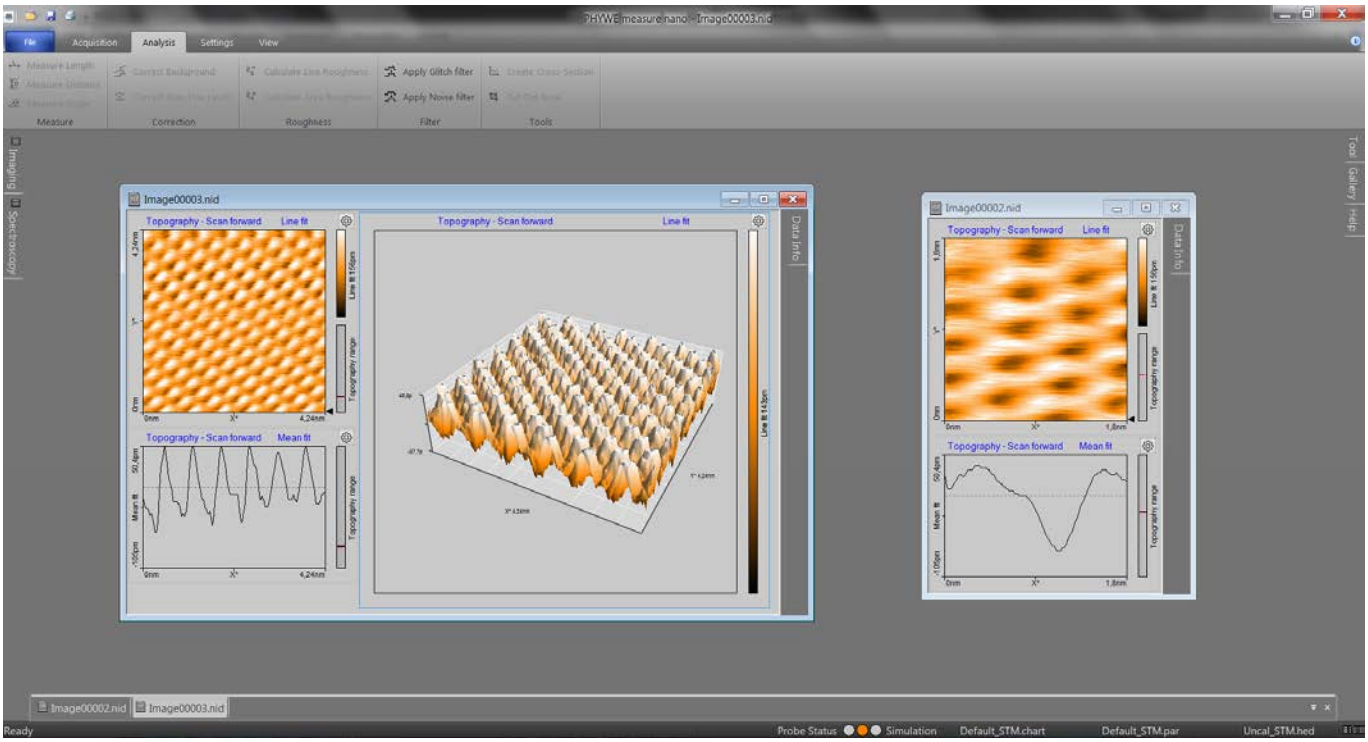


Fig. 3-2 The Main Window in "Document" workspace mode.

3.2.3 Operating window

Operating windows are used to perform specific operations with the microscope. The Operating windows are grouped together in the Measurement pane and can be accessed by clicking the respective tab. The operations themselves are usually controlled using the action buttons of the Ribbon. The Operating windows are:

- The Imaging window; used for generating images of a sample (for details see *Chapter 4.1: Imaging panel* (page 21)).
- Spectroscopy window; used for measuring various "A as a function of B" curves at certain sample locations, such as force-distance curves or current-voltage curves (for details see *Chapter 12: Spectroscopy mode* (page 107)).

All Operating windows contain three distinct elements, which are described in *Figure 3-3: Elements of Operating windows* and in the next chapters:

1. The "Parameter area", where the main parameters influencing the current measurement are grouped into different sections.
2. The "Chart area", where one or more charts, showing different aspects/signals of the current measurement, are being displayed.
3. The "Chart toolbar", where several functions that directly influence the current measurement (or display of it) are located.

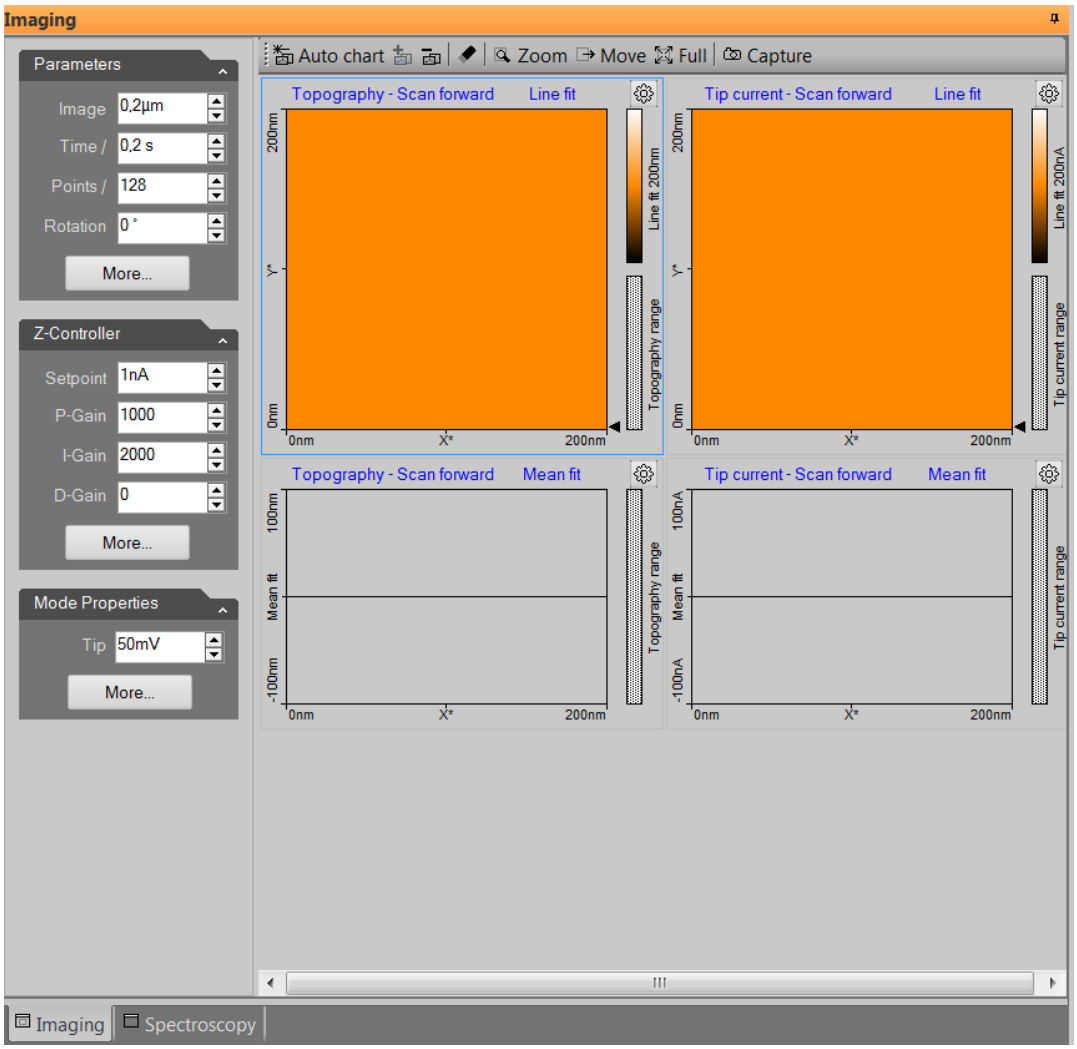


Fig. 3-3 Elements of Operating windows: Shown here is the Imaging window, with the Parameter area (Imaging panel) on the left, the Chart area on the right, and the Chart bar on top. Clicking the tabs at the bottom of the Measurement pane switches between the operating windows.

3.2.4 Document space

In the document space, stored measurements can be displayed for evaluation and analysis. Each measurement is contained within its own document window. These windows can be arranged in document space to your liking.
By default, all measurements are temporarily stored (automatically) during imaging and spectroscopy. They can be opened at all times from the Gallery panel (see *Section 4.3.3: Gallery panel* (page 36)), but should be moved to a new folder for permanent storage as soon as you have finished measuring (see *Save as* (page 70)).
Everything related to documents is described in more detail in *Chapter 4.3: Working with documents* (page 27).

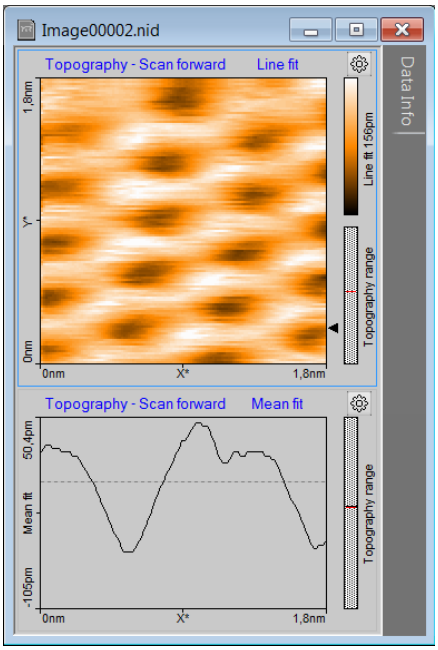


Fig. 3-4 Example of a measurement document window.

3.2.5 Panels

In the panels of the Info pane, the control software provides additional information that can be useful to the user. These panels are normally docked to the Info pane and are stacked to save space. The panels have several features, however, that allow you to arrange them in a way that is most efficient for your application (see *Figure 3-5: Arranging panels*).

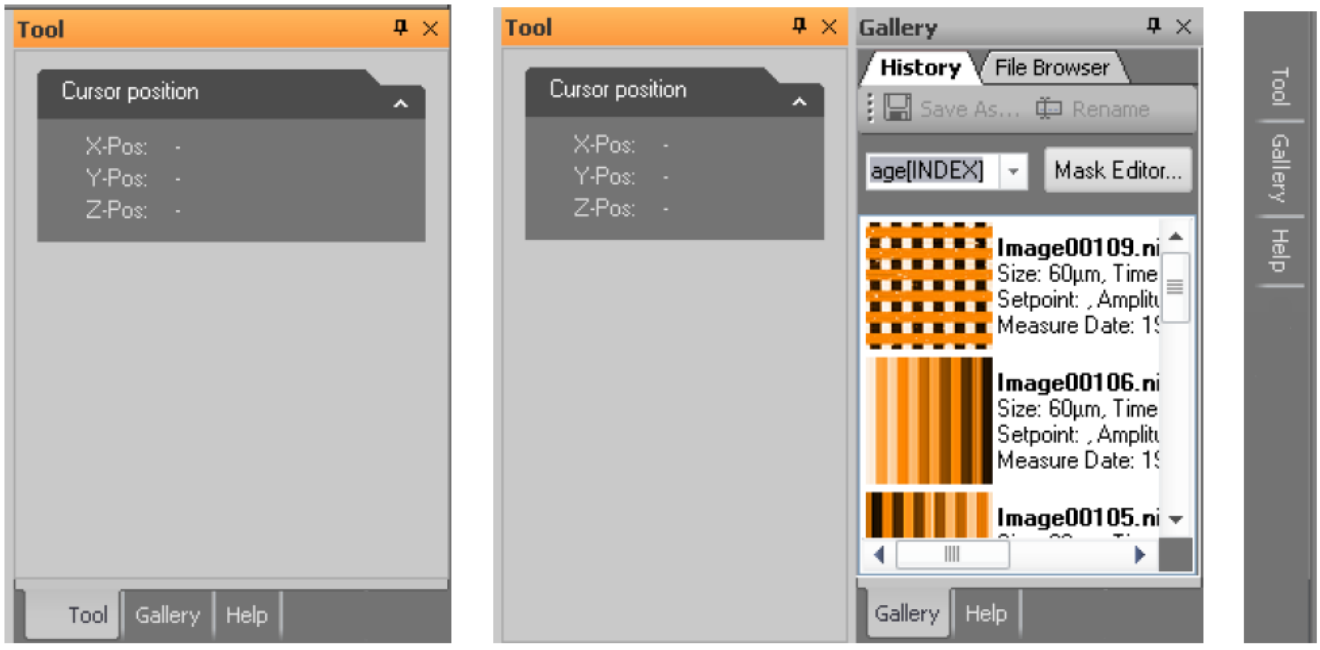


Fig. 3-5 Arranging panels. (Left) Staked panels. (Center) Separated panel that is locked to a stack. (Right) Panels that are minimized in “Document” mode.

To separate a panel and dock it individually to the side of/below another panel that is already docked to this window, drag its title bar to the desired position using the mouse cursor.

To add a control panel to a stack, drag either its title bar or its label to either the title bar or labels of the stack. To remove a panel from a stack, drag its label away from the stack.

When panels are stacked, their title labels are displayed on the bottom of the Info pane. To move a control panel to the top of the stack, click its tab.

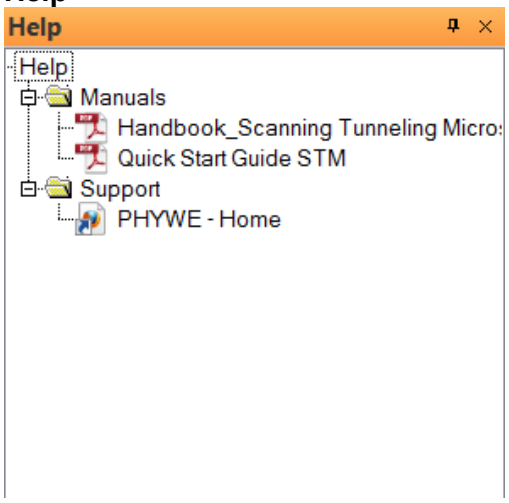
With the “pin” button ( ) in the tile bar of the individual panel or the Info pane, the auto hide feature is controlled. If “unpinned”, the panel or the Info pane minimizes to the right border of the main window and only the panel titles are visible (similar to the “Documents” workspace mode, but now only for the panel/Info pane and not for the Measurement area). A mouse hover over (or click on) a title tab will slide this panel into view.

It is possible to scroll the content of a control panel up and down, when it is too small to display all the parameters it contains. To do this, move the mouse cursor over an area where it changes to a four pointed arrow. Then, drag the content up and down with the mouse.

Tool
The Tool panel contains the results of the various analysis tools available to you during and after measurement, displays the current mouse position during selections, and displays the size of those selections (e.g. during zooming). The Tool panel is described in more detail in *Section 4.3.5: Tool panel* (page 50).

Gallery
The Gallery panel displays a list of stored measurements for quick opening (viewing and analysis). A File Browser is also integrated for general file management tasks. The Gallery panel is described in more detail in *Section 4.3.3: Gallery panel* (page 36).

Help



The Help Panel provides quick access to PDF versions of the user manuals belonging to your system, to relevant application notes and technical notes, and to online sources of information (direct links to the PHYWE website).

3.2.6 Ribbon

The Ribbon provides access to all major actions and commands by grouping them according to their usage.

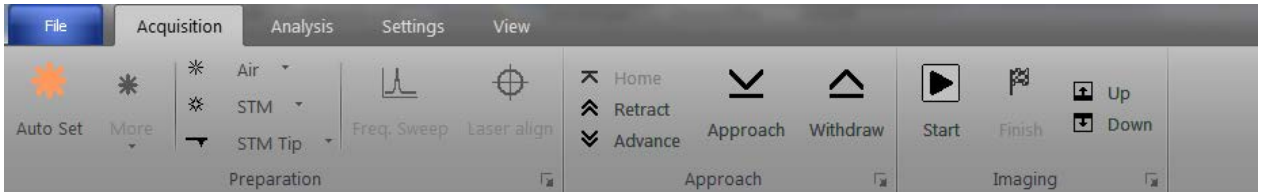


Fig. 3-6 The Ribbon.

The File menu
The File menu contains commands to open, save and print measurements. Other files such as those containing parameter settings or chart properties can be loaded or saved here as well. The file menu also provides data export functions. General program settings are configured through the Options dialog, which is opened by clicking the “Options” button of the File menu. The File menu is described in *Section 11.2: File menu* (page 89).

The Acquisition tab
Guides you through the measurement process. There are groups of buttons for measurement preparation, sample approach and the measurement itself. The Acquisition tab displays different buttons for each of the Measurement windows (*Imaging*, *Spectroscopy*) and is therefore described in their respective Chapters.

The Analysis tab
Contains measurement data functions for extracting information from your measurements (e.g. “step height” or “roughness”). It also provides functions to permanently modify your image data (e.g. “noise filtering”. All of these functions are described in *Section 4.3.4: Analysis tab* (page 41).

The Settings tab
Contains functions to configure the microscope controller hardware and calibrating the scan head. It is described in *Section 11.4: Settings tab* (page 92)

The View tab
Provides access to the workspace modes “Normal” and “Documents” (see *Section 3.2.4: Document space* and *Section 3.2.8.1: Workspace group*), the Panels of the Info pane, and Document window arrangement options. Since it has a great impact on the overall look of the user interface of the SPM Control Software, it is described below (see *Section 3.2.8: View tab*).

3.2.7 Status bar

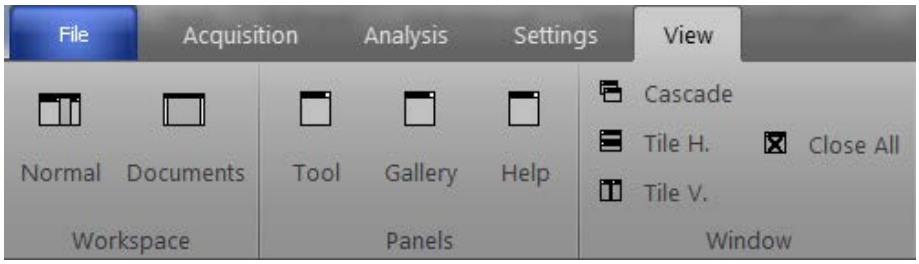
Fig. 3-7 The Status bar. Numbers in this figure correspond to those in the list below.

The status bar displays relevant microscope information and the loaded settings (see *Figure 3-7: The Status bar*). It contains the following elements:

1. Help text information about the current menu button or latest error messages
2. Status signal of the Z-feedback controller (Orange: No contact. Green: Tunneling current is flowing with the pre-set Set Point. Red: Direct contact to the surface, it is likely that tip got damaged).

3. Software status: “Online” or “Simulation” (depends on the presence or absence of a scan head, or on user choice).
4. Currently loaded file (“.chart”) used for chart settings.
5. Currently loaded file (“.par”) used for parameter settings.
6. Currently loaded scan head calibration file (“.hed”).
7. Buttons to access the Workspace “Normal” and “Document” view.

3.2.8 View tab



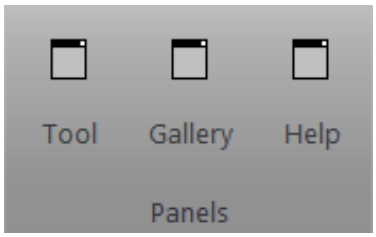
3.2.8.1 Workspace group

The workspace group in the view tab gives you the ability to switch between the two workspace modes “Normal” and “Documents”. To switch, click on either of the buttons, or on the corresponding smaller buttons () on the right-hand side of the status bar (see also *Section 12.7: Status bar*). With these you don't even need to switch to the view tab while measuring.

Normal
The optimal workspace choice during measurements.

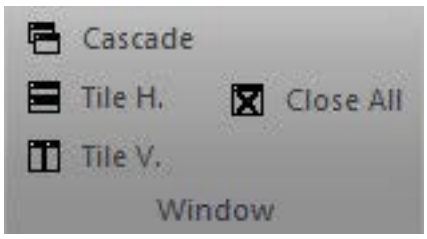
Documents
The best choice for viewing or analysis of stored documents (see *Chapter 4.3: Working with documents* (page 27)).

3.2.8.2 Panels group



The buttons of the Panels group have the same function as the tabs at the bottom of the Info pane (i.e., to bring the respective panel to the top of Info pane). If the panel was undocked from the Info pane (see *Section 12.5: Panels*) and subsequently closed (i.e., no longer visible as panel or tab), it will re-appear by pressing its button in the Panels group.

3.2.8.3 Window group



The window group provides you with tools to arrange open measurement documents in different ways or quickly close them all.

Cascade
Open document windows are stacked on top of each other and slightly offset with respect to each other so that individual windows can be easily accessed. Width and position of the windows is optimized by the control software.

Tile H.
Tiles the open document windows horizontally, so that individual measurements can be easily compared. Width of the document windows is maximized. Height is evenly distributed over the available document space.

Tile V.
Tiles the open document windows vertically, so that individual measurements can be easily compared. Height of the document windows is maximized. Width is evenly distributed over the available document space.

Close all
Closes all open document windows. If unsaved data exists, you will be asked to save it.

CHAPTER 4 :

Menus and Tools

Imaging measurements of the sample are controlled using the Imaging window which is shown in figure 4-1. This chapter describes all elements of the Imaging window in detail. For procedures describing a basic measurements, refer to *Chapter 5: Preparing the first measurements* (page 52). For details on how to use the charts see *Section 4.3.2: Charts* (page 28).

The Imaging window is contained within the Measurement pane and can be opened by clicking the Imaging tab. The largest part of the Imaging window consists of a number of charts that display the data from the ongoing measurement: The Chart area. The imaging window can display as many charts as required. Scroll bars will appear automatically as soon as the content is larger than the window can accommodate. By default, two charts groups are displayed: 2 color maps of the sample and their corresponding line graphs. Usually, these show Topography on the left and another measurement signal on the right (e.g. Tip current), depending on the current operating mode. For more information on adding and changing charts, which basically works the same for charts in Operating windows and in stored measurements documents, see *Chapter 4.3: Working with documents* (page 27).

The Parameter area on the left side of the Imaging window, the so-called Imaging panel, is organized in 3 sections: the “Parameters”, “Z-Controller” and “Mode Properties” section. These sections are an integral part of the Imaging window and represent the most commonly used parameters for the currently selected operating mode. Possible parameters in these sections are described in *Section 4.1: Imaging panel*. Advanced parameters can be accessed via the “More” button in each section, which will open the SPM Parameter dialog on the respective page (see *Section 11.5: SPM Parameters dialog* (page 97)).

At the top, the Imaging window contains a toolbar with commands to control the imaging process: The Imaging toolbar. It is described in *Section 4.1: Imaging panel*.

Apart from the necessary settings, several actions have to be performed before being able to image a sample. These are accessed via the Acquisition tab of the Ribbon, the elements of which are described in detail in *Section 4.2: Acquisition tab*.

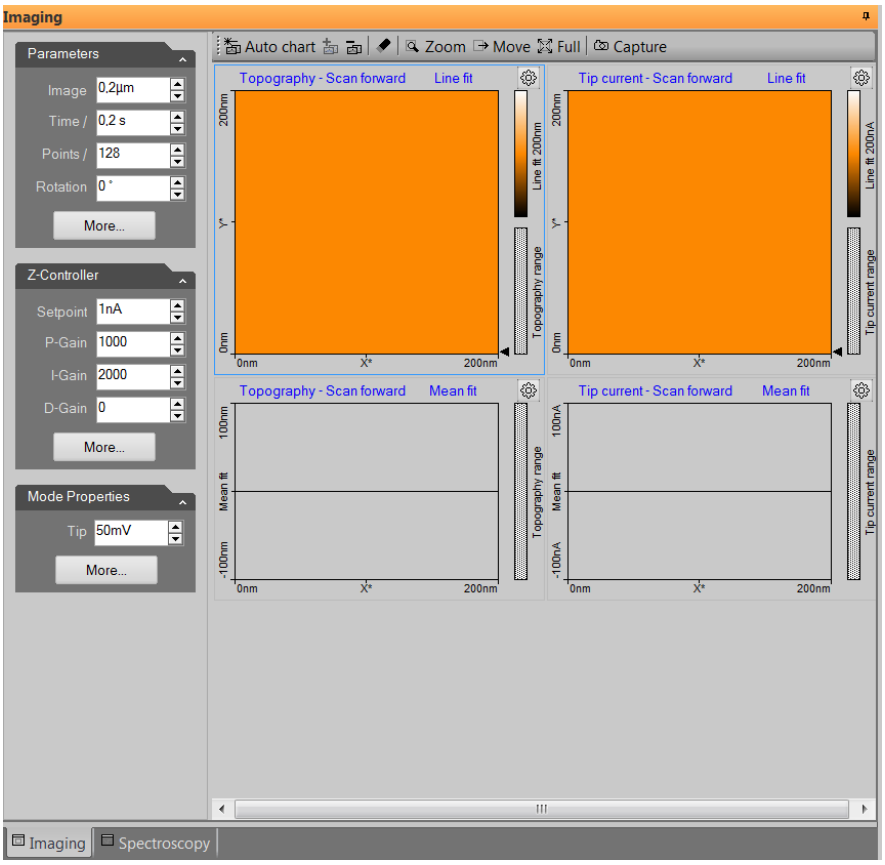


Figure 4-1: The Imaging window

4.1 Imaging panel

The imaging settings use two coordinate systems: the Scanner coordinate system and the Measurement image coordinate system. To separate the two systems, the image axes are denoted by an asterisk (i.e. X*, Y*). The relation between the two coordinate systems is determined by various parameters in the imaging panel. The effect of these parameters is illustrated in *Figure 4-2: Coordinate systems*.

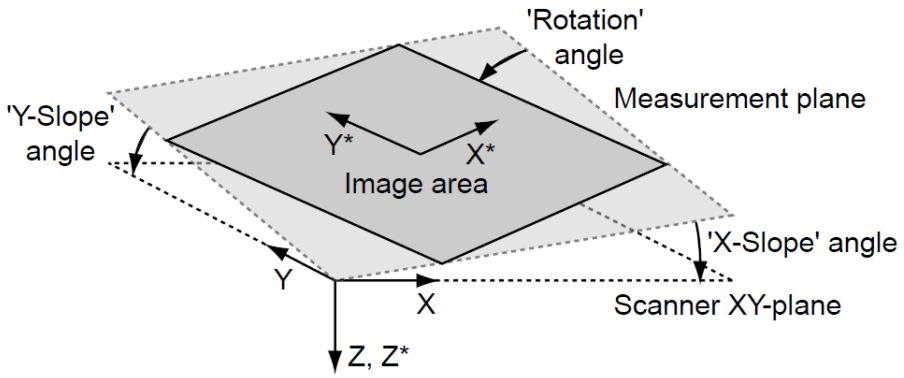


Figure 4-2: Coordinate systems

Imaging Parameter section

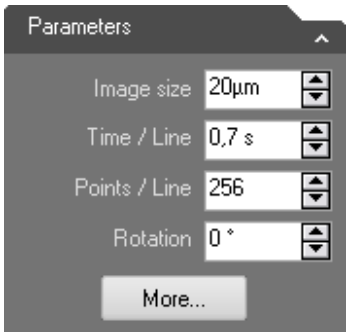


Image size
Defines the image size in both the X* and Y* direction. The size is doubled or halved when the arrows next to the edit box are used.

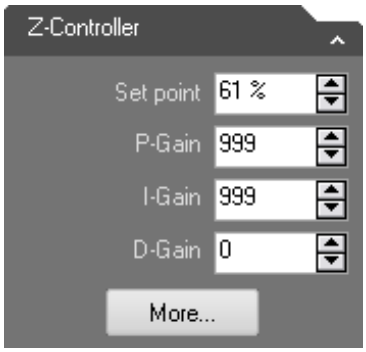
Time / Line
The time needed to acquire a single data line. The time needed for the entire image is displayed in the status bar.

Points / Line
The number of measured data points per line. It also sets the number of lines to the same value.

Rotation
The angle between the X-direction of the scanner and the X* direction of the measurement (Figure 4-2: Coordinate systems).

“More” button
Opens up the SPM Parameter dialog on the “Imaging” page for more advanced parameters (see *Section 11.5: SPM Parameter dialog* (page 97)).

Z-Controller section



During imaging, the tip–sample interaction is kept constant through the Z-Controller. The Z-Controller is a standard PID controller as is shown in *Figure 4-3: Z-Controller*.

Setpoint
The working point for the Z-Controller (i.e. the tunneling current).

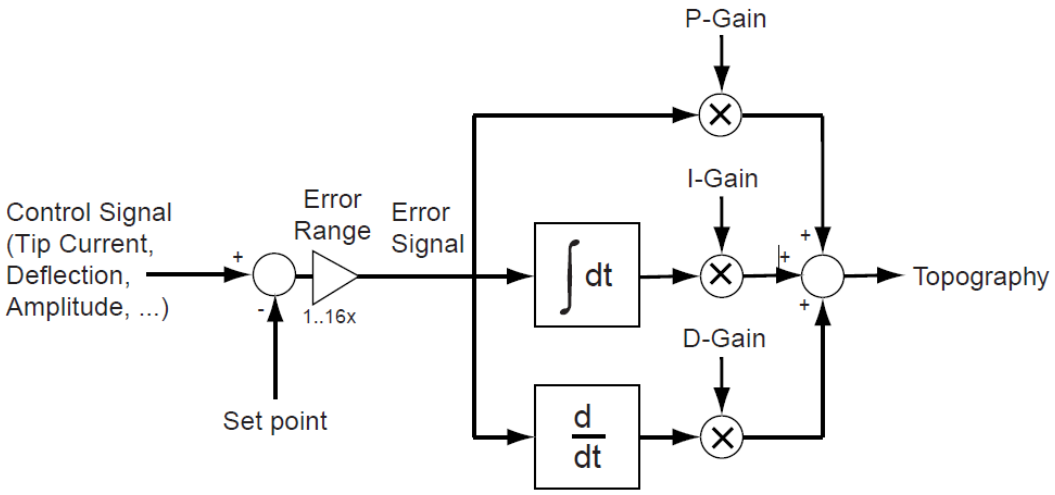


Figure 4-3: Z-Controller

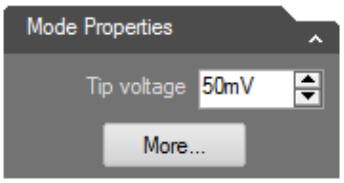
P-Gain
The strength of the Z-Controller reaction that is proportional to the error signal. Increasing the P-Gain decreases the error signal.

I-Gain
The strength of the Z-Controller reaction that is proportional to the integral of the error signal. Increasing the I-Gain decreases the error signal over time. It is the least sensitive to noise, and usually the dominant contributor to the topography measurement.

D-Gain
The strength of the Z-Controller reaction that is proportional to the derivative of the error signal. Increasing the D-Gain decreases fast changes in the error signal, but also amplifies high frequency noise.

“More” button
Opens the SPM Parameter dialog on the “Z-controller” page for more advanced parameters (see *Section 11.5: SPM Parameter dialog* (page 97)).

Mode Properties section



Tip Voltage
This parameter defines the potential to be applied to the tip. The voltage that can be used lies between -10V and +10V.

“More” Button
Opens up the SPM Parameter dialog on the “Operating Mode” page for more advanced parameters (see *Section 11.5: SPM Parameter dialog* (page 97)).

The imaging toolbar

“Auto chart” button
Using this button, the control software displays all meaningful charts for the currently selected operating mode. The actual number of charts varies depending on the mode.

“Plus” and “Minus” buttons
With these buttons you can add and remove chart groups, respectively, but not more than minimally makes sense. There will always be a chart group left. Conversely, you can't add more chart groups than the Auto tool would display. You can however still remove or add charts manually.

“Clear old chart data” (eraser) button
Deletes chart data from a previous measurement. Old chart data can be deleted at all times, regardless of whether a measurement is running or not.

Zoom
Selects an area that is to be measured in more detail. The size and area of the selected zoom area is displayed in the Tool Results panel.

The zoom area is defined by two opposite corners of the area. Pressing the left mouse button at the first corner and holding it down while moving the mouse pointer to the other corner will create a zoom area of user-defined size. Alternatively, an area that has a third of the current measurement size and a center at the current mouse pointer position is defined with a single mouse click at the desired zoom position. The area defined by the marker can be resized by dragging one of its corners, or moved to a new position by dragging its center point.

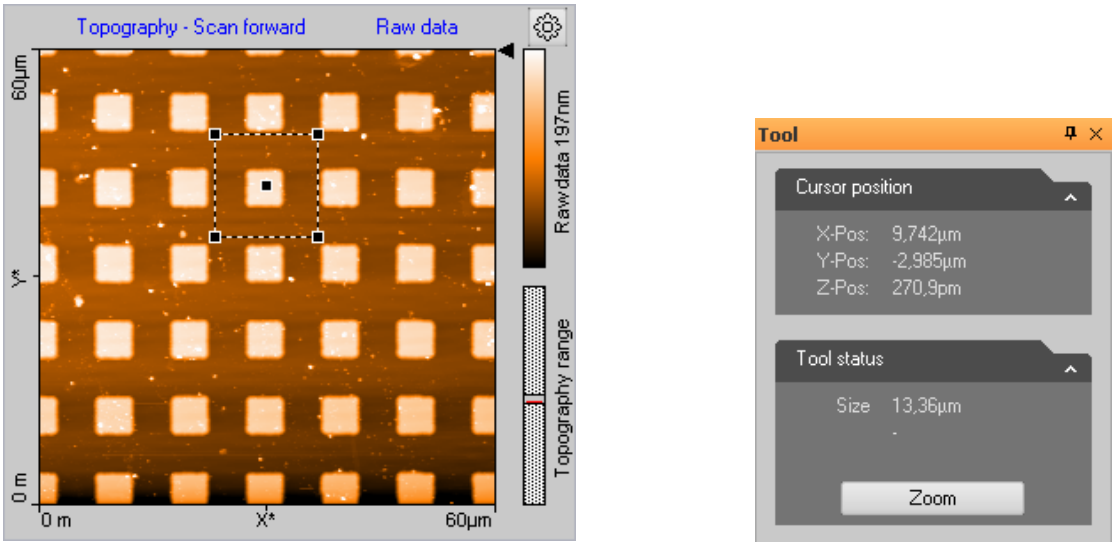


Fig 4-4 Zooming. *(Left) The Zoom tool area marker. (Right) The Zoom tool information in the Tool panel of the info pane*

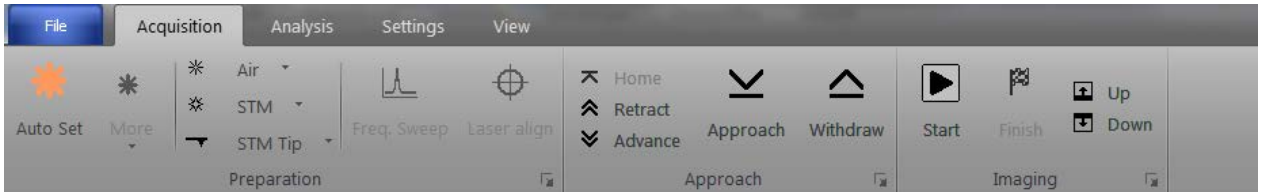
To accept the new zoom area:
> Double-click the chart with the left mouse button, or click the “Zoom” button in the Tool Results panel.
This action modifies the parameters “Image size”, “Image offset X” and “Image offset Y” in the Imaging page of the SPM Parameters dialog accordingly (see *Section 11.5: SPM Parameter dialog* (page 97)).
To abort the zoom function
> Click Zoom again, or use the right mouse button to select “Abort” in the context menu.

Move
The “Move” button moves the position of the imaged area. An interesting corner can thus be moved to the center of the measurement. The Tool Results panel numerically displays the change in position. The change in position is indicated by an arrow. The start of the arrow is defined by the mouse cursor position where the left mouse button is pressed; the end of the arrow by the position where the button is released. With a single click of the left mouse button an arrow ending in the center of the measurement is drawn. The direction of the arrow can be adjusted by dragging its end markers. It can be moved by dragging the center marker. The image is moved by double clicking, or clicking the “Move” button in the Tool Results panel. To abort the Move function, click the Move-Button again or click the right mouse button and select “Abort” in the context menu.

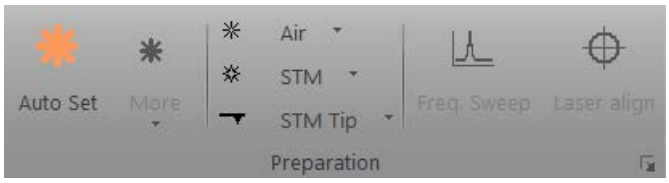
Full
The Full button returns the parameters Scan range to the largest possible values, and “X-Offset” and “Y-Offset” to zero (see *Section 11.5: SPM Parameter dialog* (page 97)).

Capture
With the “Capture” button, you can immediately copy the current measurement to the History page of the Gallery panel without waiting for the scan to be completed (see also *Section 4.3.3: Gallery panel* (page 36)). It is stored as a new document and remains open in the Document space of the SPM Control Software.

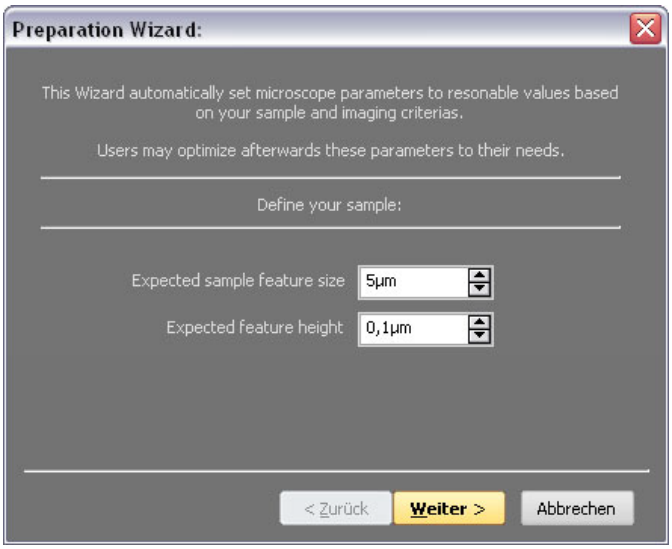
4.2 Acquisition tab



4.2.1 Preparation group



Auto set
The “Auto set” button opens the Parameter preparation wizard. This wizard sets the measurement parameters to reasonable values, based on the sample feature size and desired imaging quality that you enter:



Launcher icon
More advanced settings are available through the “Dialog Launcher” icon (at the bottom right corner of the Preparation group), which opens up the SPM Parameter dialog on the Operating Mode page (see *Section 11.5: SPM Parameter dialog* (page 97)).

4.2.2 Approach group

Home
Increases the tip–sample distance to its maximum value to ensure that the maximum motorized approach range is available during final automatic approach.

Retract
Increases the tip–sample distance at maximum speed until the button is released.

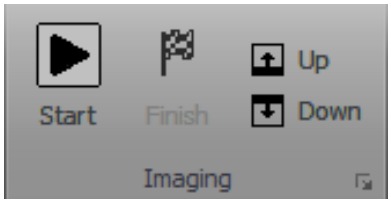
Advance
Decreases the tip–sample distance at maximum speed until the button is released.

Approach
Starts the automatic approach. During automatic approach, the tip–sample distance is decreased until the Setpoint (set in the Z-Controller section) is reached, or until the maximum number of approach steps is reached (see *Section 5.3: Approaching the Sample to the Tip* (page 58)).

Withdraw
Increases the tip–sample distance with approach speed settings.

Launcher icon
More advanced settings are available through the “Dialog Launcher” icon (at the bottom right corner of the Preparation group), which opens up the SPM Parameter dialog on the Approach page (see *Section 11.5: SPM Parameter dialog* (page 97)).

4.2.3 Imaging group



Start
Clicking “Start” starts a measurement and changes the button to “Stop”. Clicking “Stop” aborts the measurement as soon as the current scan line is finished.

Finish
Selecting “Finish” will set the “Finish” flag, which will not abort the measurement directly, but will do so when the measurement is finished. Deselecting (i.e., clicking it again) will disable the “Finish” flag so that the measurement will no longer stop automatically when it is finished. The “Finish” button is highlighted when it is flagged.

Up / Down
Starts a single measurement or restarts an ongoing measurement from the selected scanning direction. With the “Up” button the image is scanned from bottom to top. With the “Down” button it is scanned from top to bottom.

Launcher icon
More advanced settings are available through the “Dialog Launcher” icon (at the bottom right corner of the Preparation group), which opens up the SPM Parameter dialog on the Imaging page (see *Section 11.5: SPM Parameter dialog* (page 97)).

4.3 Working with documents

When working with the PHYWE Measure Nano Software, all finished measurements (a full image was recorded) will be temporarily stored according to the file name mask you specified in the Gallery panel (see *Section 4.3.3: Gallery panel*). These measurement documents can be opened and displayed in the

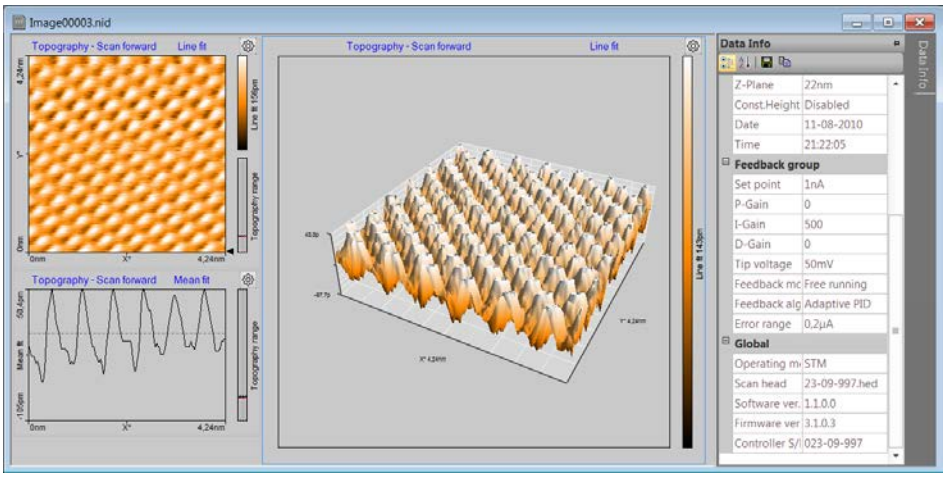


Figure 4-5: Measurement document. Typical measurement window with the Data Info panel expanded.

document area of the SPM Control Software's workspace (see *Section 3.2: Software interface* (page 10). It is strongly recommended to permanently store relevant documents to a new folder (see *Save as* (page 70). Charts and the Data Info panel together display all available measurement information.

4.3.1 Data info panel

The Data Info panel (minimized by default, but expanded upon hovering of the mouse cursor over the Data Info tab on the right side of the measurement document window) displays measurement settings and the hardware used during the measurement. Its content is self-explanatory and will therefore not be discussed in this manual.

Just like the panels of the Info pane, the Data Info panel can be "pinned" and "unpinned" to disable or enable the Auto-hide function of the panel (see *Section 3.2.5: Panels* (page 14)). It cannot be undocked from the document window, however.

The Data Info panel also contains a small toolbar, which allows you to customize the presentation of the data and offers ways to export it.

Data Info toolbar

Categorized
The "Categorized" button () will group the data entries for the measurement document by category. This is the default display method of the Data Info panel.

Alphabetical
The "Alphabetical" button () will sort the data entries for the measurement document alphabetically.

Save
The "Save" button () will save the information in the Data Info panel to file. Possible formats are text (.TXT) and comma separated values (.CSV) files.

Copy to Clipboard
The "Copy to Clipboard" button () will copy the entries of the Data Info panel to the Windows clipboard for easy pasting into other applications.

4.3.2 Charts

Charts provide a graphical display of the measured data. Charts occur in Measurement document windows, in Operating windows, and in various other windows and dialogs. You can adjust them to your needs and liking. How to do this is explained in this section. This information is valid for charts in stored measurement documents as well as for ongoing measurements in one of the Operating windows (*Imaging and Spectroscopy*).

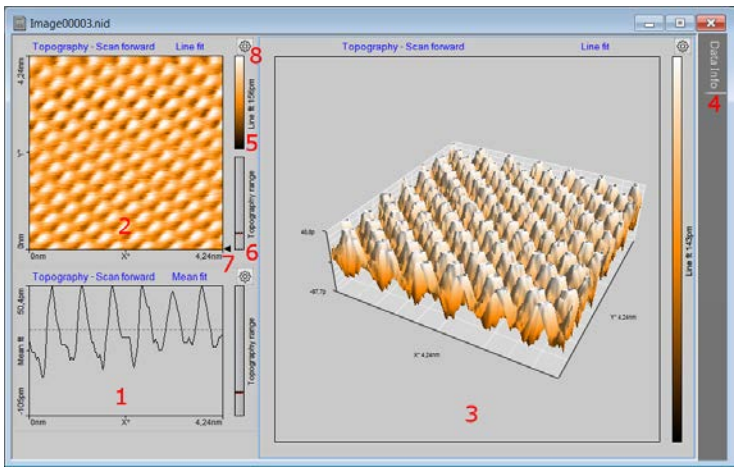
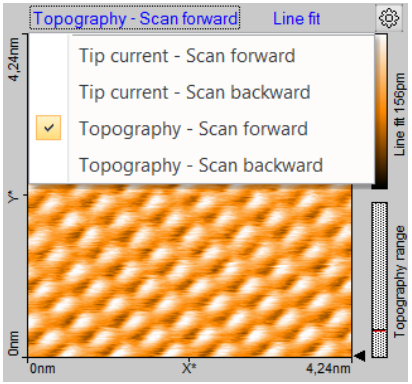


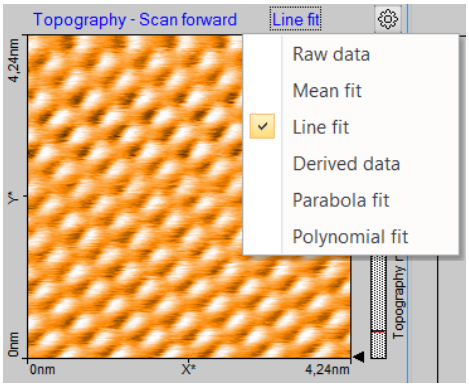
Figure 4-6: Elements of a chart. (1) Line graph. (2) Color map. (3) 3D view. (4) Data Info panel (see *Section 16.1: Data Info panel*). (5) Color scale for data Z-range. (6) Data range indicator, with scan head Z-range as dotted box, data Z-range as solid gray box and current scan line height as red line. (7) Line selection arrow. (8) “Chart Properties” button.

A Chart consists of a graphical representation of the measured data itself and elements that provide additional information. There are three basic chart types: Line graph, color map and 3D view (see *Figure 4-6: Elements of a chart*, items 1–3).

Chart titles

The title elements of each chart display the signal name and the background line filtering type that is used. A click on each of these titles opens a drop-down menu with other possible signals or filters:





Changing the titles will change the content of the chart.

Color scales

The Color scale (*Figure 4-5: Elements of a chart*, item 5) shows which measured signal level is mapped to which color. The color mapping can be changed using the Color Palette dialog (see *Section Color Palette* (page 91)).

Data range indicator

The Data range indicator (*Figure 4-5: Elements of a chart*, item 6) shows the Z-range of the scan head and of the values occupied by the measured data, and the current scan line height.

Hovering with the mouse cursor over the Color scale or Data range indicator of a color map chart opens a height histogram graph and two range selectors:

This histogram displays the current height distribution of the measurement data and the used color range. With the top and bottom range selectors, the color bar range can be adjusted to the actual height distribution of the measurement data. Changing these range settings changes the “Center” and “Span” parameters of the Chart Properties dialog (see *Section 4.3.2.2: Chart Properties dialog*) and immediately updates the color display of the data in the chart.

Line selection arrow
With the Line selection arrow (*Figure 4-5: Elements of a chart*, item 7) the shown data line on line charts displaying the same signal can be changed by holding down the left mouse button over the arrow and move the mouse up or down.

Chart properties
The “Chart Properties” button ( ); *Figure 4-5: Elements of a chart*, item 8) opens the Chart properties dialog. This dialog is the center of all chart parameters and is described in more detail in *Section 4.3.2.2: Chart Properties dialog*.

Most chart settings can also be accessed from a context menu, which is opened by right-clicking a chart.

4.3.2.1 Working with multiple charts

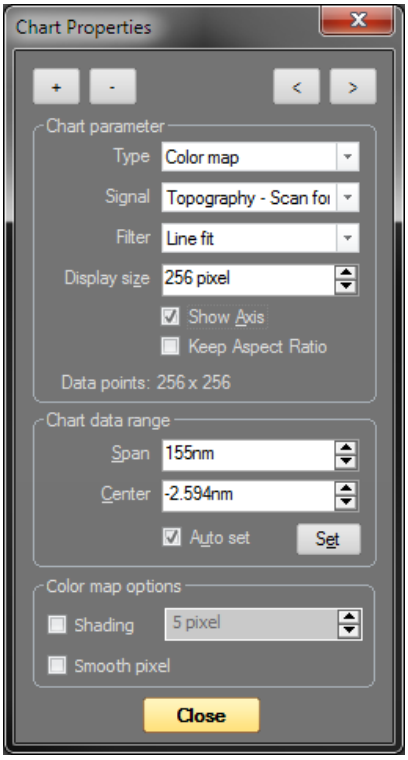
In some windows, multiple charts can be displayed and configured by the user at any time (e.g. in the Imaging window or a Document window). The same signal can be displayed in different styles (e.g. Line Graph and Color map) and/or multiple signals can be shown side by side (e.g. Topography and Tip Current Signal).

Adding or removing a chart, or setting chart parameters is all performed in the Chart Properties dialog (see *Section 4.3.2.2: Chart Properties dialog*). When opened, the settings displayed in the Chart Properties dialog refer to the currently selected chart. This (active) chart is indicated by a thin blue line around the chart area. A chart is activated by clicking on it with the mouse cursor anywhere in the chart area.

Arrangement of the charts is performed automatically by the control software based on the size of the window and based on the order in which the charts were generated/added. If the window is too small to display all charts, scrollbars are displayed at the border of the window.

Short cuts to add and remove charts are found in the chart context menu. Select "Create new chart" or "Delete current chart" from the menu list. It is also possible to use the "Insert" or "Delete" key of your computer’s keyboard for this task.

4.3.2.2 Chart Properties dialog



The Charts Properties dialog is used to set all chart properties that influence data display by the respective chart. It may be kept open at all times if many parameters have to be set for different charts.

Some parameters are chart type specific. They are therefore displayed at the bottom of the Chart Properties dialog in a separate group.

Add chart
The “Add Chart” button (+) creates a copy of the currently selected or active chart and adds it to the active window in last position.

Remove chart
The “Remove Chart” button (-) removes the currently active chart.

Previous chart
The “Previous Chart” button (<) activates the previous chart and updates the parameters displayed in the Chart Properties dialog to those of that chart.

Next chart
The “Next Chart” button (>) activates the next chart and updates the parameters displayed in the Chart Properties dialog to those of that chart.

Chart parameters

Type
Selects the chart type to be used for display of the measurement data:

- **Line graph** Data is displayed as a line plot. Points outside the range of the scanner are displayed in red. The line being displayed is selected by dragging the Line selection arrow in a Color map (see *Line selection arrow* (page 30)). In ongoing measurements (e.g. during imaging) the position of the Line selection arrow is updated automatically and corresponds to the last measured scan line (but even here it is possible to select a different line for view in a line graph by dragging/holding the Line selection arrow in a different location).

- **Color map** Z-height data is encoded using a color scale and displayed 2-dimensionally.

- **3D view** Data is shown in a 3-dimensional representation in parallel perspective. Color information (such as implemented in the Color map) is maintained.

- **Signal** Selects the input channel (signal data) to be used for the chart. The available signals depend on the operating mode (selected or used) and the status of the User inputs.

- **Filter** Selects the line filter method. The control software applies this filter to the measured data before displaying it (see *Figure 4-7: Data filter types*). No modification of the original measurement data occurs (selecting another filter is always possible). Available data filters are:
    - o **Raw data** No data processing.
    - o **Mean fit** Calculates the mean value of each line of data points and subtracts this number from the raw measurement data for each data point of that line.
    - o **Line fit** Calculates the first order least squares fit (mean value and slope) for each line of data points and subtracts the fitted values from the raw measurement data for each data point of that line.
    - o **Derived data** Calculates the difference between two consecutive data points (derivative) and displays this instead of the raw image data.
    - o **Parabola fit** Calculates the second order least squares fit for each line of data points and subtracts the fitted values from the raw measurement data for each data point of that line.
    - o **Polynomial fit** Calculates the fourth order least squares fit for each line of data points and subtracts the fitted values from the raw measurement data for each data point of that line.

- **Display size** Sets the size of the chart in pixels.

- **Show Axis** When checked (default), the axis labels, color and range scales, and titles are displayed alongside the graph. When unchecked, they are hidden.

- **Keep Aspect ratio** When checked, the axis in the color map are drawn in their correct size-relation (according to their value and unit). When unchecked (default), the size of the display is always a square and data pixels are stretched if necessary.

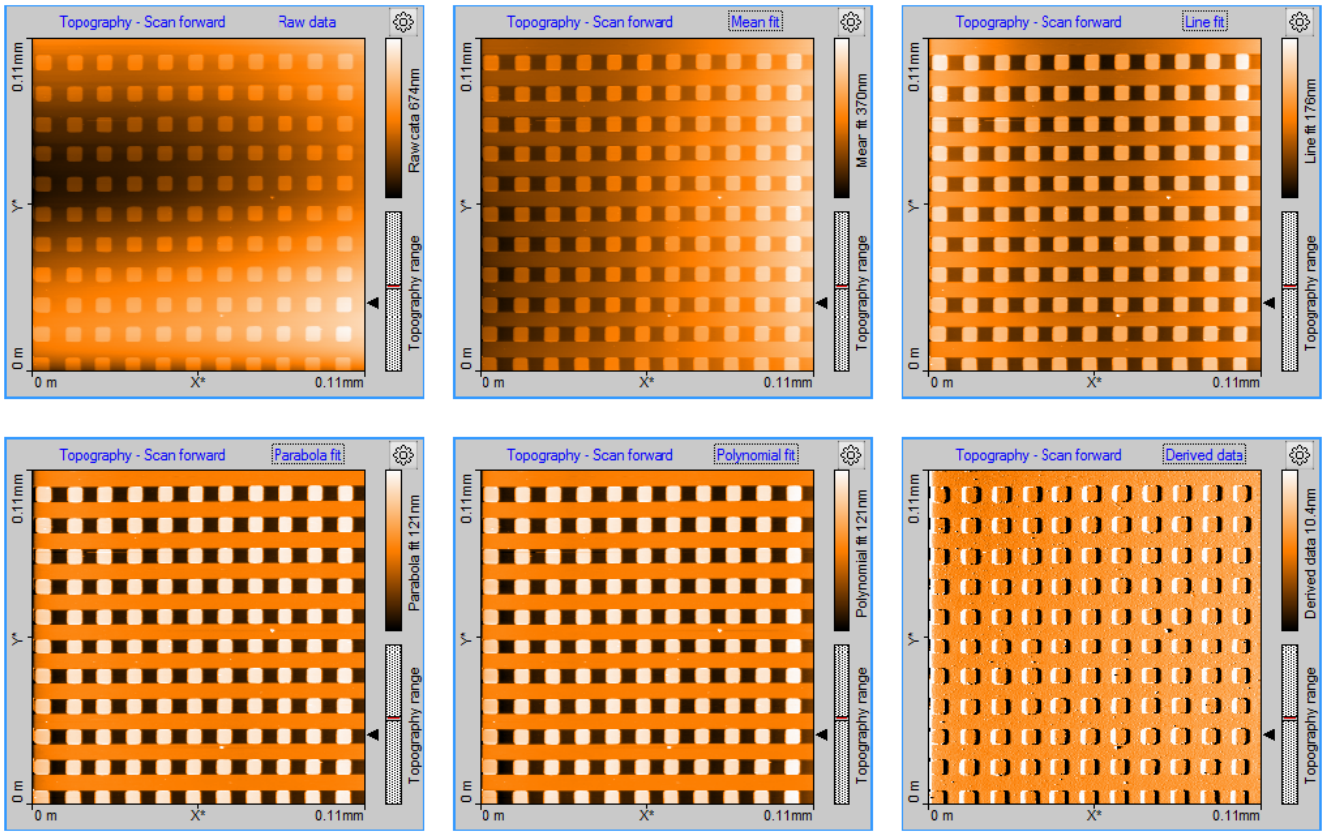
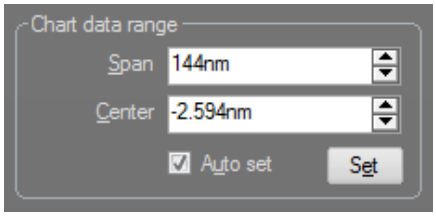


Figure 4-7: Data filter types. The same measurement data displayed using the available filters. A defective area on a calibration grid is shown here to illustrate the effect of the filters.

Chart data range



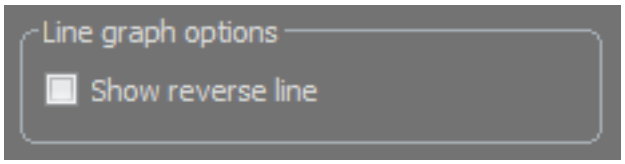
Span
The span that corresponds to the chart's displayed Z-range. Increasing Span decreases feature contrast and vice-versa. The current span is displayed next to the color scale in color maps, or can be inferred from the Z-axis labels in Line graphs and 3D views.

Center
The signal value that corresponds to the center of the "Span" parameter.

Auto set
When checked, the chart's Z-range is automatically set to optimally match the measurement data. During measurements, the Span and Center parameters will be updated continuously (i.e., the chart adapts to the available data).

Set button
Clicking this button starts the optimization of the Z-range manually. Mostly used when "Auto set" is off.

Line graph options



Show reverse line
When checked, the reverse scan data is drawn in gray (see *Figure 4-8: Show reverse line option*. It allows comparison of the forward and reverse measurements data. Whether or not this data is available depends on the measurement mode used during the acquisition of the data (see Chapter 11.6: *Operating modes* (page 106)).

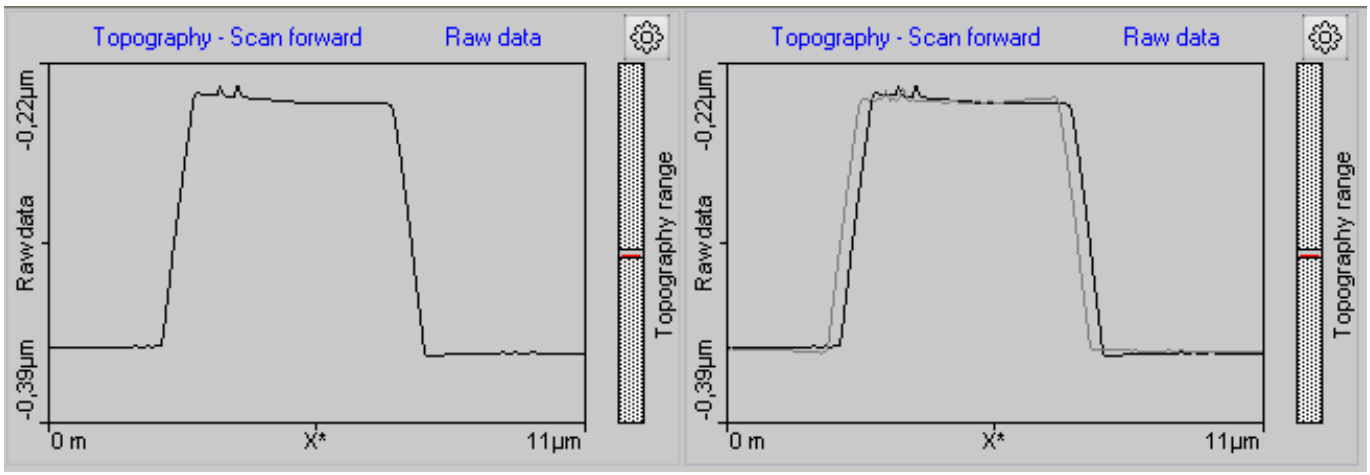
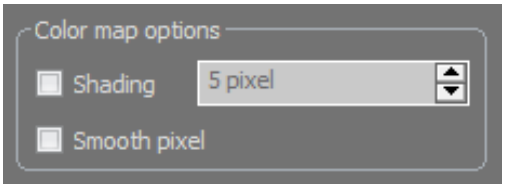


Figure 4-8: Show reverse line option. (Left) Reverse line disabled. (Right) Reverse line enabled.

4.3.2.3 Color map options



Shading
When checked, the color map creates the impression of a 3-dimensional surface which is lighted from the left. This is achieved by combining the topography with its derivative. The number of pixels in the edit box defines the amplification of the derivative add on.

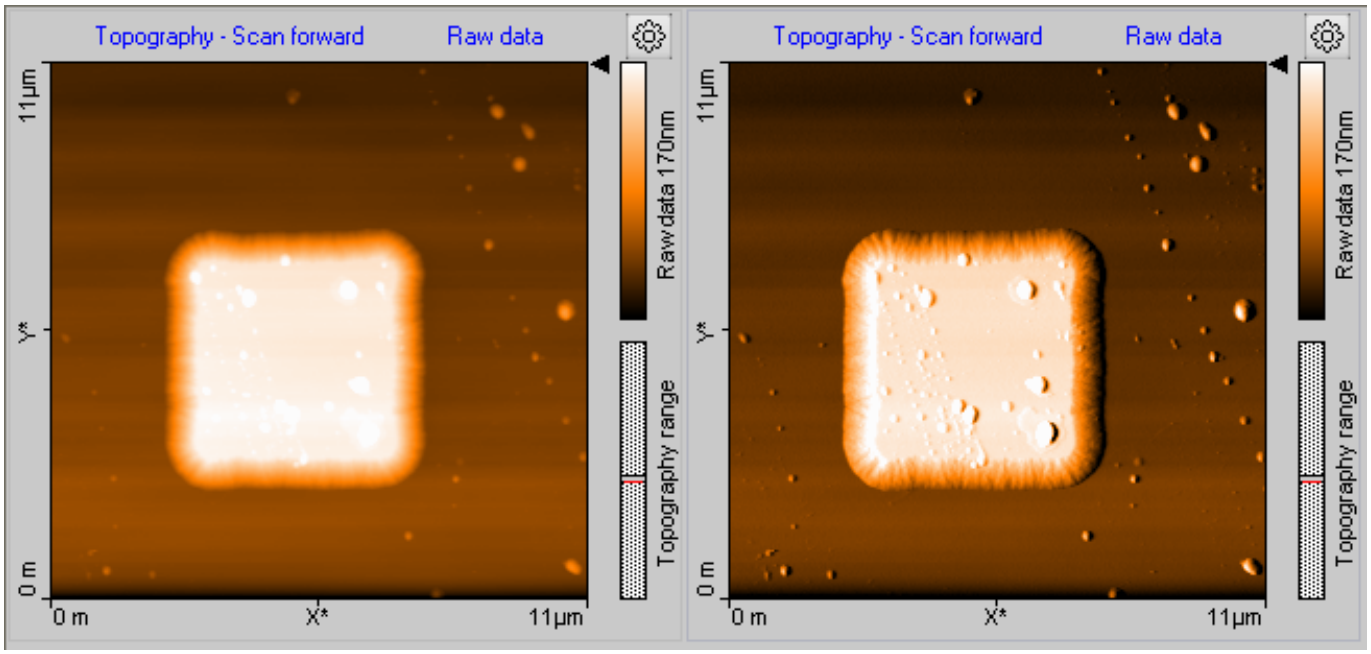
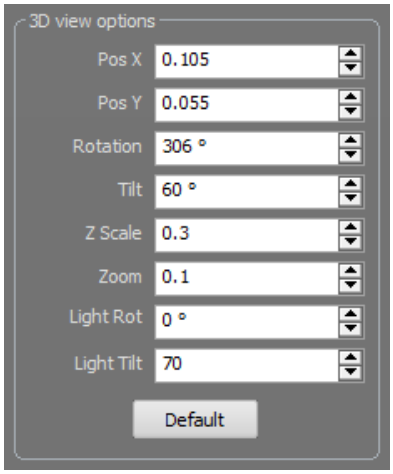


Figure 4-9: Shading option. (Left) Shading disabled. (Right) Five-pixel shading enabled.

Smooth pixel

When checked, the screen edge rendering of individual data pixels is smoothed with their neighboring pixels. Alternative data pixels are drawn as individual squares. This smoothing shows the most effect when the display size is larger than the number of measured of data points (e.g. a 256×256 measurement displayed at 512×512 pixels).

3D View options



Pos X, Pos Y

Defines the center position of the 3D plot inside the chart area.

Rotation

Defines the z-axis rotation of the 3D plot relative to the view point.

Tilt

Defines the off-plane angle of the 3D plot.

Z-Scale
Defines a Z-axis ‘stretch’ factor. Use this e.g. to enlarge surface details.

Zoom
Defines the magnification of the 3D plot.

Light Rot
Defines the rotation angle of the light source relative to the Z-axis (360°)

Light Tilt
Defines the off-plane angle of the light source. The lowest value (0°) corresponds to "sunset" lighting, the highest value (90°) corresponds to mid-day lighting at the equator.

Default
The “Default” button resets all 3D parameters to their default values.

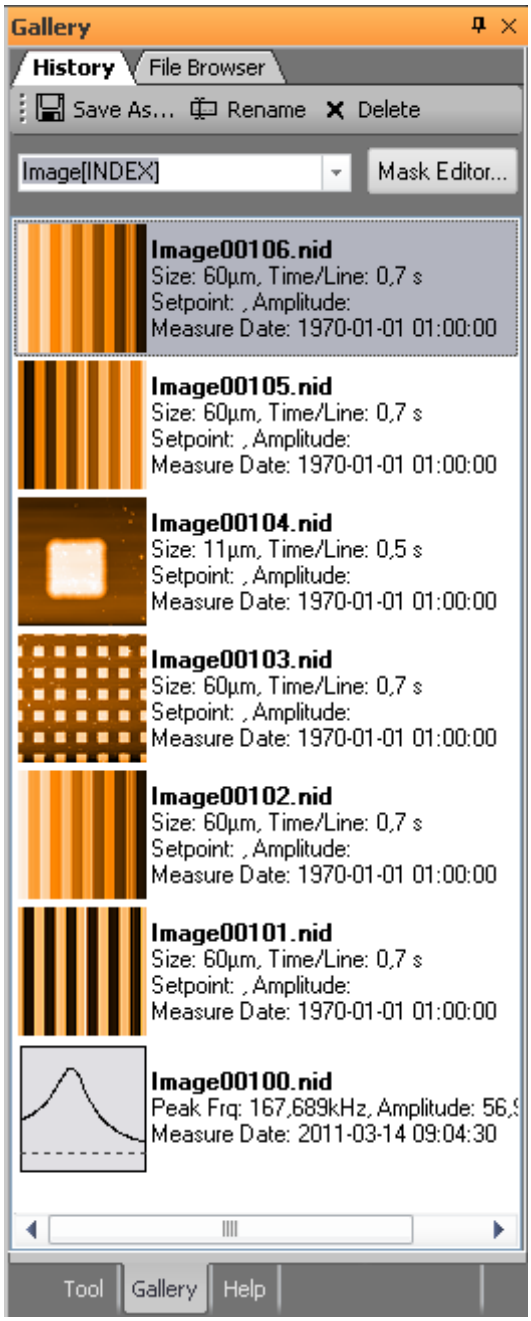
Keyboard and Mouse short cuts
Always click and hold the left mouse button on the 3D view chart while moving around the mouse to change the 3D view. The surface is reduced in feature complexity once the left mouse button is pressed to speed up redrawing on the screen. The surface will return to full detail once the mouse button is re eased.

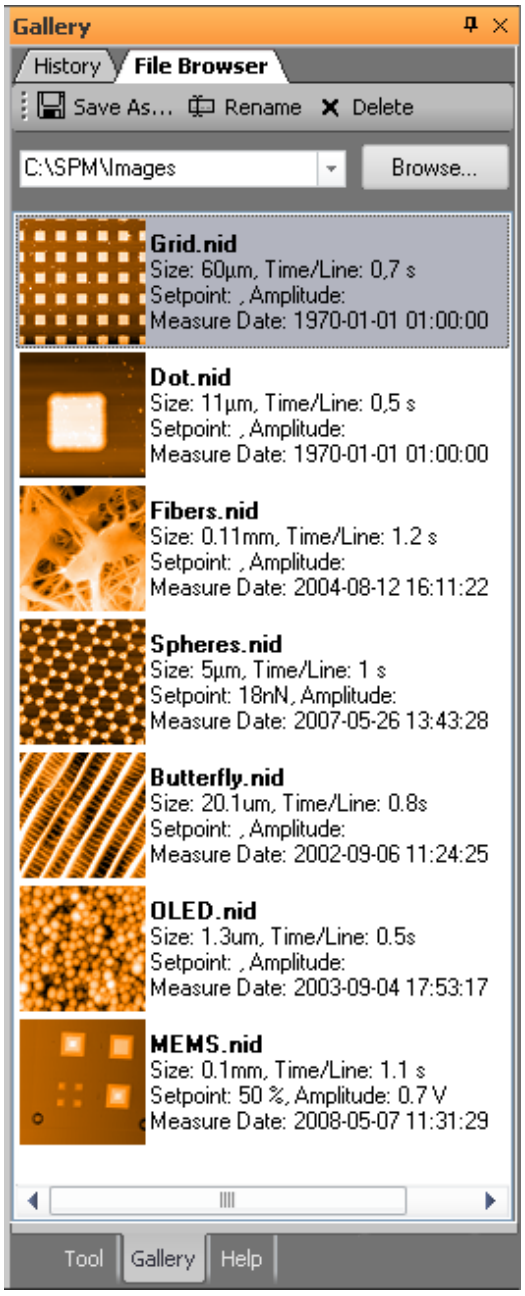
Press the following additional keys/buttons to determine which chart property is changed:

- **Surface rotation** Mouse left/right
- **Surface tilt** Mouse up/down.
- **Size displayed surface** "Ctrl"- key + mouse up/down
- **Surface position** "Shift"-key + mouse up/down/left/right
- **Z-scale magnification** Left mouse button + right mouse button + mouse up/down
- **Light source direction** "Shift"+"Ctrl"-key + mouse left/right
- **Light source height** "Shift"+"Ctrl"-key + mouse up/down

4.3.3 Gallery panel

The Gallery Panel displays lists of thumbnails representing previous measurements for quick access to those documents. It contains two pages: the "History" page, with the temporarily (automatically) stored measurements (for details on how to change the default History folder and the maximum number of files to be stored see *Gallery Settings* (page 91)) and the "File Browser" page with measurements from a user-selected directory (e.g. containing older measurements that were previously moved there). The various elements of the Gallery panel are described in the next sections.





History File mask

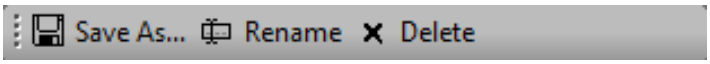
The temporary (automatic) storage of new measurements uses a File mask to create new file names each time a new measurement has to be saved. This mask can contain normal text, but also special variables like index number or date and time stamps. You may enter a new mask directly into the edit field in the History page, select an old mask from the drop-down menu, or define a new mask with the help of the Mask Editor dialog (see *Mask Editor dialog (page 38)*), which is opened by clicking the “Mask Editor” button next to the edit box.

Image list

In the Image list the stored measurement are shown. Each measurement is displayed as a thumbnail image of the measurement, together with some information about the measurement and measurement document.
The following mouse operations are possible inside the image list:

- **Double-click** Opens the respective measurement in the document space.
- **Single left mouse click** Selects the respective measurement and removes all other selections.
- **"Ctrl" key + left mouse click** Adds individual selections to the current selections.
- **"Shift" key + left mouse click** Selects all measurements from the last selection to the new selection.

Gallery toolbar



The Gallery toolbar is present in both the "History" and "File Browser" pages. It performs similar functions in both pages.

Save as
Selected measurements can be saved to a new location with this button.

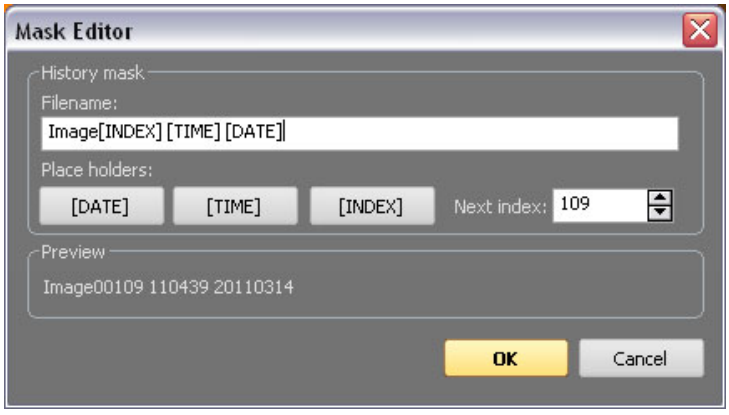
If only a single measurement is selected a standard Windows "Save" Dialog is shown. Here you select the new location and the new file name.

If multiple measurements are selected a "Folder" dialog is shown, which allows you to select (or create) the folder that all selected files are to be copied to. It is strongly recommended to do this for files in temporary storage of the History folder that you want to keep, because they may be overwritten as soon as the maximum number of files in the History folder is reached (see *Section Gallery Settings* (page 91)

Rename
Single or multiple measurements can be renamed by clicking the "Rename" button. It will open the File Rename dialog (see *File Rename dialog (page 40)*), which allows you to specify a mask for the new file names.

Delete
Single or multiple measurements can be deleted by clicking this button. The Mask Editor dialog assists you in the creation of file name masks.

Mask Editor dialog



The Mask Editor dialog assists you in the creation of file name masks.

History mask

Filename
A file name mask is the template that is used to generate the file names for documents that are temporarily stored (automatically) during measurement. A file mask consists of standard text entered by the user and of variables for software-generated text or numbers (either specified by the user or added automatically).

Mask variables
Mask variables can be entered as specific words surrounded by square brackets. The following variables are defined in the SPM Control Software:

- **[INDEX]** This variable represents a number that is automatically incremented each time a new filename is created. The index number is 5 digits in length and filled with zeros for missing digits. The next index number that will be used is shown in the “Next Index” field.
- **[TIME]** This variable represents the actual time of file name creation. It is formatted with two digit numbers for the hours, minutes and seconds (HHMMSS). This time format is used regardless of the Regional Settings of the Windows operating system.
- **[DATE]** This variable represents the date of the day the file was created (i.e., the day the measurement was performed). It contains four digit numbers for the year and two digit numbers for month and day (YYYYMMDD). This date format is used regardless of the Regional Settings of the Windows operating system.

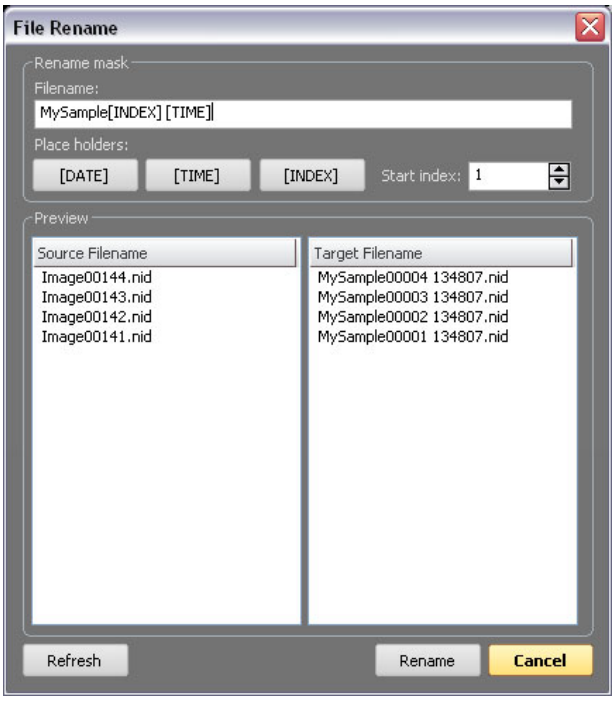
To quickly insert a Mask variable at the current cursor position in the mask edit box, click the corresponding button.

Next Index
This entry field defines the next index number to be used. By default this value is identical to the highest number present in the History files, increased by one.

If no Mask variable is used, an index is automatically added to the text string defined in the filename mask.

Preview
The filename that will be used for the next measurement document to be saved
(as specified by your entries) is shown here.

File Rename dialog



The File Rename dialog is used to rename (or move) multiple files using a Rename mask (see below). The dialog is opened by clicking the "Rename" button in the Gallery panel. To rename a file or multiple files, the Rename mask can be defined here following the same principles as for the History mask. A preview of the new filenames is shown in the preview section.

Rename mask
See *History mask* (page 39).

Preview

Source Filename
The left column of the Preview section shows the original filename(s).

Target Filename
The right column of the preview section shows what the filename(s) will be after pressing the “Rename” button.

Refresh
This button updates the preview list.

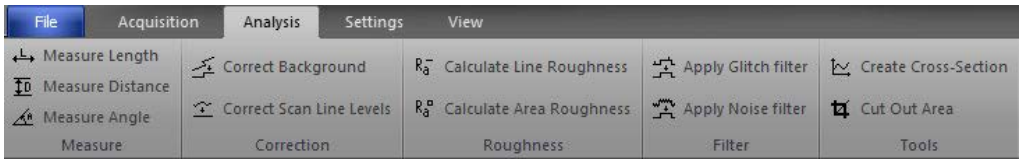
Rename
This button renames the selected files.

If no unique filename(s) would result from the specified Rename mask, an index is automatically added to the files. If this still does not result in unique filenames, the text "Copy of " is added to each filename as often as is required to make it unique.

Cancel
This button closes the dialog without renaming the files.

4.3.4 Analysis tab

Measurement data can of course not only be displayed in charts, it can be analyzed as well. The control software has several tools that allow quick numerical evaluation and modification of chart data in Operating or document windows. These tools are accessible through the various groups of the Analysis tab.



All of these tools can also be used while a measurement is still being acquired.

To use a quick evaluation tool:

1. Click on the chart that you want to evaluate to activate it.
2. Select the desired tool using one of the following approaches
   - Click on one of the tool buttons in the Analysis tab.
   - Select the tool from the Chart's context menu (right-click on the chart).
3. Define the evaluation. The procedure to define the evaluation is different for each tool. Details can be found in the tool-specific instructions below.

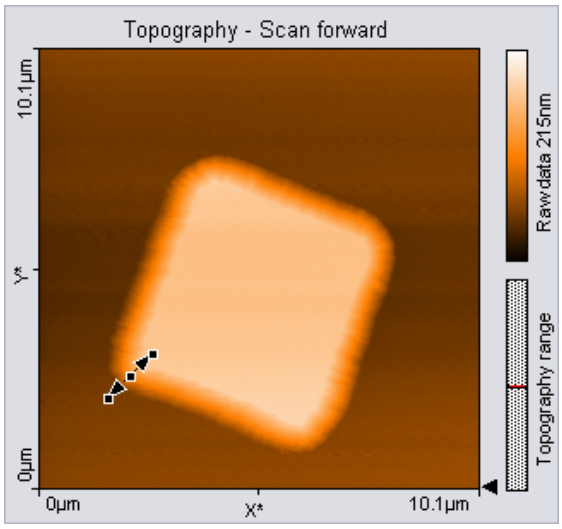
When a tool has been selected, the Tool panel (see *Section 4.3.5: Tool panel*) moves to the top of the panel stack of the Info pane.

Depending on the selected chart type, some tools may be unavailable.

To stop using a tool select another tool, select the same tool a second time, or select "Abort" in the Chart's context menu

4.3.4.1 Measure group

Measure length

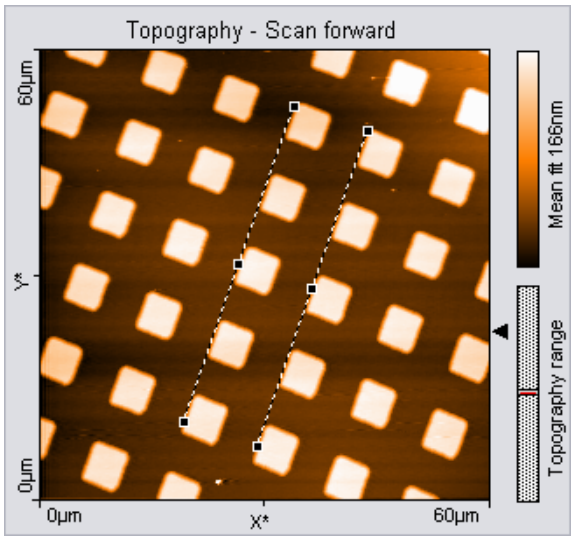


Calculates the distance and signal difference between two points. Graphically, a line with arrowheads on each end represents the selection marker. The line is defined by drawing a line on the measurement chart. The first point is positioned by moving the mouse cursor to the desired location and clicking and holding the left mouse button. The second point is positioned when the mouse button is released. When the mouse is not moved between clicking and releasing, a line parallel to the X*-axis is drawn.

The direction and length of the selection marker can be adjusted by dragging the end markers. The line can be moved as a whole by dragging the center marker.

The Tool status section of the Tool panel displays the calculated “Length”, “Delta-Z”, “Width” and “Height”. This data will also be stored in the “Tool” data category of the Data Info panel (see *Section 4.3.1: Data Info panel*) for the respective measurement document as long as the tool is active when the document is stored. For more information on the data in the Tool status section (see *Tool status section* (page 50)).

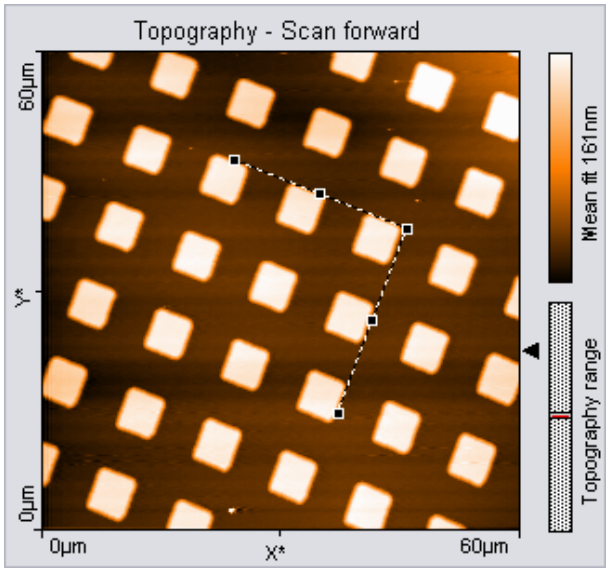
Measure Distance



Calculates the distance between two parallel lines. The parallel lines are defined by drawing them in the chart. The first point of the first line is defined by the mouse cursor position where the left mouse button is clicked, the second point by the position where the button is released. When the mouse is not moved between clicking and releasing, a line parallel to the X*-axis is drawn. After releasing the mouse button, a second parallel line sticks to the mouse cursor, that is released by clicking its desired position. The direction of the parallel lines can be adjusted by dragging their end markers; they can be moved by dragging the center marker.

The Tool status section of the Tool panel displays the calculated distance. The distance value only depends on the cursor positions, it does not on depend the displayed data values. The distance data will also be stored in the “Tool” data category of the Data Info panel (see *Section 4.3.1: Data Info panel*) for the respective measurement document as long as the tool is active when the document is stored. For more information on the data in the Tool status section (see *Tool status section* (page 50)).

Measure Angle

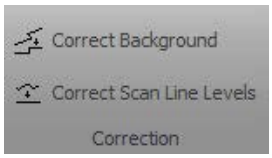


Calculates the angle between two lines. In Line graph-type displays, this tool can only be used when the chart displays data that has the unit "meters".

The two lines are defined by drawing them in the chart. The first point of the first line is defined by the mouse cursor position where the left mouse button is clicked, the second point by the position where the button is released. When the mouse is not moved between clicking and releasing, a line parallel to the X*-axis is drawn. After releasing the mouse button, the end of the second line sticks to the mouse pointer. The end is released by clicking its desired position. The angle can be changed by dragging the line end point markers or the corner mark; it can be moved by dragging the line center markers.

The Tool status section of the Tool panel displays the calculated angle. This data will also be stored in the “Tool” data category of the Data Info panel (see *Section 4.3.1: Data Info panel*) for the respective measurement document as long as the tool is active when the document is stored. For more information on the data in the Tool status section (see *Tool status section* (page 50)).

4.3.4.2 Correction group



In contrast to the evaluation tools of the Measure and Roughness groups, the tools of the Correction group (and also those of the Filter group (see *Filter group* (page 46)) actually change measurement data. This is done in a copy of the original measurement document, though, so you won't lose any data and will always be able to access the original measurement data in addition to the corrected or filtered data.

Correct Background
Removes the effect of an ill-aligned scan plane when the line filter options (see *Filter* (page 32)) do not give satisfactory results. This may be the case when the scan lines in different parts of the measurement have a different average height. An example of such a measurement is shown in *Figure 4-10: Correct Background*.

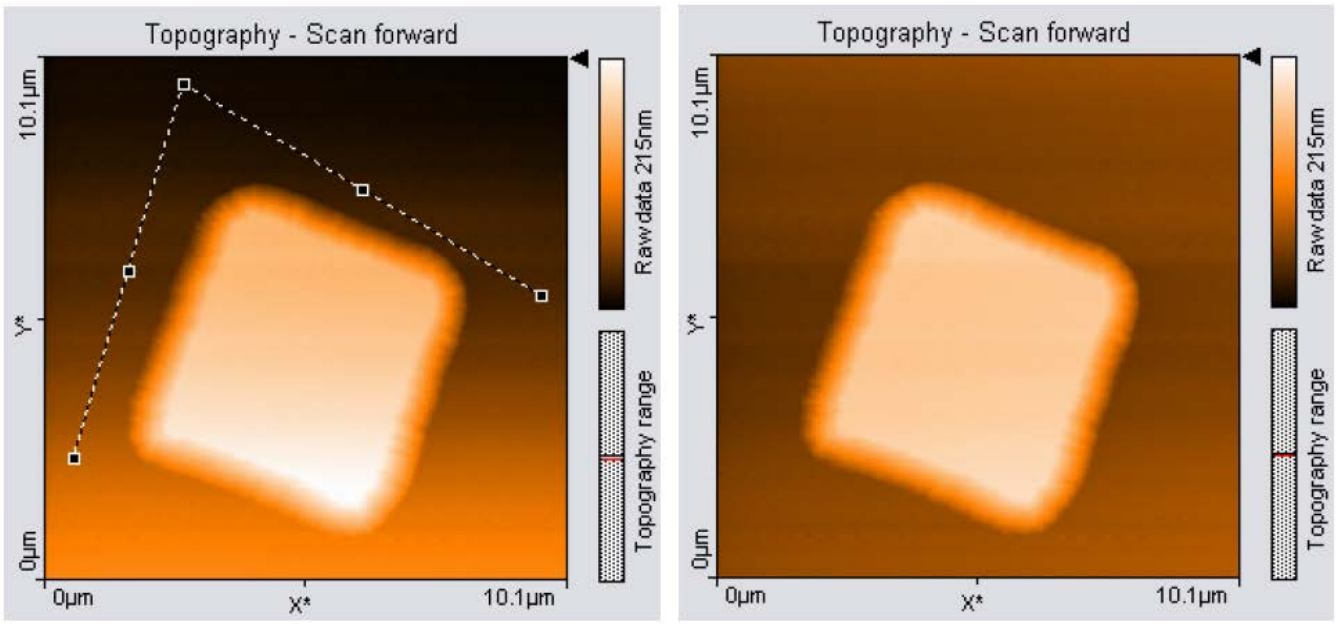


Figure 4-10: Correct Background (Left) Uncorrected image; the end points of the selection marker have been moved to points that should have the same height. (Right) Corrected image.

To use the tool, select three points that should be on the same height. This is done in the same way as with the angle tool (see *Measure Angle* (page 43)). The selected points become the end points of the selection marker.

After clicking the "Execute" button in the Tool status section of the Tool panel, a copy of the original measurement document is made and the plane that is defined by the selection maker is subtracted from the measurement data in the newly created document. To get useful results, the Data filter option for the corrected image in the new document will be automatically set to "Raw data".

Correct scan line levels
Removes the effect of drift when the line filter options (see *Filter* (page 32)) do not give satisfactory results. This may occur when the scan lines in different parts of the measurement have a different average height. An example of such a measurement is shown in *Figure 4-11: Correct scan line levels*.

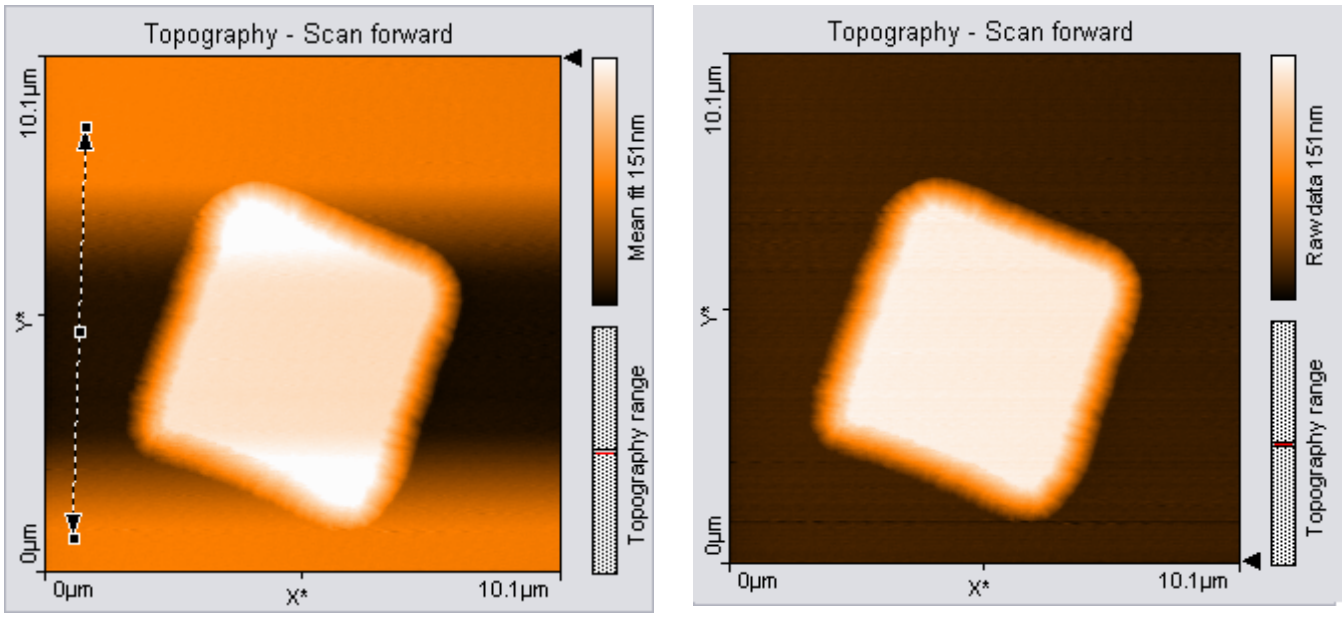
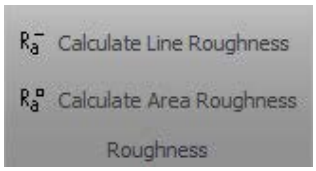


Figure 4-11 Correct scan line levels. (Left) Uncorrected image with a selection marker through points that should be at the same height. (Right) Corrected image

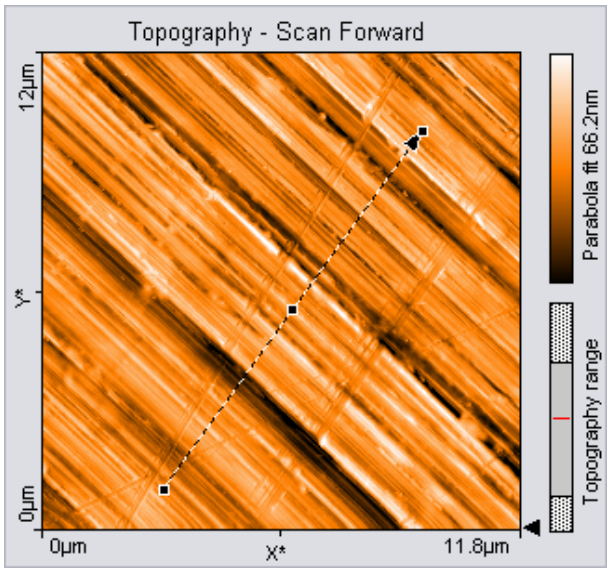
To use the tool, draw a line through points that should have the same height in the same way as with the Measure Length tool.

After clicking the “Execute” button in the Tool status section of the Tool panel, a copy of the original measurement document is made and the average level of each scan line in the newly created document is adjusted so that all points along the drawn line have the same height. To get useful results, the Data filter option for the corrected image in the new document will be automatically set to “Raw data”.

4.3.4.3 Roughness group



Calculate Line Roughness
Calculates several roughness parameters from the data at points along a selected line. The line is selected in the same way as with the Measure length tool (see *Measure Length* (page 42).



The Tool status section of the Tool panel displays the calculated “Length” and “Delta-Z” of the selected area.

The Tool result section displays the roughness values that are calculated from the data according to the following formulas:

The Roughness Average, S_a

$$S_a = \frac{1}{N}\sum_{l=0}^{N-1} | z(x_l) |$$

The Mean Value, S_m

$$S_m = \frac{1}{N}\sum_{l=0}^{N-1} z(x_l)$$

The Root Mean Square, S_q

$$S_q = \sqrt{\frac{1}{N}\sum_{l=0}^{N-1} (z(x_l))^2}$$

The Valley depth, S_v

S_v = *lowest value*

The Peak Height, S_p

S_p = *highest value*

The Peak-Valley Height, S_y

$S_y = S_p - S_v$

The roughness values depend on the line filter option (see *Filter* (page 32)) that is applied to the chart, because they are calculated from the filtered data.

Clicking the "Store" button in the Tool result section stores the roughness values in the “Roughness” data category of the Data Info panel for the active measurement document.

Calculate Area Roughness
Calculates several roughness parameters from the data points in a selected area.

The area is selected in the same way as with the Cut out Area tool.

The Tool status section of the Tool Results panel displays the calculated Size or Width and Height of the selected area. For more information on the Tool status section, see the *Tool status section* (page 50).

The Tool result section displays the roughness values that are calculated from the data according to the following formulas:

The roughness values depend on the Data filter that is applied to the chart, because they values are calculated from the filtered data. More information on data filters is provided in *Section 4.3.4.4 Filter group*.

Clicking the "Store" button in the Tool result section stores the roughness values in the “Roughness” data category of the Data Info panel for the active measurement document.

The Area Roughness tool can be used to determine the mean height difference between two plateaus with more accuracy than with the "Measure Distance" tool. To determine the mean height difference, select an area on each plateau, and calculate the difference between their S_m-values.

4.3.4.4 Filter group

Glitch Filter
The Glitch Filter removes the effect of small defects in the image such as single short glitches in the scan. Compared to the Noise Filter (see below), it has the advantage of not reducing resolution on step edges. The glitch filter is implemented as a Median filter on a 3×3 pixel matrix.

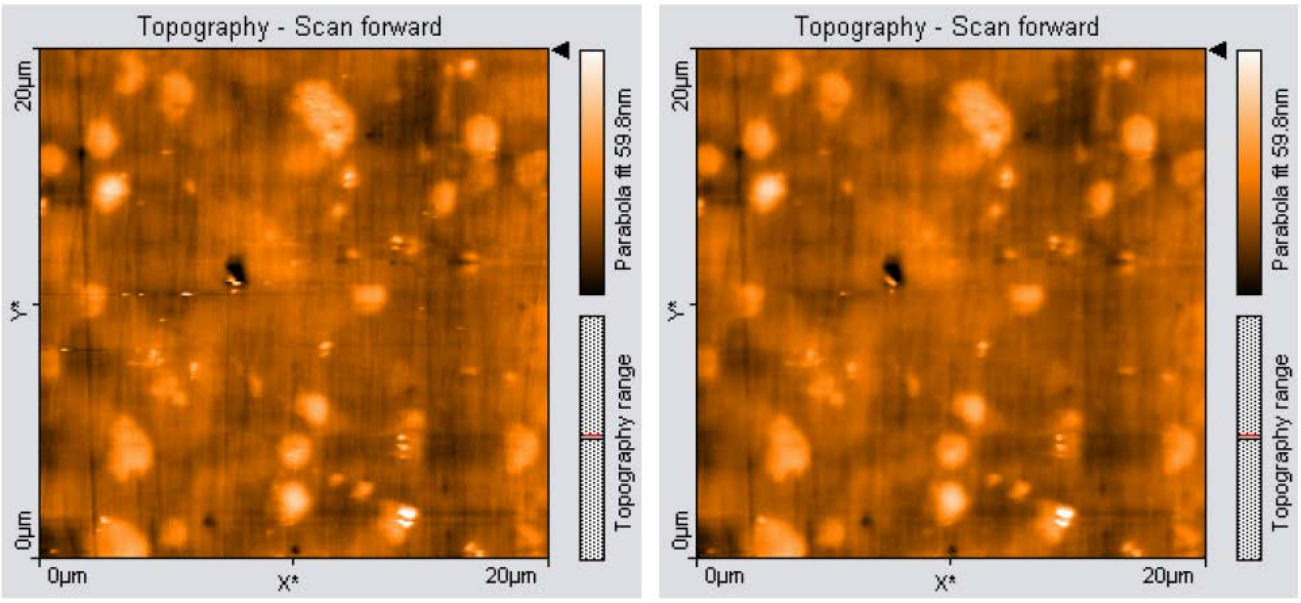


Figure 4-12 Glitch Filter (Left) Unfiltered image with some glitches where the tip lost contact with the sample. (Right) Corrected image.

To apply the filter, activate the color map chart that is to be filtered, then click the "Glitch Filter" button. A new Measurement document with the filtered data is created.

Noise Filter
The Noise filter removes high frequency noise from the image, but applying the filter will also decrease the resolution of the image. The Noise Filter is implemented as a convolution with a 3×3 pixel Gaussian kernel function.

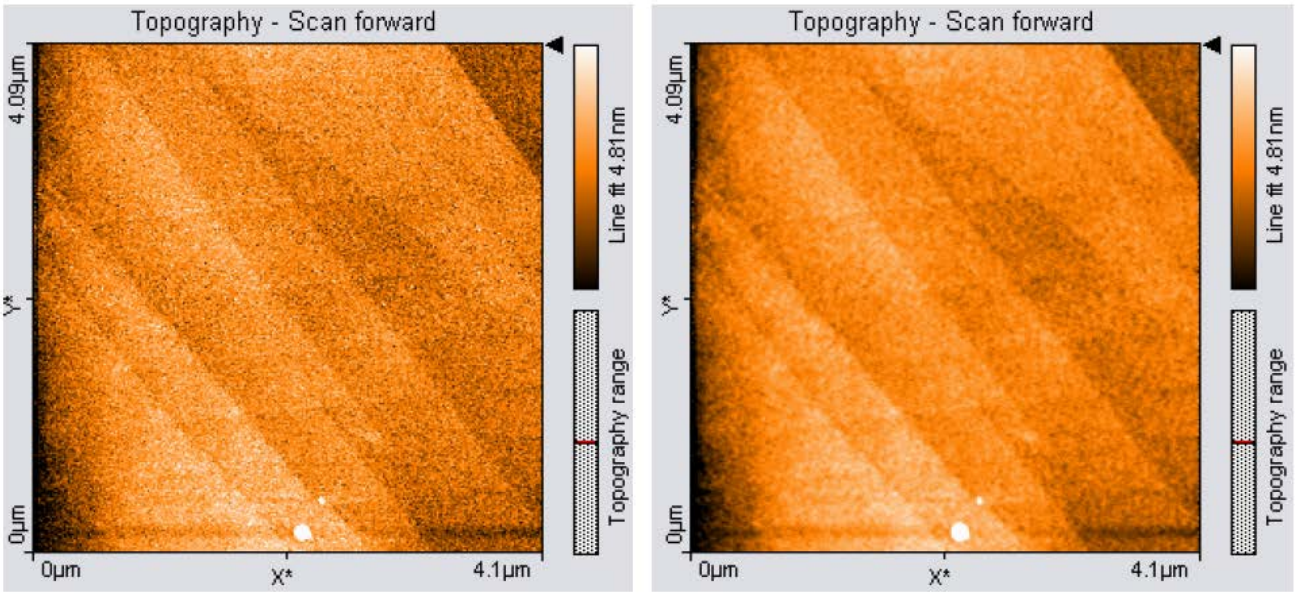
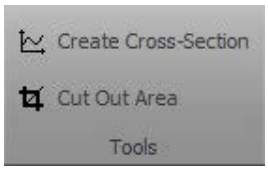


Figure 4-13 Noise Filter (Left) Noisy (unfiltered) image of an AFM measurement on HOPG. (Right) Filtered image

To apply the filter, activate the color map chart that is to be filtered, then click the "Noise Filter" button in the Tools bar. A new measurement document with the filtered data is created.

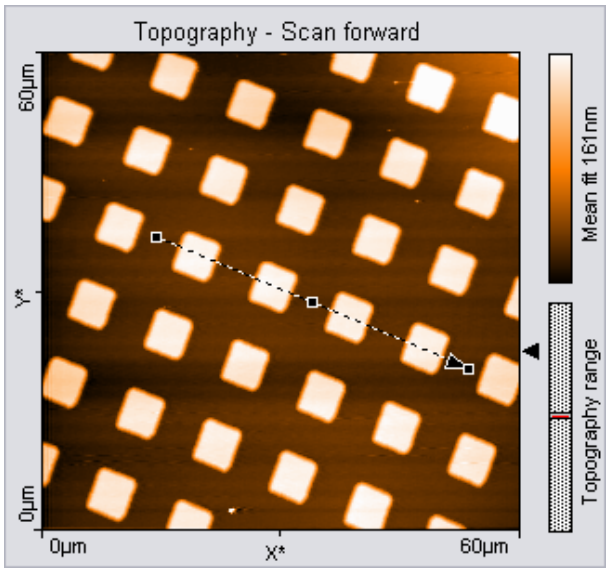
- *Filters are especially useful for improving the appearance of 3D views.*
- *Applying filters may changes the result of the other tools. This may result in incorrect results, e.g. when evaluating sample roughness.*

4.3.4.5 Tools group



Create Cross-Section
Creates a new measurement document containing a line cross-section of a Color map or Line View display.

The line is defined by drawing a selection arrow. The arrow points toward the forward direction of the line. The start of the arrow is defined by the mouse cursor position where the left mouse button is clicked, the end of the arrow by the position where the button is released. When the mouse is not moved between clicking and releasing, an arrow ending in the center of the measurement is drawn. The direction of the arrow can be adjusted by dragging its end markers; it can be moved by dragging the center marker.



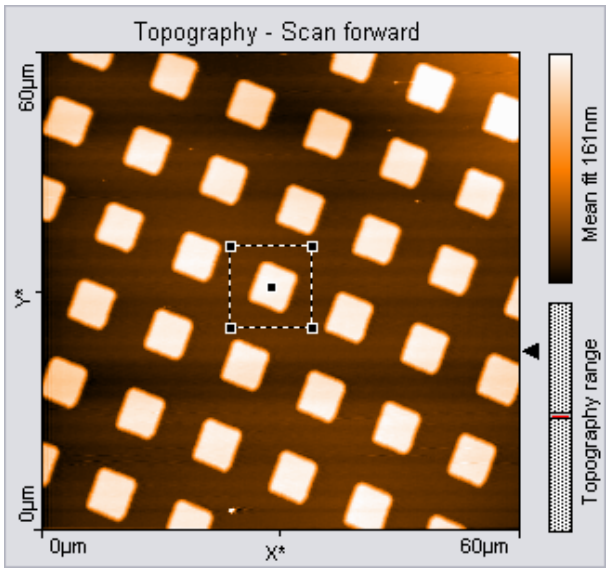
Double-clicking the graph, or clicking the "Cut out line"-button in the Tool status section of the Tool panel creates a new document that contains the line section.

The Tool chart section of the Tool Results panel displays a preview chart of the selected line.

The Tool status section of the Tool Results panel displays the calculated “Length” and “Delta-Z” of the selected line. For more information on the data in the Tool status section (see *Tool status section* (page 50)).

Cut Out Area
Creates a new measurement document containing a subsection of an existing measurement.

One corner of the area is defined by the mouse cursor position where the left mouse button is clicked, the opposite corner by the position where the button is released. When the mouse is not moved between clicking and releasing, an area is defined that has a size of 33% of the current measurement, and is centered on the clicked location.



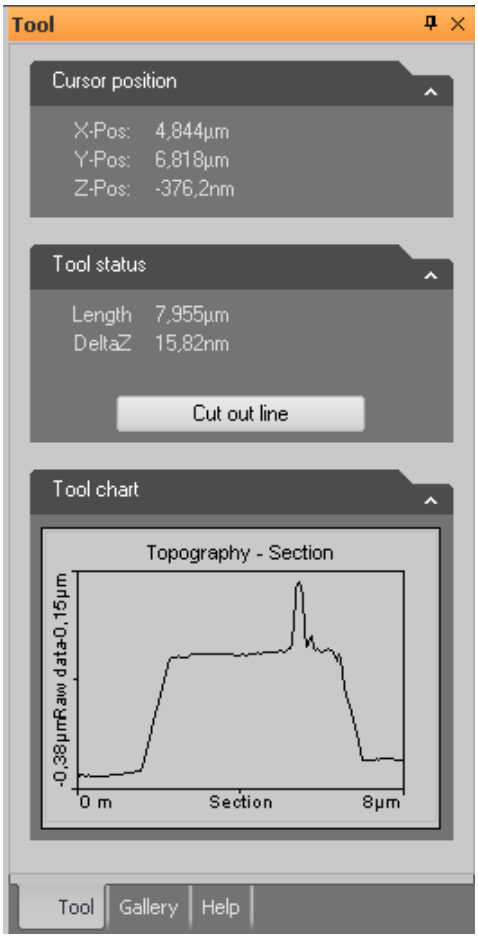
Once an area is defined, it can be resized by dragging one of its corners, and moved as a whole by dragging its center point.

Pressing the "Shift" key while dragging a corner defines a non-square (i.e. rectangular) area.

Double-clicking the graph or clicking the "Cut out area" button in the Tool Results panel creates a new measurement document that contains the selected area.

The Tool status section of the Tool Results panel displays the calculated “Size” or “Width” and “Height” of the selected area. For more information on the data in the Tool status section (see *Tool status section* (page 50)).

4.3.5 Tool panel



The Tool panel of the Info pane displays varying information, which depends on the tool currently selected in the Analysis tab.

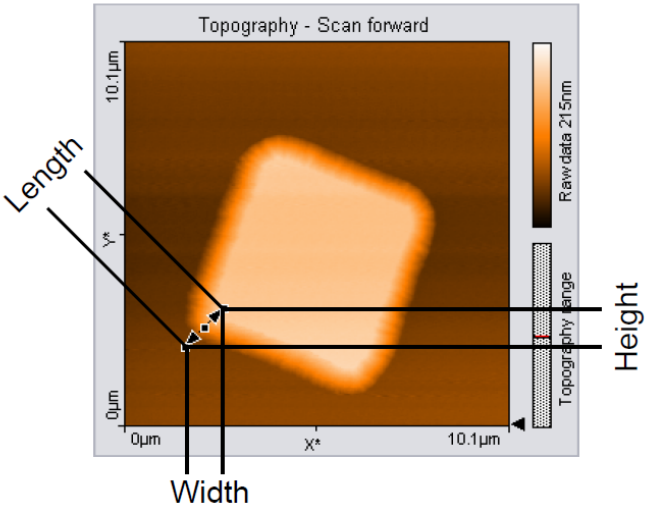
Cursor Position section
This section is always visible. It displays the mouse cursor position in the physical units of the selected chart.

Tool status section
This section appears when a tool is being used. It displays the evaluation result of the currently active tool.

The tools that require drawing a selection marker to define the evaluation have some common parameters that are described here. The other parameters are described in the sections that describe the respective tool (see above).

Length
The length of the selection marker in the plane of the chart. “Length” is related to the evaluation results “Width” and “Height” (see below) according to the formula:

$$Length = \sqrt{Width^2 + Height^2}$$



In a Color map chart, length is calculated in the XY-Plane. In a Line graph chart, length is calculated in the XZ-Plane.

"Length" is not displayed when "Width" and "Height" are of different physical units (e.g. in Amplitude Spectroscopy, where the X-Axis is given in [m] and the Z-Axis in [V]).

Width, Height
The “Width” and “Height” of the measurement tool in the chart, calculated in the chart plane.

Delta-Z
The difference between the “Z-Pos” values at both ends of the selection marker. In a Color map chart, “Delta-Z” is the difference in the (filtered) sample height between the start and the end point.

The calculated values of "Length", "Width" and "Height" only depend on the cursor positions; they do not depend on the displayed data values.

CHAPTER 5 :

Preparing the first measurement

The following precautions must be taken to keep the equipment free of dust and grease to achieve atomic resolution on your sample.

- Never let your fingers touch the wire for tips, the sample, or the parts of the STM scan head (you could use a pair of latex gloves - not included in the set).
- Only touch the Sample Holder at the black plastic end.

Once the system has been set up on a very steady table you have to prepare the system for your measurements. The preparation consists of three steps: Preparing the STM tip, mounting it on the tip holder and installing the sample with its Sample Holder in the right position. See below.

5.1 Preparing and Installing the Tip

The STM tip can be prepared and installed by yourself. This is a difficult part of the preparation which has to be carried out very thoroughly. It usually needs patience and practice to get the first good tip. Only an accurately cut tip enables optimal measurements. Cutting and installing should be carried out with great care. On delivery the tip which was used to calibrate your STM is mounted. This tip should give atomic resolution, so you may wish to try using this tip before preparing your own.

5.1.1 Before cutting the Tip

1. Clean the cutting part of the wire cutters, the flat nose pliers and the pointed tweezers with ethanol. Only touch the Pt/Ir wire with these tools.
2. Remove the used tip from the instrument using the pointed tweezers.

5.1.2 Preparing the Tip

1. Hold the end of the wire tightly with the pliers.
2. Holding the wire with the pliers, move the cutters at a length of approximately 4 mm, as obliquely as possible (in a very sharp angle).
3. Close the cutters until you can feel the wire, but do not cut the wire.
4. In order to obtain the required sharpness, the tip needs to be torn off by pulling the wire cutter quickly away from you, rather than cutting cleanly through the wire.
5. Use the pointed tweezers to hold the tip wire right behind the tip.
6. Release the flat pliers.

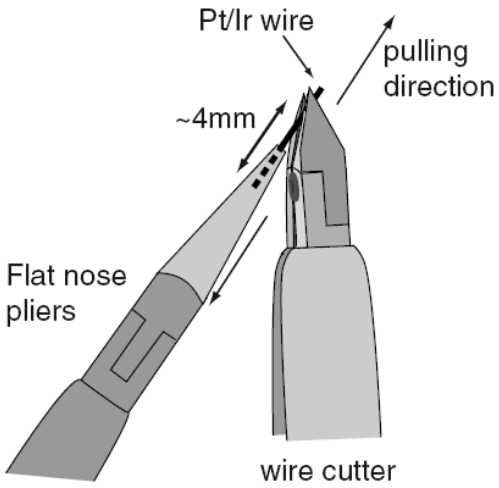


Fig. 4-1 Sketch of the tip preparation.

Now that you have prepared a fresh tip it is necessary to handle it with care. Use the pointed tweezers to bring the tip to the tip holder. Grab your wrist tight with your remaining hand to avoid shaking. Look at the pictures below carefully and try to imagine how to bring the wire under the tip clamp of your microscope. When you release the wire too early without being perfectly in the groove, it is likely to snap away and your freshly prepared tip is wasted.

Proceed with mounting the STM tip. See below.

IMPORTANT

- Never touch the end of the tip with anything.
- Ensure that the tip wire is straight.
- Do not twist the tip clamp in any way, nor lift it too high.

5.1.3 Mounting the STM tip

This picture shows the tip holder with its groove and the clamp which fixes the tip wire.

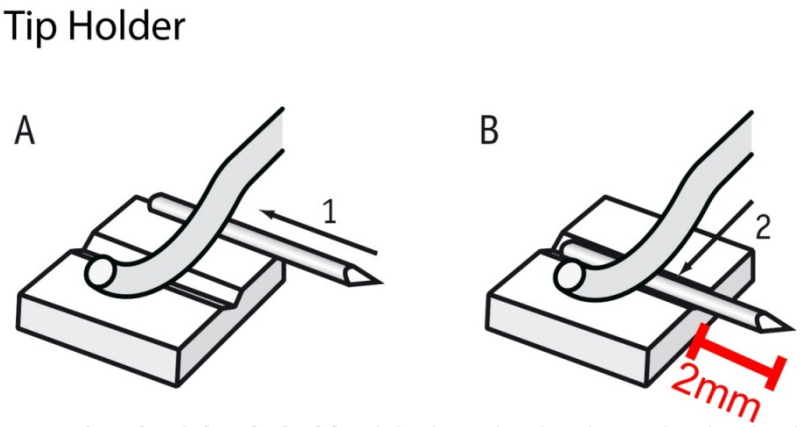


Fig. 5-2 Sketch of the tip holder (A) Place the tip wire under the tip clamp. (B) Move the tip sideways into the groove. Let the tip stick out to a maximum of 2 mm.

1. Put the tip wire underneath the clamp on the tip holder (A), parallel to the groove and push it all the way to the end.
2. Move the tip wire sideways until it is in the groove and held securely under the clamp (B). It should stick out about 1–2 mm beyond the tip holder.

The tip is now installed.

If you want to further improve your success in preparing good tips, you can also use a screw clamp (not included in the set). You have to fix the clamp on a table surface and fasten a piece of the Pt/Ir-wire of about 4cm. Now you can easily stretch the wire with the flat nose pliers and cut it with the wire cutter by pulling. After preparing some tips you should be able to determine good tips by their cutting sound. Good tips will give a short high pitch sound against relative bad tips which give a longer metallic sound (don't throw them away until you tested their quality with a quick measurement).

5.2 Preparing the Sample

The STM can be used to examine electrically conductive materials. In practice, however, the choice of material is more limited, because to obtain useful results, the surface of the sample has to be totally clean and mirror-like, and additionally be in a non-oxidized state to be conductive.

5.2.1 Graphite (HOPG) on Sample Support

You will find a Graphite sample in your tool box, which will be the sample of choice to have a close look on the sample surface and achieve atomic resolution.

Sample specifications:

Size: 5mm × 5mm
Material: Highly Oriented Pyrolytic Graphite (HOPG)
Sample support: Magnetic Steel disc, galvanized with Nickel.

IMPORTANT

Never touch the sample surface once it is prepared.

In most cases it is enough to cleave the Graphite sample once in a while. If you have problems to find a clean area or you don't get good images with several fresh prepared tips, clean the sample surface as described below.

Due to the layered structure of graphite, it can easily be cleaved using a piece of adhesive tape:

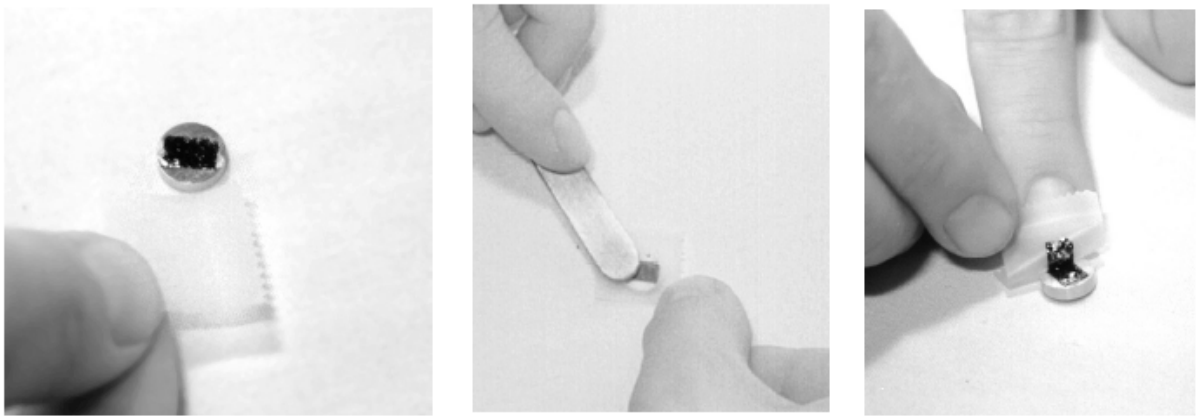


Fig. 5-3 Sample preparation Put adhesive tape on the sample and remove the upper layer.

Put the sample on the table using a pair of tweezers.

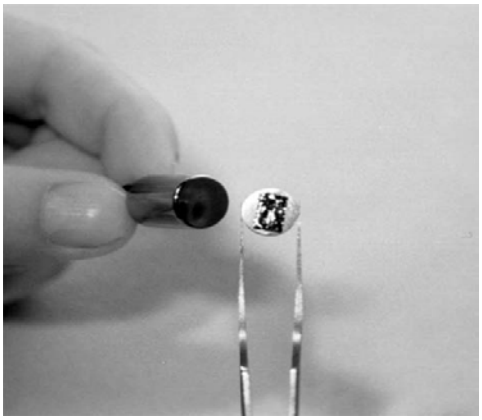
1. Stick a piece of adhesive tape to the graphite surface and apply very little pressure with your thumb or the end of the tweezers.
2. Use the tweezers to go under the adhesive tape and press the sample down to the table. Pull off the adhesive tape gently. The topmost layer of the sample should stick to the tape. If you are not satisfied with the cleaving (e.g. the surface looks uneven or there are too many flakes remaining), start from the beginning.
3. The middle of the sample surface should be very flat and mirror-like. Any loose flakes in the outer regions of the sample can be removed with the tweezers.

The graphite sample is now ready for use and should not be touched anymore.

Now that you have prepared the sample you need to mount it onto the Sample Holder.

5.2.2 Mounting the Sample onto the Sample Holder

1. Unpack the Sample Holder touching only its black plastic handle.
2. Use the tweezers to push the sample to the edge of the supporting magnet in the sample package.
3. Grab the sample with the tweezers (as shown in the picture) and place it on the magnet of the Sample Holder.



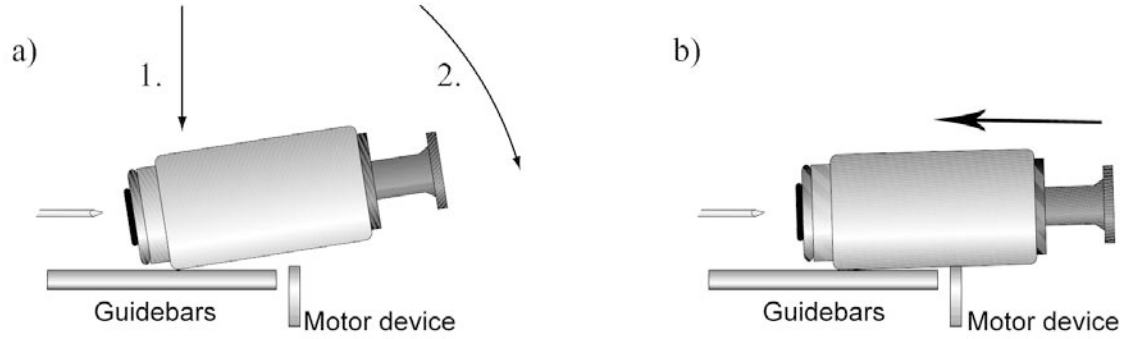
IMPORTANT

Always store the Sample Holder in its package, in order to prevent corrosion.

5.2.3 Mounting the Sample Holder on the Microscope

1. Put the Sample Holder down on to the Sample Holder Guide Bars first (a) and release it gently on to the approach motor's support (Be careful: the magnet that holds the Sample Holder in its place can drag the sample off the Sample Holder, make sure you bring the sample behind it).

2. Push the Sample Holder carefully in the direction of the tip (b), but don't let it touch the tip (1cm distance).



5.3 Approaching the Sample to the Tip

To start measuring, the sample must be very close to the tip, to enable a tunneling current to flow. Approaching the sample without touching the tip is a delicate operation carried out in three steps.

5.3.1 Manual coarse approach

In this step, the sample surface is brought close enough to the tip by hand, to facilitate further motorized approach afterwards.
To perform a manual coarse approach:

1. Push the Sample Holder carefully and very slowly to within 5 mm distance of the tip (or even closer). Make sure the surface doesn't touch the tip at any time (Fig. 5-4, a).
2. If the tip is pointing towards a rough area of the sample, try turning the Sample Holder around its axis so that the tip points towards a flat, mirror-like area of the sample (pull the Sample Holder away from the tip before doing so).
3. Put the Magnifying cover glass over the scan head without touching the Sample Holder (Fig. 5-4, b). The cover reduces air flow around the scan head and also thermal drift in measurements at atomic scale.
4. Place the magnifier in a way that you can see the tip of the wire and the surface of your sample (if you are already close enough you should see the mirror image of the tip in the sample surface, see Fig. 5-5).

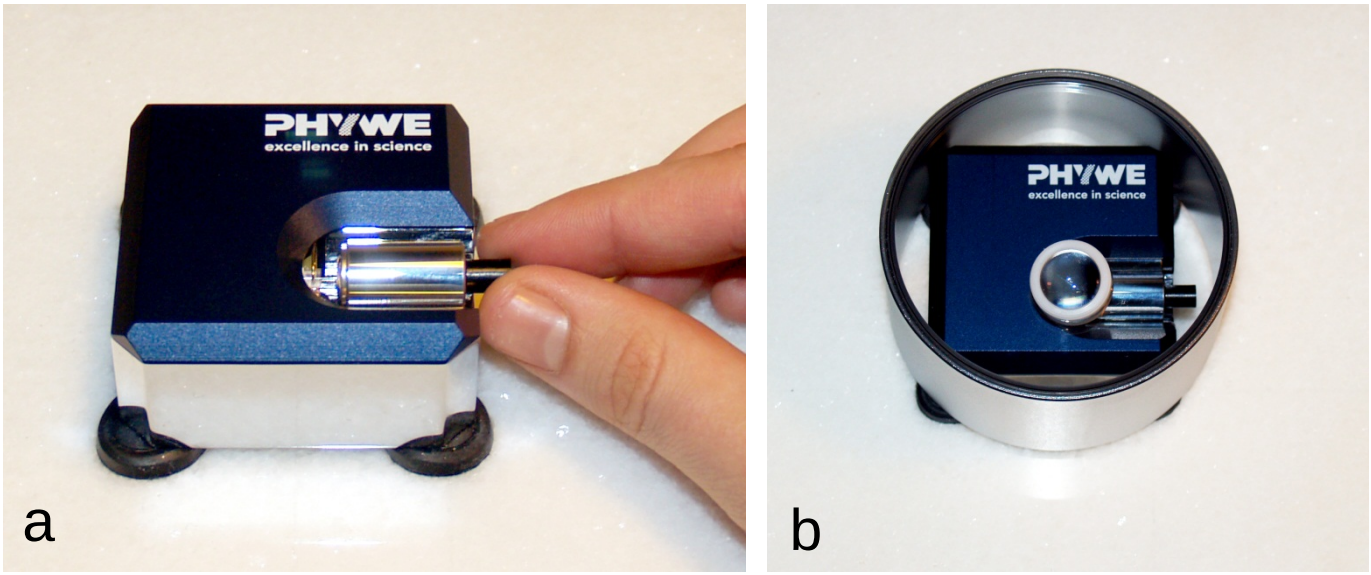
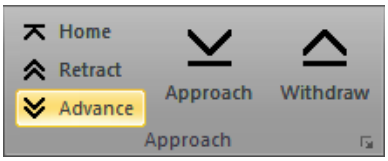


Fig. 5-4 Coarse Approach. Push the Sample Holder carefully in the direction of the tip (a) and put the cover glass on your microscope after the manual coarse approach (b).

5.3.2 Manual approach using the approach motor

Use the Approach Panel in the measurement software to bring the sample in a close distance to the tip.



1. Press the “Advance” button to move the sample towards the tip.
2. Watch carefully the mirror image of the tip. The tip of the wire and its mirror image will approach each other.
3. Always check for the “Probe Status” in the bottom area of the software interface. While advancing the status light should be orange. If it switches to red, you advanced too far and your tip is likely to be damaged.
4. When the tip and its mirror image are about to touch, stop further advancing. The distance should be only a fraction of a millimeter. This is difficult to determine, you will get used to a good distance by practicing.

Fig. 5-5 Approaching the sample to the tip. Tip and its mirror image are about to touch.

IMPORTANT

Never advance too close: Once the tip crashed into the sample surface, it is useless and you have to prepare a new one.

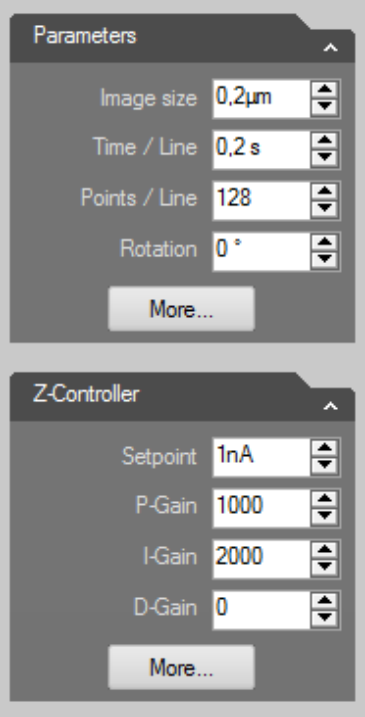
When it gets very hard to see if the tip and the surface are approaching correctly, press the “Retract” button to pull the sample away from the tip. You also can adjust the Magnifying glass for a better view. Then you can start over again.

5.3.3 Automatic final approach

In this last step, the sample automatically approaches the tip until a given set point is reached. At first, check that the set point and the feedback values (Gain) are set properly. To perform this:

1. In the Preparation group of the Acquisition tab, click the “Auto Set” button: A dialog will pop up, which will ask you some basic questions about your sample and your measurement needs.
2. Answer the questions of the wizard to the best of your knowledge.

If you don’t know that much about the used sample or are not satisfied with the auto set values you can follow these steps for a manual configuration:



1. Set “Image size” to 0.2 µm
2. Set “Set point” (tunneling current) to 1.00 nA.
3. Set “P-gain” (proportional speed of the feedback loop) to 1000.
4. Set “I-gain” (integral speed of the feedback loop) to 2000.
5. Set “D-gain” (differential speed of the feedback loop) to 0.
6. Set “Tip voltage” (tip–sample-voltage) to 50 mV.

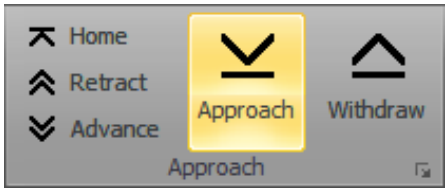
The displayed values are a good start for most samples.

Note

If measuring a very rough surface you should choose a lower initial Image size because the tip might otherwise crash into the sample.

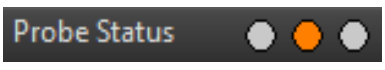
Now that the set-point and feedback settings are correct and the surface of the sample is about to touch the tip, the automatic final approach can be started.

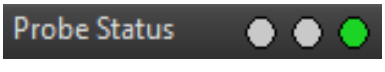
- Press the “Approach” button in the approach panel of the measurement software.



The motor unit will drive the sample towards the tip in very small steps, until a tunneling current can be established. You should hear a clicking sound from the approach motor. If the sample already was in a very close distance to the tip, approaching will only last a few seconds. When the gap between the sample and the tip was a little too big, the approach procedure can take up to several minutes. In this case, you can either abort the approach process by pressing the “Withdraw” button and trying to approach again with the “Advance” button, or you wait a little longer. After you have carried out some experiments, you should be able to bring the sample in a good distance where an automatic approach is possible, that only lasts about 10 to 30 seconds.

Always check for the “Probe Status” in the bottom area of the software interface. During the approach procedure, the status light should be orange. After a successful approach, the status light will switch to green. If you have set appropriate parameters for your measurement, the status light will remain green.

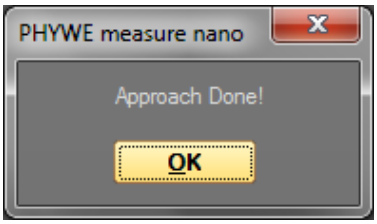




If the parameter for the set-point is set too high, it is likely that the status light will change to red. Then the tip is in direct contact to the sample and probably useless.

If the automatic final approach keeps not working, refer to “Problems and Solutions” (Chapter 9, page 76).

After a successful approach you get notified by a pop up window, press “OK”.



CHAPTER 6 :

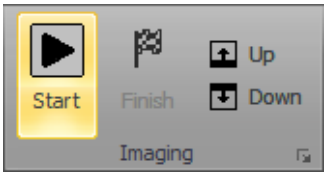
Measuring Graphite (HOPG)

Now you can investigate the HOPG sample in more detail

To start and view the measurement:

- Open the Imaging Window.

By default, the instrument is set to automatically start measuring after the automatic approach. If this is not the case start measurements manually by clicking the “Start” button in the Imaging group of the Acquisition tab:



If the preparation of the tip, the sample and the approach were successful (see *Chapter 5.2*), images of the measurement will show a more or less straight line in the Line Graph and a plane in the Color Map. Watch the displays for a while until the Color Map image has been drawn about three times.

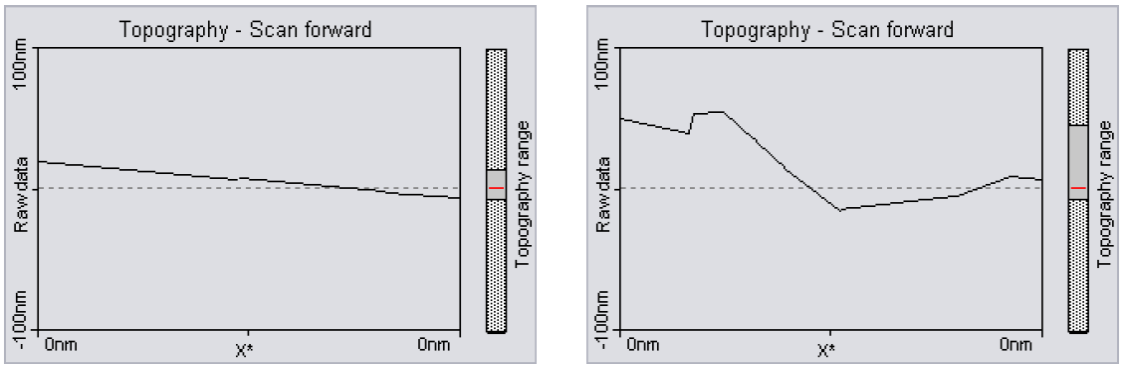


Fig. 6-1 Line graphs Straight Line Graph of a flat sample surface (left). Nervous Line Graph indicating a bad tunneling current (right).

If you have set the image size to rather large values (0.1 – 0.2µm), the Line Graph should picture an almost straight line referring to a very flat surface. When the Line Graph shows a rather ‘nervous’ line, this indicates a bad tunneling contact. Usually this is caused by the tip being too blunt, or your “Loop Gain” values don’t match the current measurement. You should try to adjust these. If the Line Graph won’t change to a stable straight line, you should stop measuring and prepare a new tip.
If the line in the Line Graph is calm and reproduces consistently, you can continue with the next section.

6.1 Achieving Atomic Resolution

Once the topography in the Line Graph is reproducing stably, the scan range has to be decreased in order to observe atomic structures.

IMPORTANT

Measurements on the micrometer/nanometer scale are very sensitive to environment influences. Direct light or fast movements (causing air flow and temperature variations near the Scan Head) can influence and disturb the measurement. It is best to let a promising measurement run for some time in order to let the sample stabilize thermally.

To decrease the Imaging area:

1. Click the Color Map chart to activate it. A blue square is now drawn around the Color Map chart.
2. Click “Zoom” in the upper tool bar, move the mouse cursor to a ‘flat’ region (similar color) in the Color Map and click on it. The software will now draw a square that indicates the new scan range. The size of the new scan range is displayed in the “Tool Status” Panel.
3. Change the size of the new scan range to about 30–50 nm by clicking and dragging a corner of the square with the mouse cursor.
4. Double-click the Color map when the new scan area is set as you want it (or press “Zoom” in the “Tools Status” Panel).

The imaging settings are now set in such a manner that the new measurement will correspond to the area that was indicated by the square you have set.

- Let the topography reproduce stably again.

To achieve atomic resolution, the image size should be decreased even further, considering that one nanometer is the diameter of between four and eight atoms. Atomic arrangements can normally be recognized at an image size of about 4 nm. Therefore:

- Set the image size in the Imaging panel to 4 nm or use the “Zoom” option on your last image.

Some parts of the scan head react to the slightest temperature changes. Since these thermal fluctuations influence the measurements on the nanometer scale, the sample has to be scanned as fast as possible:

- Set the “Time/Line” in the Imaging Panel to 0.03s (with 128 “Points/Line”) for atomic resolution.

With a good tip and properly set parameters, you should be able to observe atomic arrangements like shown in the picture.

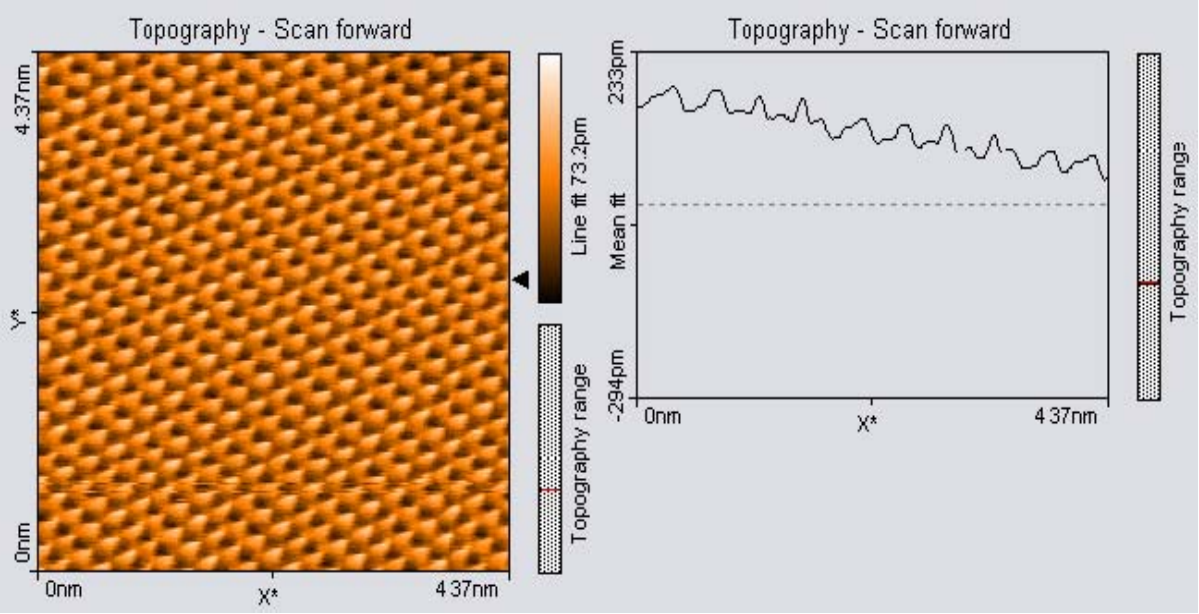
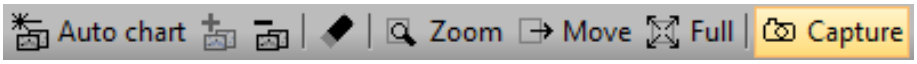


Fig. 6-2 Color map and Line Graph of a running measurement.

If you see extra spikes on the hills in the Line Graph, you may try to reduce this noise by decreasing the "Loop Gain" of the "Z-Controller".
Get used to your running measurements and the values of all parameters you can adjust.

By default, each completed measurement is temporarily stored (automatically) on your computer so that it can be used later. Additionally, you can also take snapshots of measurements still in progress. To do this click the "Capture" button in the Chart bar:



The current measurement is immediately stored and will show up in the History page of the Gallery panel, together with all other finished/stored measurements (see *Section 4.3.3: Gallery panel* (page 36)) for details). In addition, the captured document will remain open in the Document space of the SPM Control Software

6.2 The Graphite Surface

In a good Color Map chart of graphite, you will see a pattern consisting of bright, intermediate, and dark spots. It looks like a three dimensional image of balls lying next to each other, but be careful: These are not the single atoms! To interpret the image correctly, you must first be aware that bright spots show topographic high points and dark spots low ones. In the lattice model of graphite you can see that there are two different positions of the carbon atoms in the graphite crystal lattice:

- An atom with a neighboring atom in the plane below (gray) and one without a neighbor in the lattice below (white). As a consequence, the electrical conductivity of the graphite surface slightly varies locally, so that the atoms without neighbors appear higher (brighter) than the others. If carbon atoms have direct neighbors in the layer below, these atoms will drag electron density from those above so they appear lower (darker). Black Spots correspond to the space between the surrounding carbon atoms of a C_6-ring, showing a minimum in the electron density of the surface.

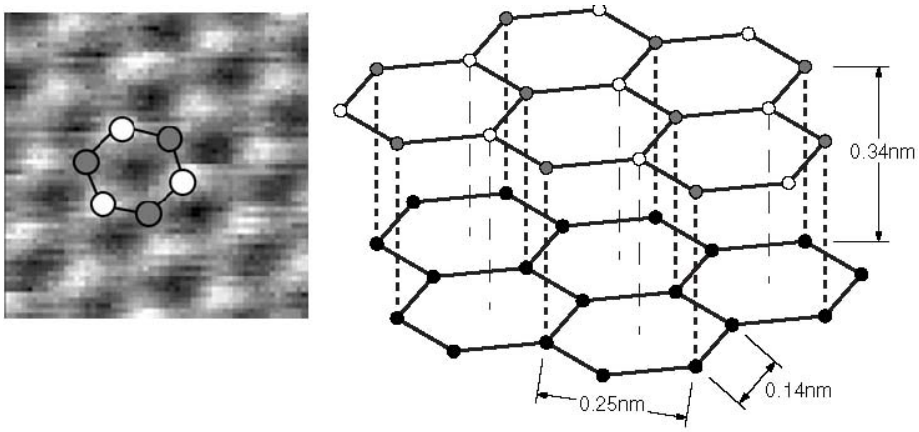


Fig. 6-3 Layered structure of Graphite.

This layered structure is the reason why the lattice constant between the bright 'hills' to have the higher than normal value of 0.25 nm (value from various literature is 0.246 nm).

6.3 Improving measurement quality

Removing interfering signals
Interfering signals can be recognized because they have a fixed frequency, usually a multiple of the local mains frequency (50 or 60 Hz) throughout the image. Thus, they are manifested by straight lines that run throughout the entire image. Possible interference sources are:

- Mechanical vibrations from machines or heavy transformers in direct vicinity (e.g. pumps, Pc ventilators).
- Electrical interference (e.g. cathode ray tubes, neon lamps).

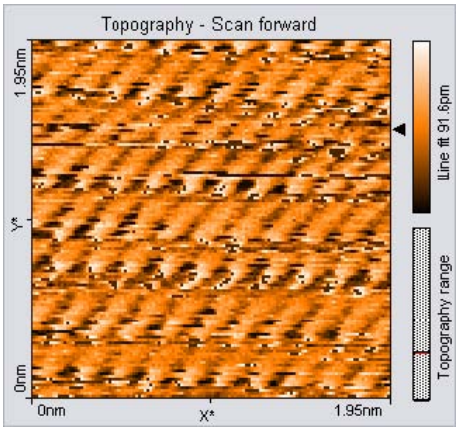
Decreasing thermal drift
Temperature variations cause so-called "thermal drift". This will cause images to be distorted. This effect is present when the observed upward scan is very different from the downward scan, for example showing two differently distorted lattices.
Thermal drift is very clearly visible on an atomic scale. Variations of 0.1°C already cause variations of several nanometers in length of (for example) the steel sample holder.
To decrease thermal drift, keep the measurement running for some time to let the system stabilize (up to about one hour), and prevent air currents in the room from reaching the scan head.

Adjusting the measurement plane
Ideally, the sample surface and the XY-plane of the scanner run parallel to each other. In most cases, however, the sample plane is tilted with respect to the XY-plane of the scanner. In this case, the sample cross section in the X* measurement direction has a certain slope. This slope is undesirable for several reasons:

- It makes it difficult to see small details on the sample surface, because the Average, Plane fit, or higher order filters cannot be used properly.
- The Z-Controller functions less accurately, because it continuously has to compensate for the sample slope.

After approach, the measurement plane should therefore be adjusted electronically. This can be done automatically or manually (see *Section 11.5.1: Imaging* (page 98)).

6.4 Judging Tip and Tunneling Contact Quality



When all prerequisites for the measurement are optimal, the measurement quality mainly depends on the quality of the tip and the tunneling contact. A sharp tip and a good tunneling contact are necessary for high quality images of atomic resolution. If during a good measurement the image quality diminishes dramatically, the tip has most probably picked up some particles or you are near a layer edge on the sample surface.

In this case: Continue measuring for a while (4–5 images). The tip may eventually lose the picked up material again. If this does not help, try to induce changes at the tip's end, using one of the following procedures.

- While measuring, increase the gap voltage in the "Feedback" Panel to 2 V and then reduce it to the old value again.
- Increase the tunneling current to 20 nA for a short period of time and then reduce it to its old value again.

Retract the sample (either by pressing "Withdraw" or "Retract") and then perform a new approach. If no improvement can be seen after going through these procedures, you have to prepare a new tip.

Examples of images made with unusable tips. Prepare a new tip when your image looks like one of the examples below.

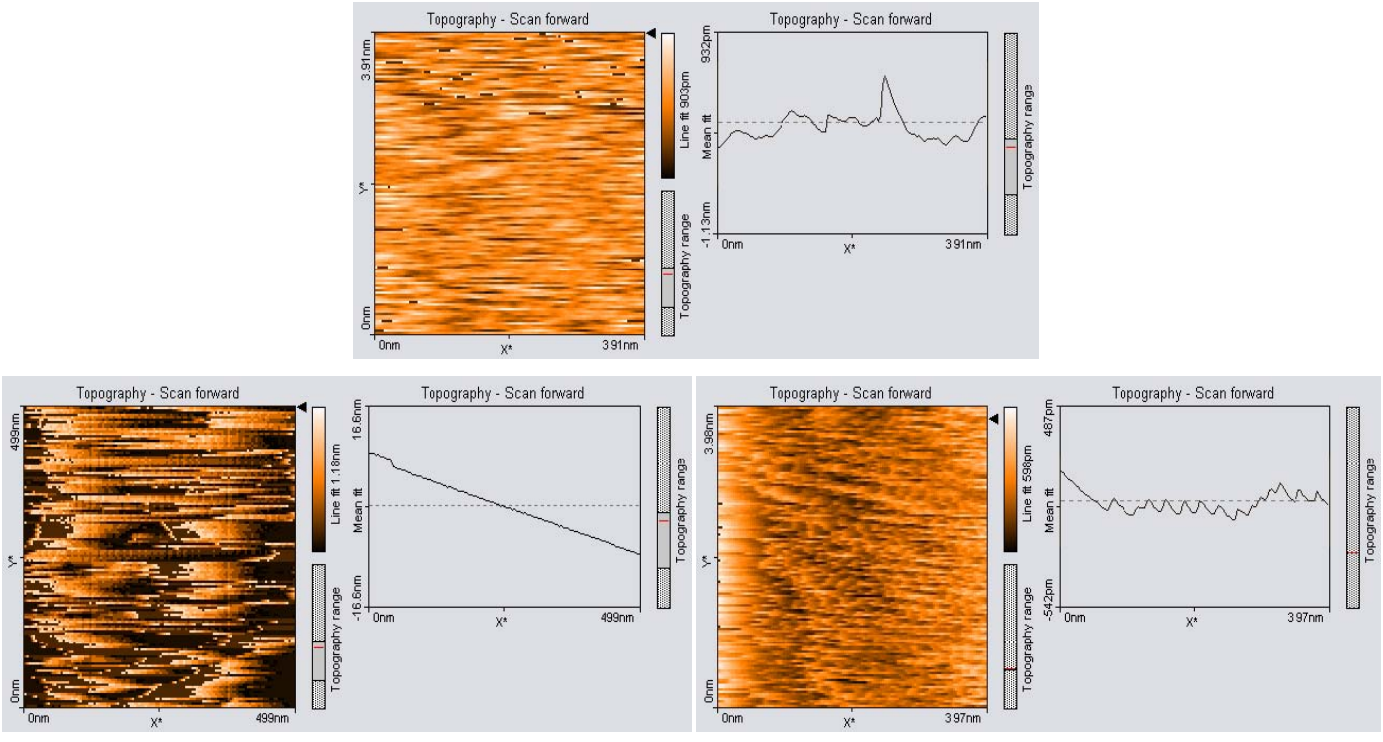


Fig. 6-4 Samples of images taken with crashed or otherwise unusable tips.

CHAPTER 7:

Finishing Scanning

7.1 Shutting Down the System

Once you are done measuring:

1. Click either “Withdraw” to abort the running measurement directly, or stop imaging by pressing “Stop”.
2. Now retract the Sample Holder by clicking “Retract” in the approach panel. Do this until the Sample Holder is far enough away from the tip, so it can be removed safely.
3. Remove the Magnifying cover from the scan head.
4. Remove the Sample Holder by pulling and lifting it from the guide bars.
5. Remove the sample from the Sample Holder with the tweezers. Push it a little on one side over the edge from the Sample Holder. Now you can grab it with the tweezers and store it in its case safely.
6. Store the Sample Holder in its container.

Turning off the Instrument

1. Verify that you have saved all measurements you would like to keep. When you try to close unsaved photos you have taken, you will be asked if you want to save these in a specific folder on your hard drive. Browse to a folder or create a new one to store your measurements.
2. Exit the control software.
3. Turn off the power switch on the side of the control unit.

Storing the Instrument

If you perform measurements regularly:

- Leave the instrument with the Magnifying cover glass over the scan head to protect it against dust.

If you are not using the instrument for an extended period of time, if you have to transport it, or if you send it in for repairs, put the instrument in the instrument case by following these steps:

1. Turn off the instrument as described and remove all cables.
2. Remove the sample and Sample Holder. The tip can be left in the scanner.
3. Store the Sample Holder in its container.
4. Pack all components in the original packaging material and store them in the instrument case.

IMPORTANT

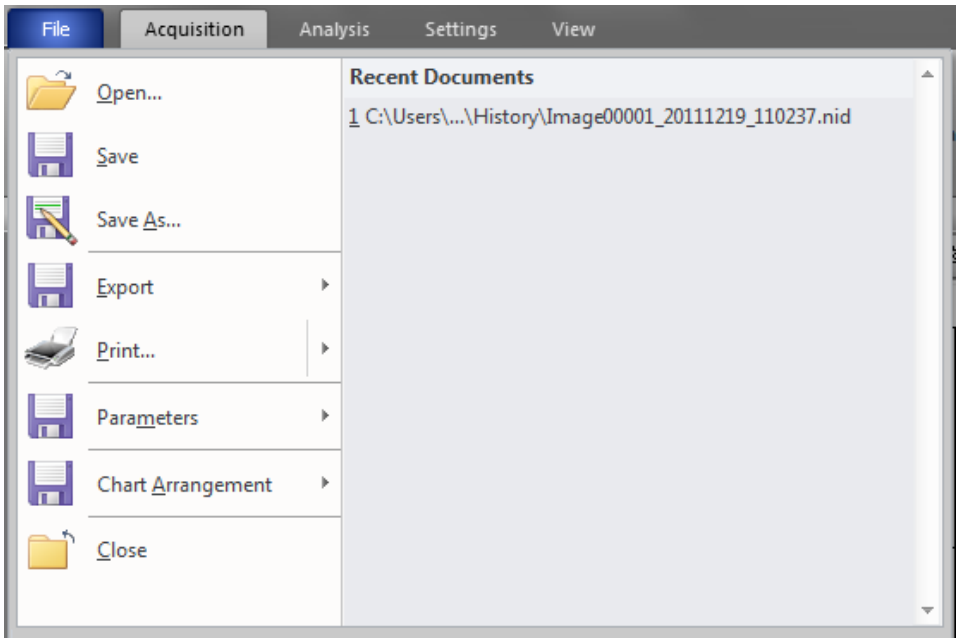
Before transporting your microscope, always put the instrument and tools in the original packaging material and store everything in the instrument case.

7.2 Storing and Printing Measurements

Storing and Printing of measurement documents can be performed using the File menu. The functions Open, Save and Print are also available via the File bar.
Menu item “File” contains the items for opening , closing, saving , and
printing the measurement documents and for exiting the program. In the following only the special functions are mentioned.

7.2.1 Open

Opens a dialog for opening PHYWE Measure Nano image data “.nid” files. The same dialog is opened using the menu “File” >> “Open...”. It is possible to select more than one file at the same time by using the “Shift” and “Ctrl” keys.



7.2.2 “Save” and “Save as...”

Press to save a measurement document in PHYWE Measure Nano image data format (file extension “.nid”). The same dialog is opened using the menu “File” >> “Save” and “File” >> “Save as...”.

7.2.3 Export Current Chart as / Current document as

Exports either the active chart or the whole active measurement document for use in other programs or image processing software. Available data types for documents are tagged image file format (.tif), portable network graphics (.png), Windows bitmap (.bmp), 16 bit data file (.dat), and plot file (.plt). For charts, additional available data types are comma separated z values (.csv), and (X,Y,Z)-points (.csv). When the data is exported using the function “Export” >> “Current document as...”, every Chart in the measurement document is stored in the export file consecutively. In the binary format, the blocks of data from each Chart are stored directly one behind the other. In the “ASCII” text format the blocks of data for each Chart are separated by two empty lines.

- *Tagged image file format (.tif), portable network graphics (.png), and Windows bitmap (.bmp)*

All of these image file formats are suitable for inclusion in electronic documents, e.g. of word- or image-processing software. The exact image as seen on the computer screen will be saved in the file (“screen shot”).

- *Data file 16Bit (.dat)*

A binary data file can be processed in image processing software. This “binary” data format contains only the measured data. The data is stored consecutively line by line upwards as 16-bit values (–32768 to +32767). The data is first processed using the settings chosen in the Data filter setting of the Chart bar.

- *Plotfile ASCII (.plt)*

This is an “ASCII” text format which contains the measured data as well as a small header with a description of the scan. The data is stored using the setting “Data filter” in the “Chart bar”. A measurement as a plotfile can be used for detailed data analysis by various mathematical software packages such as
MathLab or plotted by GnuPlot. If “Line graph” is selected as “Display” in the “Chart bar”, only the visualized lines will be stored. Each data point is stored as a pair of floating point numbers on a separate line. The number pairs are separated by a blank character (SPACE). If any other chart type is selected, all measured values are stored. All values in a data line are stored on a separate line in the text file. An empty line is inserted after every data line. The data lines are stored from the bottom to the top. A small header at the beginning of the first data line contains the names of the channel and frame, as well as X-, Y-, and Z-ranges with their physical units.

- *Comma separated z values (.csv)*

This format stores all the measured data in a chart, as a matrix of floating point numbers in ASCII format separated by a “comma” and “SPACE” character. This enables easy data exchange with commonly used spread sheet and database applications.

- *(X, Y, Z)-Points (.csv)*

This format stores the coordinates of all measured points in a chart as a list of floating point number pairs. For Line

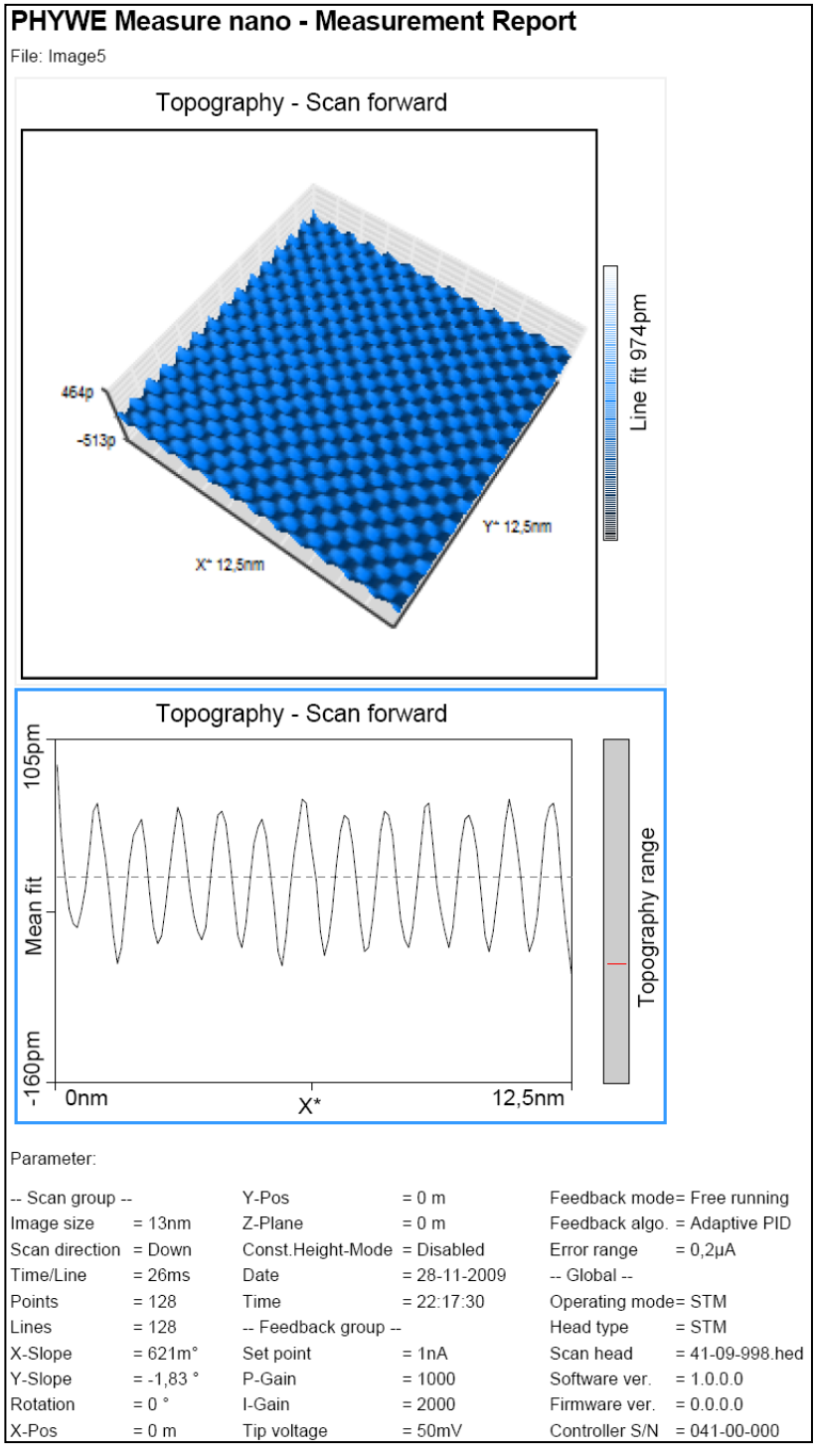


Fig. 7-1 Print preview.

graphs, only X and Z points are exported.

- *Print, Print preview...*

 Prints the currently selected measurement document together with the values shown in the Data Info panel.

7.3 Storing and Retrieving the Chart Arrangement

The chart arrangement of the Imaging and Spectroscopy windows is stored in a configuration file with the extension ".chart". When the PHYWE Measure Nano software is started, a default arrangement is loaded from a file that is selected in the Controller Configuration. Functions for storing and retrieving the chart arrangement are accessed via the menu "File" >> "Chart Arrangement".

- "Save": saves the chart arrangement to the currently selected chart file. The name of this file is indicated in the status bar at the bottom of the main window.
- "Save as...", saves the chart arrangement under a new file name. "Load" loads a previously saved chart file.
- "Load": loads a previously saved chart arrangement file.

7.4 Storing and Retrieving Measurement Parameters

All measurement parameters are stored in a configuration file with the extension ".par". When the PHYWE Measure Nano software is started, default values are loaded from a file that is selected in the Controller Configuration dialog. Functions for storing and retrieving parameters are accessed via the menu "File" >> "Parameters".

- "Save": saves the parameters to the currently selected parameter file. The name of this file is indicated in the status bar at the bottom of the main window.
- "Save as...": saves the parameters under a new file name.
- "Load": loads a previously saved parameter file.

CHAPTER 8:

Maintenance

To ensure fault-free operation of the microscope the following instructions for maintenance have to be followed.

It is very important to keep the Sample Holder and the open parts of the scanner clean. If it exposed to moisture (high humidity), corrosion will occur.

8.1 Protecting the Sample Holder against Corrosion

The Sample Holder is made of magnetic steel and therefore suffers from corrosion in a humid environment. The approach motor will not run well if the Sample Holder is dirty or corroded. To reduce corrosion and increase life expectancy, the Sample Holder must be stored in its container, together with the moisture absorbing Silica Container. The container is waterproof but not airtight. The silica contains a blue indicator that turns pink when saturated.

Fig. 8-1 Sample holder and its package and silica container to prevent corrosion.

To regenerate the Silica:

- Heat the Silica Container at 100°C in the oven for at least two hours until it turns completely blue again.

8.2 Cleaning Parts of the Microscope

If you have touched the metal part of the Sample Holder or it has otherwise become dirty, or if the approach motor does not move, the Sample Holder must be cleaned.

Cleaning the Sample Holder

1. Take a soft cloth, if necessary moistened with alcohol.
2. Place the Sample Holder downwards on a clean table. Clean the Sample Holder by moving the cloth along the Sample Holder in the axial direction, up- and downwards. Do not move it around its circumference!
3. Let the parts dry before operating the motor again.

If the Approach Motor still does not move

1. Take a cotton swab, if necessary moistened with alcohol.
2. Clean the Sample Holder guide bars.
3. Clean the surfaces of the approach motor that touch the Sample Holder.
4. Clean the tip holder (remove the tip when doing this).
5. Let all parts dry before operating the motor again.

The problems described here can occur during normal operation of the microscope.

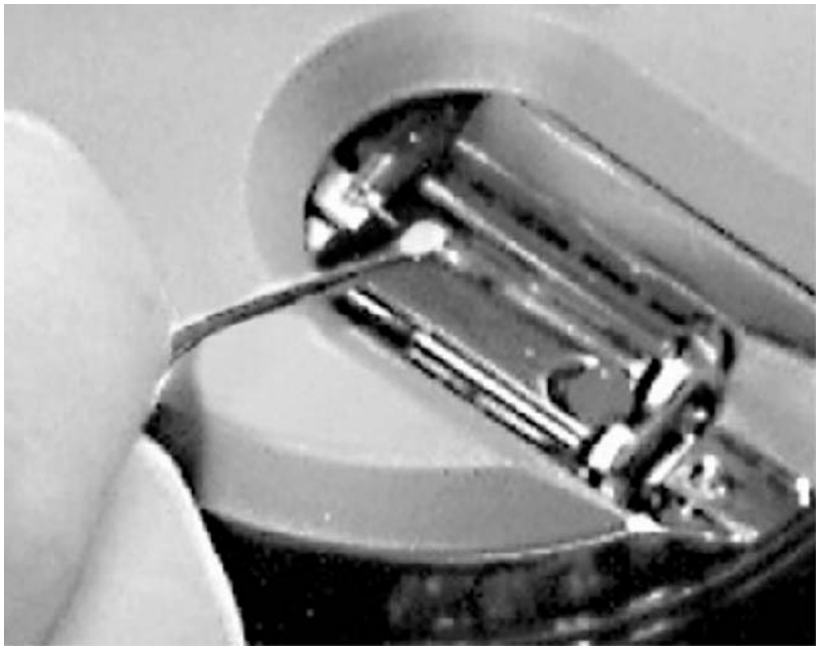


Fig. 8-2 Carefully clean the sample holder guidebars and the motor unit with alcohol using a soft cloth or a cotton swab.

CHAPTER 9:

Problems and Solutions

9.1 STM Measurement Problems

The problems described here can occur during normal operation of the microscope.

9.1.1 Manual Approach is too slow / stops sometimes

If the manual approach using the “Advance” or “Retract” button is affected:

- Clean the Sample Holder, the guide bars and the surfaces of the approach motor following the procedure described earlier (*Chapter 8.2: Cleaning Parts of the Microscope*, page 74).

9.1.2 Automatic Final Approach is too slow / stops sometimes

Even if the manual approach works, the automatic final approach (using the “Approach” button) may not work.

- Clean the Sample Holder, the guide bars and the surfaces of the approach motor following the procedure described earlier (*Chapter 8.2: Cleaning Parts of the Microscope*, page 74).

If cleaning does not help, the step size of the motor unit may be too small. To solve this problem:

1. Open the “User Interface Configuration” dialog using the menu “Options” >> “Config User Interface...”.
2. Set the “User Interface Mode” to “Standard level” or “Advanced level”.
3. Increase the value of Appr. Speed in the “Approach Options” section of the approach panel by a few percent until the approach works. Now the motor moves the Sample Holder with larger steps during the automatic approach.
4. Save the new value of Appr. Speed using the menu “File” >> “Parameters” >> “Save”.

9.1.3 Automatic Final Approach crashes the Tip into the Sample

In this case the motor moves the Sample Holder towards the tip with too large steps:

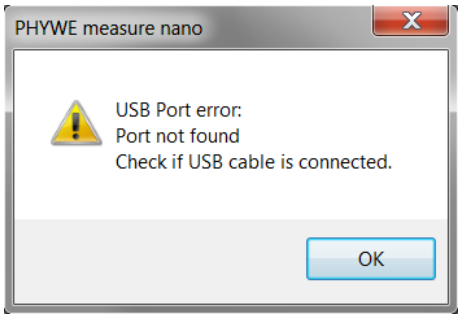
1. Open the “User Interface Configuration” dialog using the menu “Options” >> “Config User Interface...”.
2. Set the “User Interface Mode” to “Standard level” or “Advanced level”.
3. Decrease the value of Appr. Speed in the “Approach Options” section of the approach panel by 10%.
4. Repeat the approach with a new tip. If the approach fails again, reduce Appr. Speed further.
5. Save the best Appr. Speed value using the menu “File” >> “Parameters” >> “Save”.

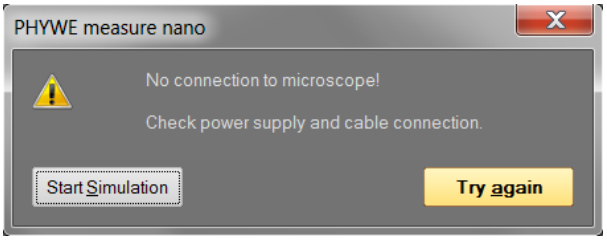
9.1.4 Software and Driver Problems

The USB serial converter is not available
The USB cable is not properly connected. In this case the USB power light on the microscope controller rear panel does not light up. To fix this problem:

- Check if there is a second copy of the PHYWE Measure Nano software is already running and occupying the USB port.
- Check that the USB cable is properly connected.

If this does not solve the problem, check if there is a driver problem with the USB Serial port/USB Serial converter drivers, as described in the next section.





Driver problems
If you have trouble connecting to the controller, it is possible that one of the drivers of your instrument is causing problems, for example because the installation did not work, or the installation of some other hardware is in conflict with the drivers of the PHYWE STM.
In order to solve driver problems:

1. Check for driver updates on www.phywe.com (Product page).
2. Insert the installation CD for your instrument.
3. Log in with Administrator privileges.

The device manager can then be opened to view and correct any driver problems:

1. Open the windows menu “Start” >> “Settings” >> “Control Panel”. The control panel now opens.
2. Click “Switch to classic view” if you do not see an icon called “System”.
3. Double-click the System icon. The System properties dialog now opens.
4. Select the tab “Hardware”
5. Click the “Device Manager”-button. The device manager now opens.

When the device manager opens and your controller is connected to your computer, you may see these drivers (information may vary depending on the configuration of your system):

• Generic USB Hub
• USB Serial converter, USB Serial port

If there are problems with any of these drivers, or a wrong driver is installed, you can try the following to fix it:

1. Double click on the driver. Properties dialog for the device now opens.

2. Select the “Driver”-tab.
3. Click the “Update Driver”-button. Windows will now ask you were to look for the driver.
4. Tell windows to take the driver from the Installation CD.

9.2 Image Quality Suddenly Decreases

There are several possible causes for this phenomenon.

9.2.1 Z-Drift

The tip drifted outside the Z-range of the scanner. In this case, the “Probe Status” light will either light up yellow or red.

If the light is yellow, the tip has lost contact with the sample:

- Click “Withdraw” in the approach panel and approach afterwards to reestablish the contact to the sample again.

If the light is red, the tip has drifted into the sample. You can try to move the sample surface within the Z-range of the scanner, although the tip may already have been damaged:

- Click “Withdraw” in the approach panel.

If the light is still red after withdrawing, try to withdraw again. Now you can approach the sample again by pressing “Approach”. If the image quality is bad or you are not able to establish a good tunneling contact, prepare a new STM tip.

9.2.2 XY-Drift

The scanner may have drifted close to a deformity in the sample's surface. Try to find a different measurement position:

1. Increase the scan range.
2. Try to find a new flat region.
3. Use the “Zoom” tool to decrease the image size again.

If you can’t find a good area for measurements you can withdraw, retract a little and turn the Sample Holder slightly to advance and approach to a new area on the sample afterwards.

9.2.3 Tip Modification

The tip may have picked up some particles or other material from the sample surface.
Try to give a Cleaning Pulse using the corresponding button in the “Tip Properties” panel.

9.3 Support

9.3.1 Self Help

The fastest way to solve a problem is often to solve it yourself. If the previously suggested actions did not help, or the problem is not described here, refer to the PHYWE support pages:

- Open http://www.phywe-systeme.com/
- Browse to the product page in the “Product Catalog” (for example search for “Tunneling”).

9.3.2 Assistance

If the standard solutions are not sufficient, contact your local distributor for help. In order to resolve the problem as fast as possible, please provide as much information as possible, such as:

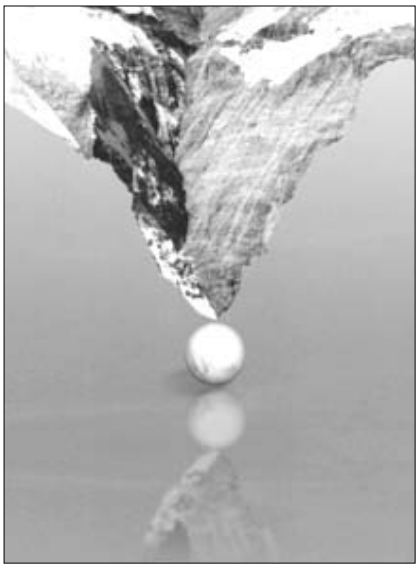
- A detailed description of what happened before the problem occurred.
- If an error message was displayed: The exact text of the message.
- The serial number of your Scan Head which you can find on the bottom of your controller unit.
- A description of the computer hardware and software on which the control software is running: computer brand, type (laptop or desktop), operating system, software version etc.
- Original PHYWE Measure Nano image data (.nid) files that show the problem, rather than bitmap screen shots, because these files contain all the settings that were used to make them.
- Parameter (.par) files with the instrument settings that were used when the problem occurred.

CHAPTER 10:

STM Theory

10.1 What is Scanning Tunneling Microscopy?

Microscopy is one of the most exciting scientific techniques. The insight into small dimensions has led to a new understanding of the structure of materials and forms of life. With the help of the scanning tunneling microscope (STM) it is possible to look into the fascinating world of the atoms. This completely new microscopy technique works without focusing a light beam on a specimen and features atomic resolution (laterally and vertically). The Scanning Tunneling Microscope was developed by Gerd Binnig and Heinrich Rohrer in the early 80's at the IBM research laboratory in Rueschlikon, Switzerland. For this revolutionary innovation Binnig and Rohrer were awarded the Nobel Prize in Physics in 1986.



In the STM, a small sharp conducting tip is scanned across the sample's surface, so close that the so-called "tunneling current" can flow. With the help of that current, the tip-surface distance can be controlled very precisely. Therefore an enormous resolution is achieved so that the atomic arrangement of metallic surfaces can be "probed". Being able to get such excellent pictures of atomic resolution is almost incredible, considering that the size of an atom in relation to the tip is that of a golf ball to a mountain.

In your PHYWE measurement system a platinum-iridium tip is moved in three dimensions using piezo-crystal translators that are driven with sub-nanometer precision. The sample to be examined approaches the tip within a distance of 1 nanometer (1 nm = 1/1,000,000,000 m). Classical physics would prohibit the appearance of electrons in the small gap between the tip and the sample, but if a sharp tip and a conducting surface are put under a low voltage (U~0.1V), a very small tunneling current (I~1nA) though may flow between tip and sample. This tunneling current is due to a quantum physics effect.

The strength of the tunneling current depends exponentially on the distance between the tip and the sample (usually referred to as Z-distance). This extreme dependence on the distance makes it possible to measure the tip–sample movement very precisely. One of the three piezo crystals, the Z-piezo, can now be used in a feedback loop that keeps the tunneling current constant by appropriately changing the Z-distance.

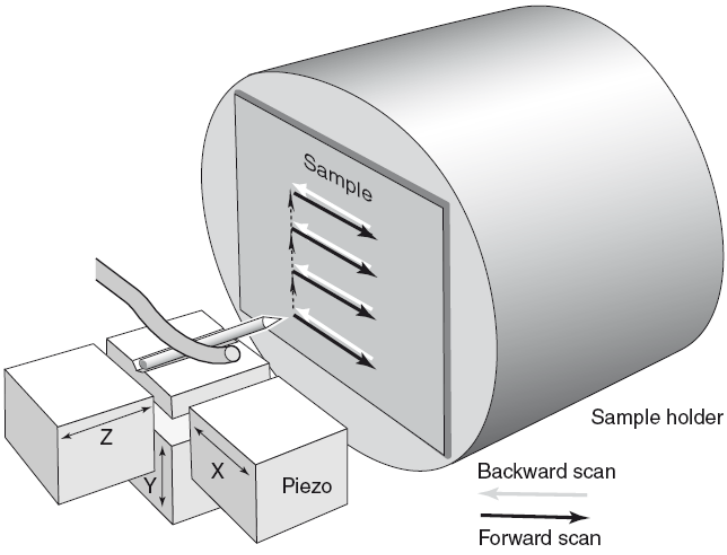
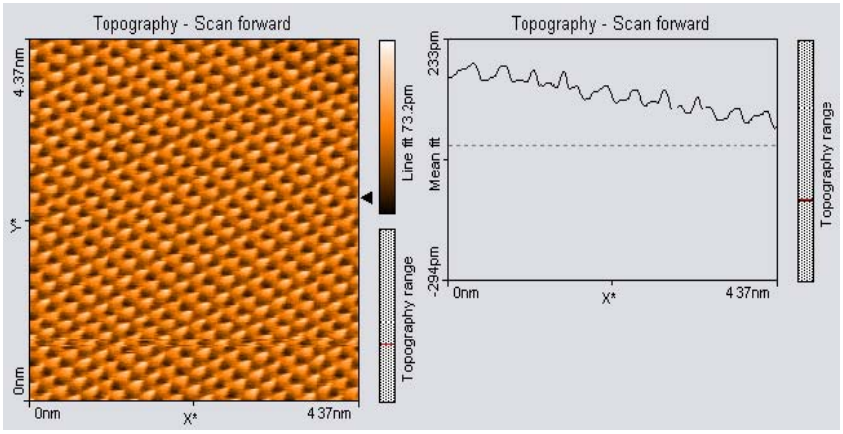


Fig. 10-1 Tip movement during a measurement Controlled with a three axis pieozo-electric device

To obtain an image of the sample, the tip is scanned using the X- and Y-piezo crystals. The feedback loop will now let the tip follow the structure of the sample's surface. A height image can be made now by recording the position of the Z-feedback loop as a function of the XY-piezo position. This 'landscape' (or topography) of the atomic surface is then drawn line by line on the computer screen.



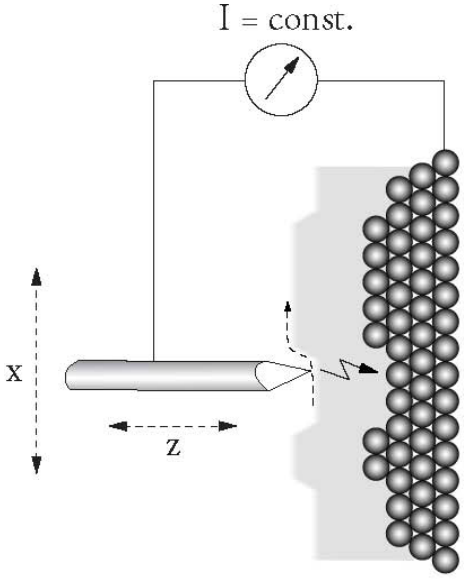


Fig. 10-2 Color map and Line Graph representation of measured data (left). Tunneling current measurement leads to surface topography (right).

The sample can also be scanned in a second mode: When the feedback loop is slowed down greatly, the tip scans at a fixed distance from the sample ("constant height mode"). This time the variations in the tunneling current are measured, and drawn line by line on the computer screen. However, this mode only works when the sample is atomically flat, because the tip would otherwise 'crash' into the sample.

A perfect tip has only one atom at its end that will realize the tunneling contact.

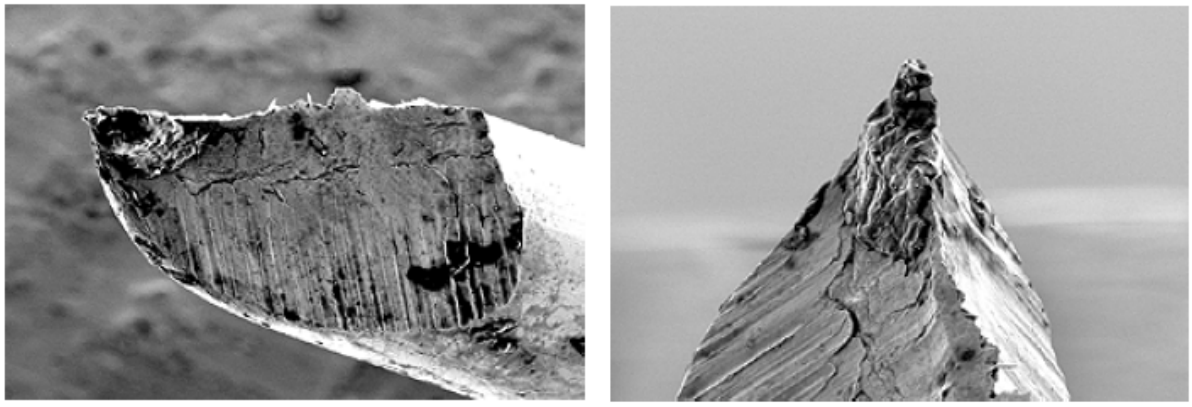
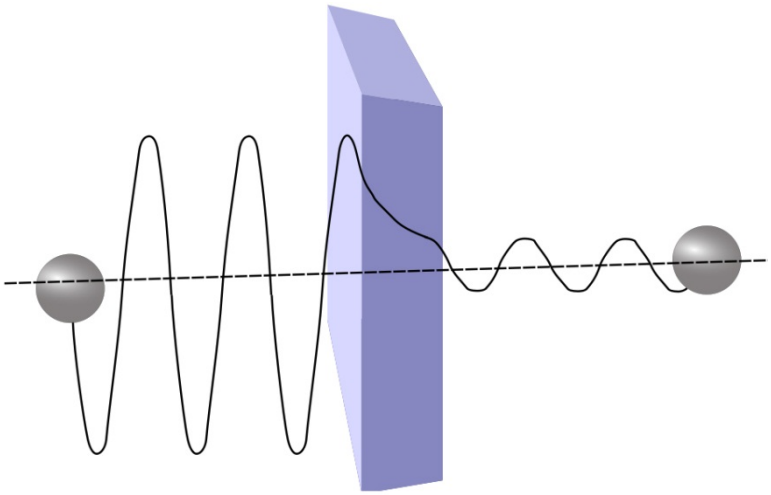


Fig. 10-3 Scanning electron micrographs of a STM tip.

10.2 Quantum Mechanics

Tunneling is a functioning concept that arises from quantum mechanics. Classically, an object hitting an impenetrable barrier will not pass through. In contrast, objects with a very small mass, such as the electron, have wavelike characteristics which permit such an event, referred to as tunneling. In other words, the probability to find an electron behind a barrier is unequal zero. Inside the barrier the wave function of the electron decays exponentially.



Electrons behave as beams of energy, and in the presence of a potential $U(z)$, assuming a 1-dimensional case, the energy levels $\psi_n(z)$ of the electrons are given by solutions to Schrödinger's equation,

$$-\frac{\hbar^2}{2m}\frac{\partial^2\psi_n(z)}{\partial z^2} + U(z)\psi_n(z) = E\psi_n(z)$$

where $\hbar$ is the reduced Planck's constant, z is the position, and m is the mass of an electron[4]. If an electron of energy E is incident upon an energy barrier of height $U(z)$, the electron wave function is a traveling wave solution,

$$\psi_n(z) = \psi_n(0)e^{\pm ikz}$$

with

$$k = \frac{\sqrt{2m(E-U(z))}}{\hbar}$$

if $E > U(z)$, which is true for a wave function inside the tip or inside the sample. Inside a barrier, such as between tip and sample, $E < U(z)$ so the wave functions which satisfy this are decaying waves,

$$\psi_n(z) = \psi_n(0)e^{\pm\kappa z}$$

where

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

quantifies the decay of the wave inside the barrier, with the barrier in the $+z$ direction for $-\kappa$.

Knowing the wave function allows one to calculate the probability density for that electron to be found at some location. In the case of tunneling, the tip and sample wave functions overlap such that when under a bias, there is some finite probability to find the electron in the barrier region and even on the other side of the barrier. Let us assume the bias is V and the barrier width is W.

This probability, *P*, that an electron at *z*=0 (left edge of barrier) can be found at *z*=*W* (right edge of barrier) is proportional to the wave function squared,

$$P \propto |\psi_n(0)|^2 e^{-2\kappa W}$$

If the bias is small, we can let $U - E \approx \varphi M$ in the expression for κ, where φM, the work function, gives the minimum energy needed to bring an electron from an occupied level, the highest of which is at the Fermi level (for metals at *T*=0 kelvins), to vacuum level. When a small bias *V* is applied to the system, only electronic states very near the Fermi level, within *eV* (a product of electron charge and voltage, not to be confused here with electronvolt unit), are excited. These excited electrons can tunnel across the barrier. In other words, tunneling occurs mainly with electrons of energies near the Fermi level.

However, tunneling does require that there is an empty level of the same energy as the electron for the electron to tunnel into on the other side of the barrier. It is because of this restriction that the tunneling current can be related to the density of available or filled states in the sample. The current due to an applied voltage *V* (assume tunneling occurs sample to tip) depends on two factors: 1) the number of electrons between E_f and *eV* in the sample, and 2) the number among them which have corresponding free states to tunnel into on the other side of the barrier at the tip. The higher density of available states the greater the tunneling current. When *V* is positive, electrons in the tip tunnel into empty states in the sample; for a negative bias, electrons tunnel out of occupied states in the sample into the tip.

Mathematically, this tunneling current is given by

$$I \propto \sum_{E_f - eV}^{E_f} |\psi_n(0)|^2 e^{-2\kappa W}$$

.

One can sum the probability over energies between $E_f - eV$ and *eV* to get the number of states available in this energy range per unit volume, thereby finding the local density of states (LDOS) near the Fermi level. The LDOS near some energy *E* in an interval ε is given by

$$\rho_s(z, E) = \frac{1}{\epsilon} \sum_{E-\epsilon}^{E} |\psi_n(z)|^2$$

and the tunnel current at a small bias V is proportional to the LDOS near the Fermi level, which gives important information about the sample. It is desirable to use LDOS to express the current because this value does not change as the volume changes, while probability density does. Thus the tunneling current is given by

$$I \propto V \rho_s(0, E_f) e^{-2\kappa W}$$

where $\rho_s(0,E_f)$ is the LDOS near the Fermi level of the sample at the sample surface. By using equation (6), this current can also be expressed in terms of the LDOS near the Fermi level of the sample at the tip surface,

$$I \propto V \rho_s(W, E_f) V$$

The exponential term in (9) is very significant in that small variations in W greatly influence the tunnel current. If the separation is decreased by 1 Å, the current increases by an order of magnitude, and vice versa.

This approach fails to account for the *rate* at which electrons can pass the barrier. This rate should affect the tunnel current, so it can be treated using the Fermi's golden rule with the appropriate tunneling matrix element. John Bardeen solved this problem in his study of the metal-insulator-metal junction (MIM). He found that if he solved Schrödinger's equation for each side of the junction separately to obtain the wave functions ψ and χ for each electrode, he could obtain the tunnel matrix, M, from the overlap of these two wave functions. This can be applied to STM by making the electrodes the tip and sample, assigning ψ and χ as sample and tip wave functions, respectively, and evaluating M at some surface S between the metal electrodes, where z=0 at the sample surface and z=W at the tip surface.

Now, Fermi's Golden Rule gives the rate for electron transfer across the barrier, and is written

$$w = \frac{2\pi}{\hbar}|M|^2\delta(E_\psi - E_\chi)$$

where δ(Eψ-Eχ) restricts tunneling to occur only between electron levels with the same energy. The tunnel matrix element, given by

$$M = \frac{\hbar}{2\pi}\int_{z=z_0}(\chi * \frac{\partial\psi}{\partial z} - \psi\frac{\partial\chi*}{\partial z})dS$$

is a description of the lower energy associated with the interaction of wave functions at the overlap, also called the resonance energy.

Summing over all the states gives the tunneling current as

$$I = \frac{4\pi e}{\hbar}\int_{-\infty}^{+\infty}[f(E_f - eV) - f(E_f + \epsilon)]\rho_s(E_f - eV + \epsilon)\rho_T(E_f + \epsilon)|M|^2 d\epsilon$$

where *f* is the Fermi function, ρ_s and ρ_T are the density of states in the sample and tip, respectively. The Fermi distribution function describes the filling of electron levels at a given temperature T.

Further reading:

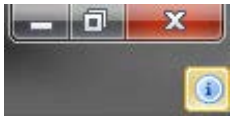
- Tersoff, J.: Hamann, D. R.: Theory of the scanning tunneling microscope, Physical Review B 31, 1985, p. 805 - 813.
- Bardeen, J.: Tunnelling from a many-particle point of view, Physical Review Letters 6 (2), 1961, p. 57-59.
- Chen, C. J.: Origin of Atomic Resolution on Metal Surfaces in Scanning Tunneling Microscopy, Physical Review Letters 65 (4), 1990, p. 448-451
- G. Binnig, H. Rohrer, Ch. Gerber, and E. Weibel, Phys. Rev. Lett. 50, 120 - 123 (1983)

CHAPTER 11:

Advanced Users Section

11.1 About dialog

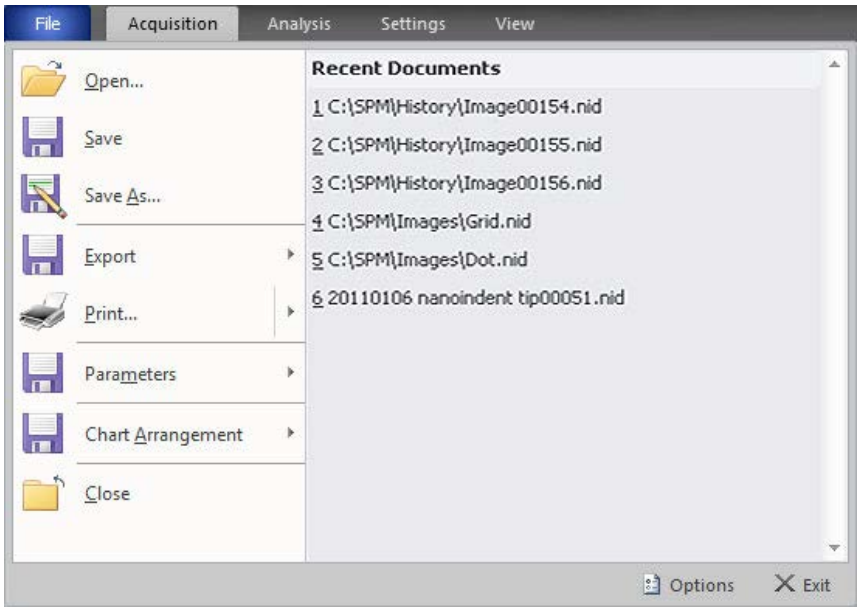
The About dialog displays information that may be useful for diagnostics when you have problems with your instrument. The About dialog is opened by clicking the “Information” button on the upper right corner of the program window, just below the “close window” button:



The About dialog contains the following information:

- The version number of the control software.
- The serial number of the controller (when the microscope simulation is active, the serial number "0xx-00-000" is displayed).
- The version number of the firmware that is running on the controller.
- The version number of all modules built into the controller.
- The version number of all installed software options.
- Contact information for getting more support.

11.2 File Menu



The File menu is accessed by clicking the blue File tab at the left side of the Ribbon. It provides access to mostly software related settings and options, but also to functionality such as opening, storing and printing of measurements. The latter function can also be performed using the Quick Access toolbar, in which these commands are present by default:

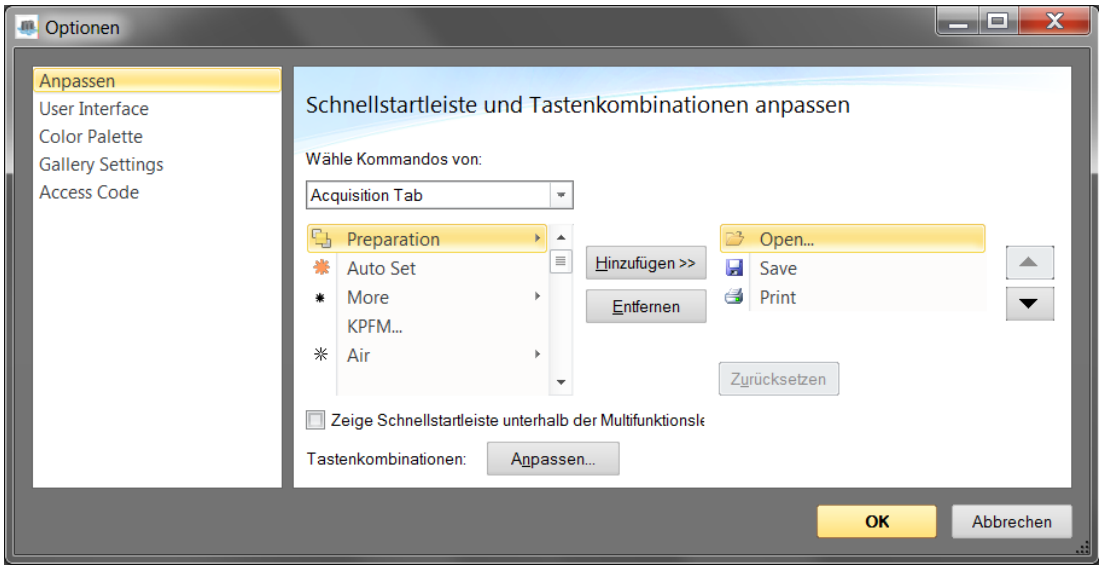
Options
The “Options” button opens the Options dialog (*Section 11.3: Options dialog*), which configures various general control software settings.

Exit
With exit you can close the SPM Control Software. If you exit the program while still having unsaved data, you will be asked to save it.

For detail on the remaining menu entries see chapter 7.2 – 7.4.

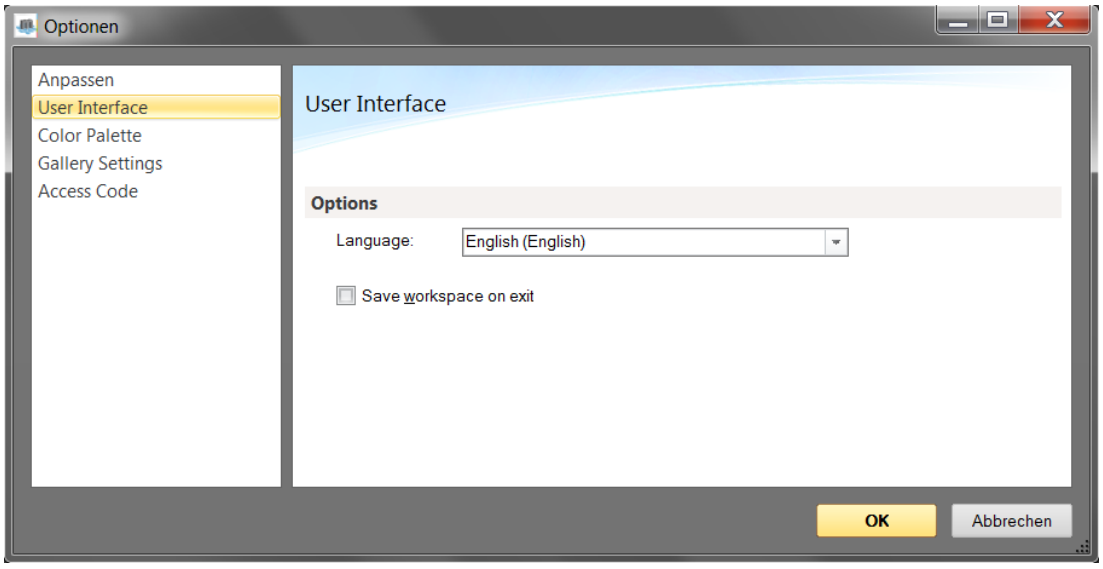
11.3 Options Dialog

Customize



Allows changes to the content of the Quick Access toolbar (see *File menu* (page 89)). This process is very similar to that in the Microsoft Office applications and is therefore not explained in this manual. You are free to try it out and can always use the “Reset” button to reload standard settings.

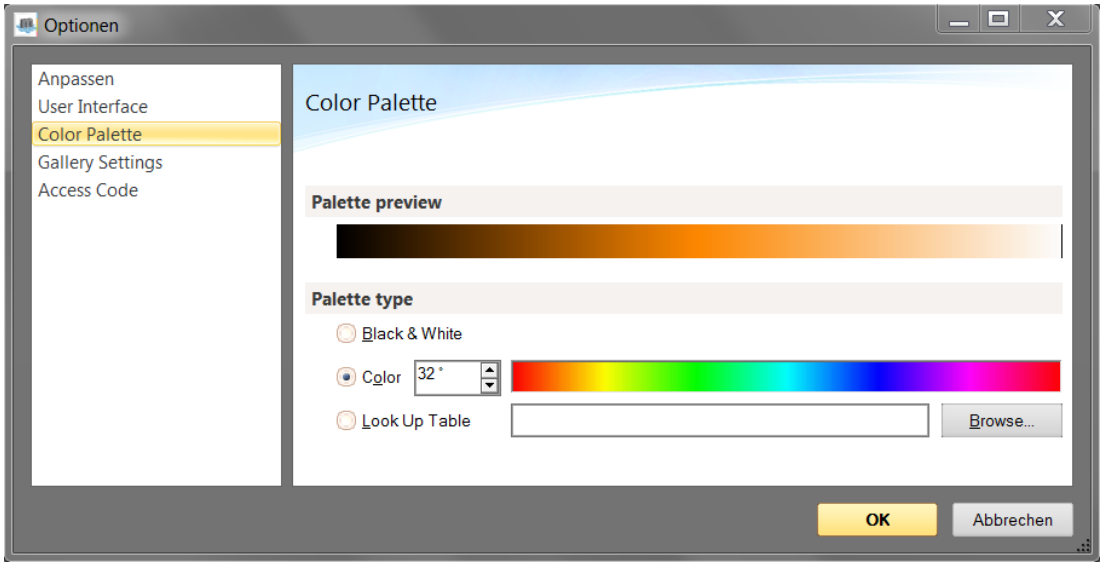
User Interface



Language
Specifies the SPM Control Software language.

Save workspace on exit
When checked, the workspace settings are automatically saved to the system registry when the control software is closed (see also *Chapter 7.3: Storing and retrieving the chart arrangement* (page 72)).

Color Palette



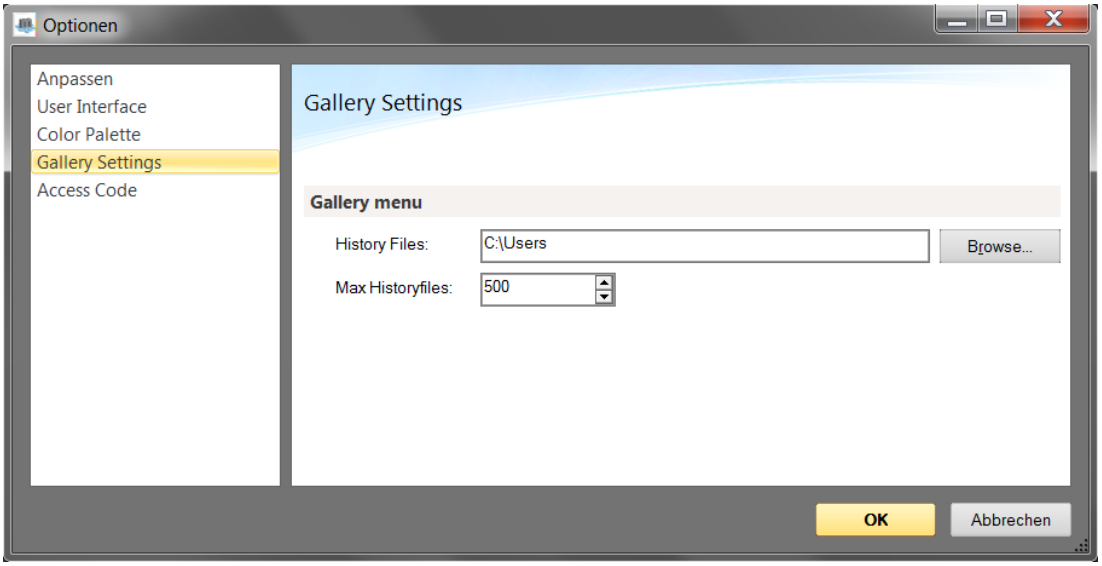
The color palette dialog is reached via the menu item “Options” >> “Config Color palette...”. The color palette is used to map the display range of the measured values to a color. Three different palette types are available:

- **Black&White** The color map is a linear gray scale.

- **Color** The color selection uses the HSB-color model where the color (H) is set in ° value. The color is selected by entering a number or by clicking a color in the color bar.

- **Look Up Table** A user definable palette (with max 256 color entries) can be selected. This palette is stored in a “.lut” file that contains an ASCII table with RGB color values. A different look up table can be selected by clicking the “Browse...” button.

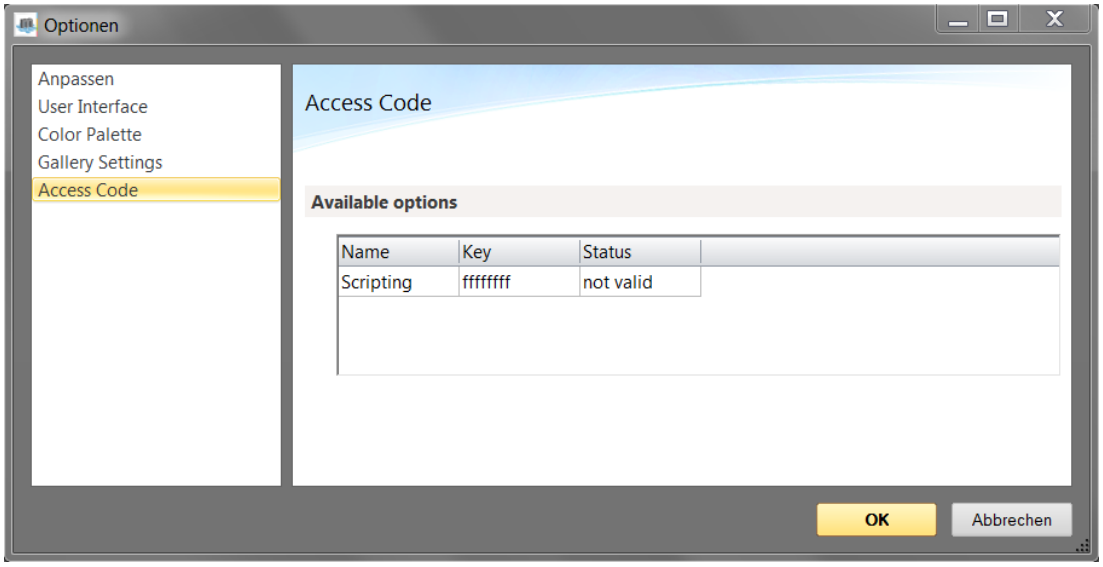
Gallery Settings



History files
Sets the directory where the temporarily (automatically) stored measurements (which are listed in the Gallery panel of the Info pane) are stored.

Max History Files
Sets the maximum number of files to keep in the above directory. When the maximum is reached, the oldest measurement is deleted from disk to allow the latest measurement to be saved.

Access Code

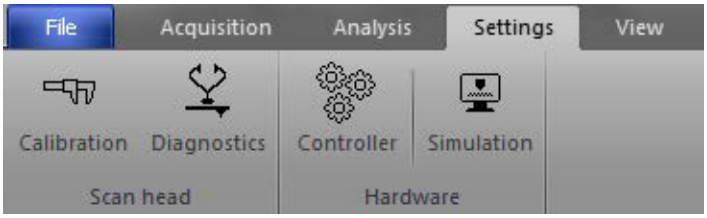


Used to enter the access code for software modules, such as the Scripting Interface and the Lithography Option..."
Provides access to many hardware related settings.

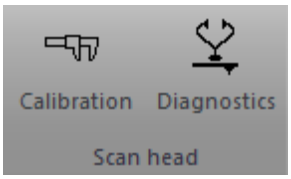
Note

If you receive the warning "To change access codes you need Windows administrator rights", please re-start the control software with the "Run as Administrator..." option from the Windows Explorer context menu.

11.4 Settings tab



Provides access to many hardware related settings.

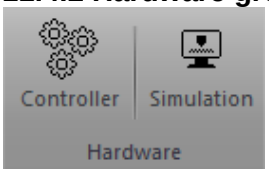


11.4.1 Scan head group

Calibration
This button opens the Scan Head Selector dialog to load, save or edit a scan head calibration file. For more details, see *Scan Head Selector dialog* (below).

Diagnostics
This button opens the Scan Head Diagnosis dialog where the actual health state of the scan head can be seen. For more details, see *Section Scan Head Diagnostics dialog* (page 96).

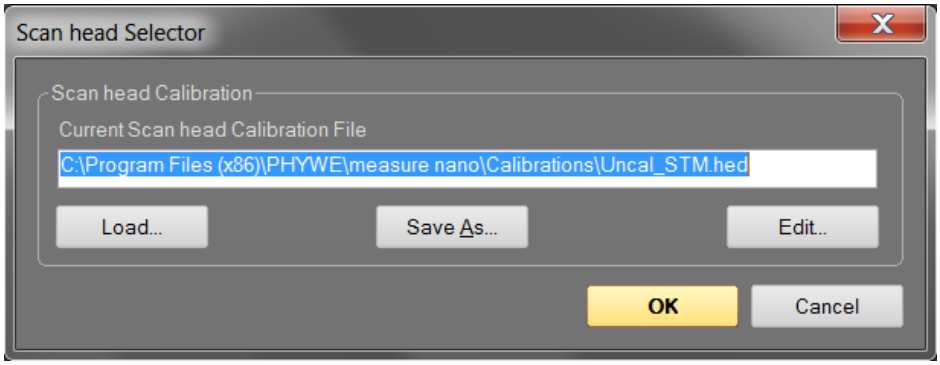
11.4.2 Hardware group



Controller
Opens the Controller Configuration dialog where different hardware related settings can be defined. It defines communications port, video driver settings, startup parameter and others. For more details, see *Controller Configuration dialog* (page 97).

Simulation
Check or uncheck the Simulation button to enter or exit the control software's microscope simulation mode. Once the simulation mode is active, the status bar of the control software displays the text "Simulation". Otherwise, this field displays the text "Online". In microscope simulation mode, many functions of the microscope are performed on a mathematically generated surface. Thus, software functionality and acquisition procedures can be practiced without danger of harming the instrument.

Scan Head Selector dialog



The Scan Head Selector dialog is used to load, save or edit scan head calibration files. These files store all calibration values specific to a certain scan head. The Scan Head selector dialog is opened via the "Calibration" button in the Scan Head group of the Settings tab.

The configuration of each scan head is stored in a file with a filename that corresponds to the serial-number of that particular scan head and with the extension ".hed" (e.g. "10-11-584.hed" for an PHYWE Measure Nano STM scan head). The currently loaded scan head calibration file is displayed in the status bar.

Tip

The specific scan head calibration file(s) for each customer is automatically copied and selected as default during the installation of the control software from the installation CD. It can be found in the "Calibrations" sub directory of your installation path.

IMPORTANT

When you change a scan head, you have to load the correct configuration file too. If you do not, scan ranges and other important calibration settings are incorrect and the scan head may not operate properly.

Load...
Loads a different scan head calibration file.

Save as...
Saves the current scan head calibration file with a different name.

Edit...
Edit the currently loaded scan head calibration file using the Scan Head Calibration Editor dialog (see *Scan Head Calibration Editor dialog* below. Always save a backup copy of the original scan head calibration files by clicking 'Save As...' first.

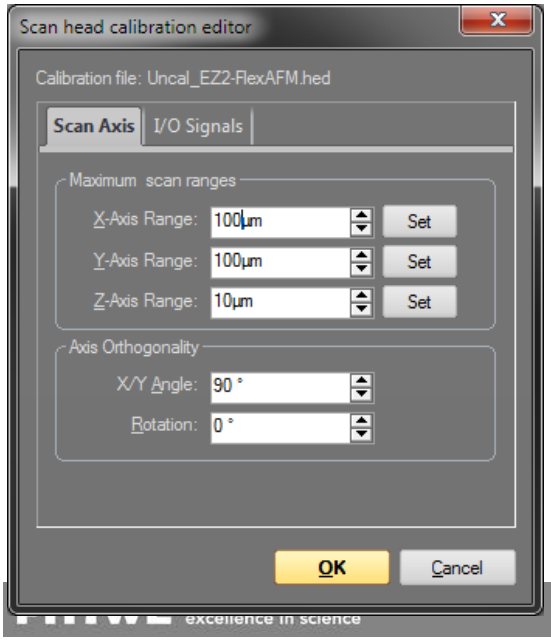
Scan Head Calibration Editor dialog

Through this dialog, the calibration of all standard Inputs and Outputs can be configured individually for a particular Scan Head

CAUTION!

Changes to these settings should be performed with great care. False settings can lead to false interpretation of the data and incorrect operation of the controller.

Scan axis



Maximum scan ranges

X/Y/Z-Axis Range
The calibration values of each of the scanner axes. The calibration values are given as the maximum motion range of the scanner (Overscan is set to 0% and X/Y Angle set to 90° and the Axis Orthogonality Rotation of 0° [or a multiply of 90°]).

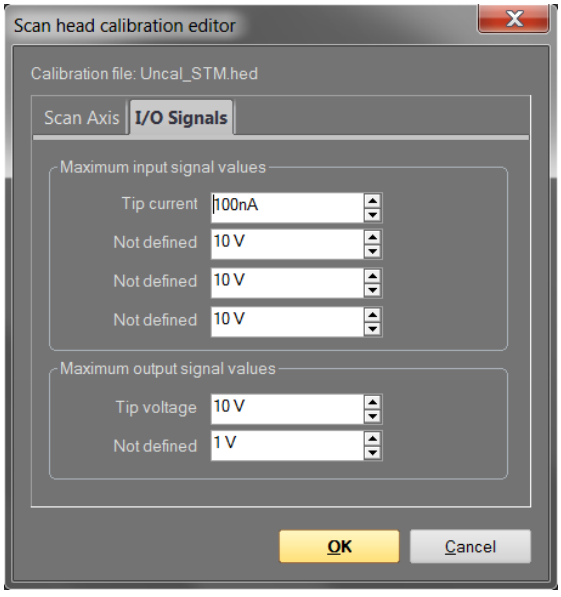
Set
The “Set” buttons open the Scan Axis Correction dialog (see next section).

Axis Orthogonality
The X- and Y-Axes of the scanner are generally not perfectly orthogonal, and their orientation with respect to the AFM housing may vary. The controller corrects these errors by adding/subtracting some of the X scanner command signal to the Y scanner command signal and vice versa.

X/Y Angle
The angle between then the X- and Y-axis of the scanner hardware. The control software uses this value to correct the scan command signals such that the scan axes are orthogonal.

Rotation
The angle between the X-axis of the scanner and the X-axis of the microscope body (see *Figure 4-2: Coordinate systems* (page 21)). The control software uses this value to correct the scan command signals in such a way that the scan axis is parallel to the X-axis of the microscope body.

I/O Signals



Maximum input signal values

Tip current
Limits the maximum current between tip and sample.

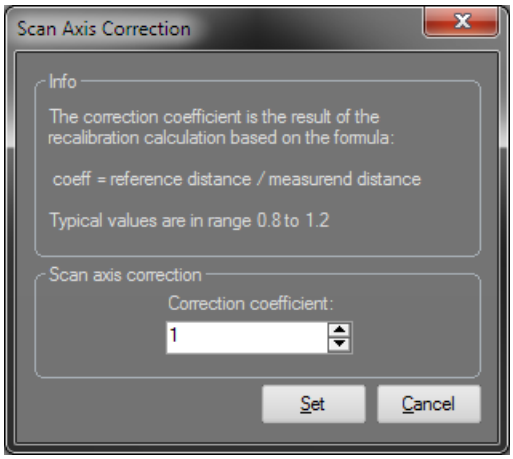
Maximum output signal values

Tip voltage
Gives the maximum voltage which can be applied between tip and sample.

IMPORTANT

Adjusting these values too high can damage the device. It is recommended to not change these values!

Scan Axis Correction dialog



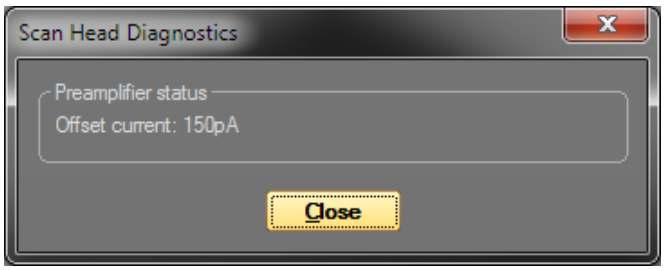
This dialog can be used to correct the scan range by entering a correction factor based on a measured distance and a known real distance.
This correction factor could for example be determined by evaluating the height information in a measurement of a calibration grid with known properties.
Correction coefficient The scan range is multiplied with this number when the “Set” button is clicked.

Scan Head Diagnosis dialog

The Scan Head Diagnosis dialog displays the current status of the scan head. It is opened by clicking the “Diagnosis” button in Scan Head group of the Settings tab.

Tip

The Scan Head Diagnostics dialog cannot be accessed once the tip has been approached to the sample. In this case, retract the tip first.



Information about the preamplifier that is present in the STM scan head is displayed here. The offset current is a leakage current that is measured by the preamplifier when it is not in contact with the sample.

Note

The offset current is not only a measured value, but is also used as a compensating value during measurement. Therefore, even high values are not problematic for measuring at low tunneling current.

Controller Configuration dialog

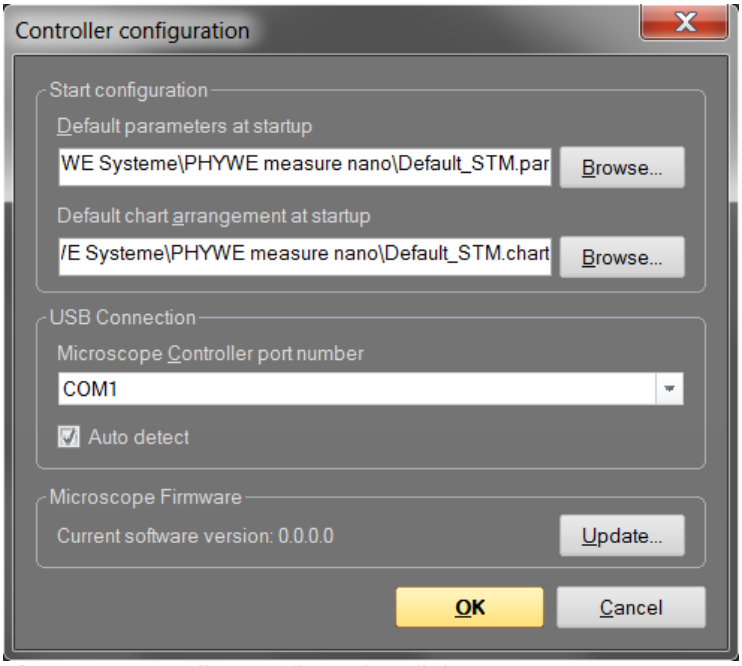


Fig. 11-1 Controller Configuration dialog

With this dialog some controller hardware related settings can be configured. On a correctly installed system, it should not be necessary to change these settings manually. The Controller configuration dialog is opened via Controller" button in the Hardware group of the Settings tab.

Start configuration
The parameter and chart arrangement files that are loaded when the SPM Control Software starts. Each Windows user has his/her own set of these two files a personal "Local Settings" directory. Therefore, each windows user can configure the control software to his/her own personal preferences without any consequences for other users.

USB Connection
The SPM controller uses a virtual serial port that is connected to the USB port. The number of this virtual serial port should be the same as the one shown in your the windows device manager dialog. Activate "Auto detect" to let the control software search for the right COM port at each program start. This is highly recommended, because Windows assigns individual COM port numbers to different USB connectors. With auto detect, you will be able to plug in the USB cable to different ports.

Tip

If the port number is set to "No Controller (Simulation only)" and "Auto Detect" is switched off, the control software will always start in Simulation mode. This could be useful if the software will be used mainly for analysis and is installed on a PC without microscope hardware.

Microscope Firmware
Here the currently used firmware version of the controller is shown. In case of a standard software update the manual update of new firmware is normally not necessary since it is performed automatically at the start of the updated PHYWE Measure Nano Software. Automatic firmware update is always performed each time a different (older or newer!) software version is started since last time. Click the "Update" button to install individual firmware updates you received from PHYWE support.

11.5 SPM Parameter Dialog

This SPM Parameter dialog contains many advanced parameters for measuring. It can stay open during all operations to provide the advanced user with a permanent and detailed control over all measurement parameters. The dialog is organized in several (sub-)pages:

- *Imaging*
- *Spectroscopy*
- *Operating Mode*
- *Approach*
- *Z-Controller*

These pages are described in more detail in the next sections.

11.5.1: Imaging

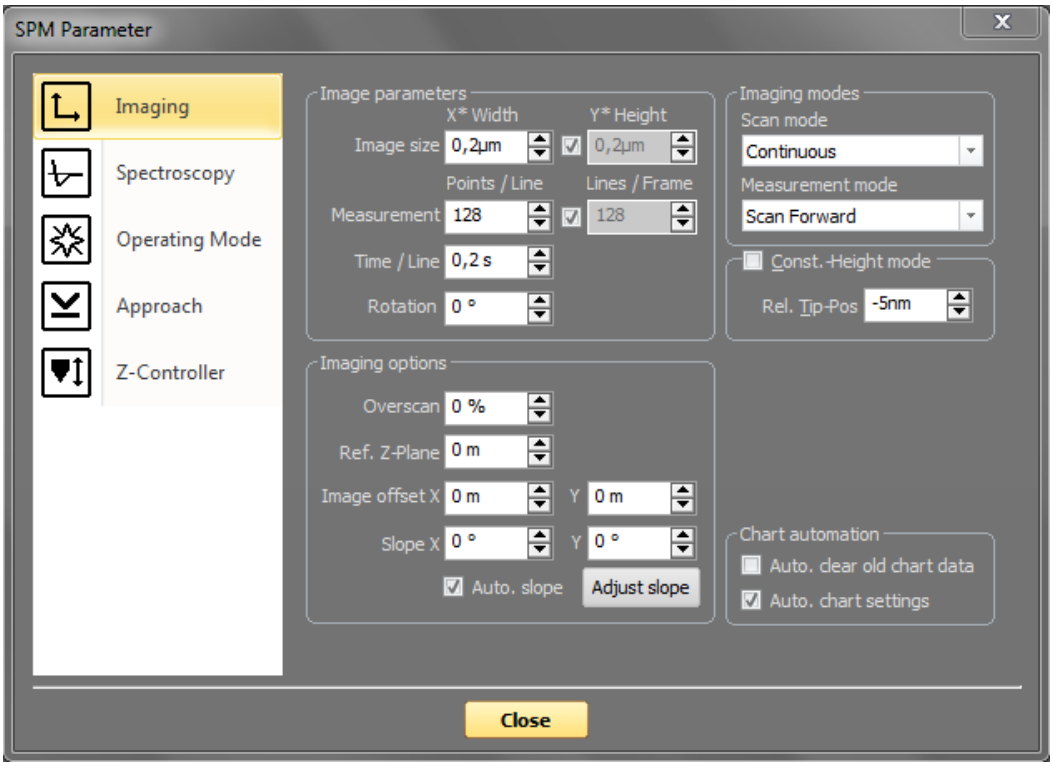


Image parameter

Image size
The image size in X*-direction and the image size in Y*-direction. When the Check-box is active, the image Height is always identical to the Image width.

Measurements
The number of measured data points and data lines in an image. When the Check-box is active, the number of Lines is always equal to the number of Points / Line.

Time / Line
The time needed to acquire a data line. The time needed for the entire image is displayed in the status bar.

Rotation
The angle between the X-direction of the scanner and the X* direction of the measurement (Figure 15-2: Coordinate systems)

Imaging options

Overscan
The “Overscan” determines how much the effective scan range is increased relative to the image width. This will eliminate edge effects caused by the reversal of the scanning motion by not recording or displaying them in the measurement image. Disadvantages of using Overscan are that the maximum scan range is reduced, the tip moves slightly faster over the sample with the same “Time/Line” setting, and the tip may hit large features outside the measured image.

Ref. Z-Plane
The height of the reference plane. This height reference is used when the Z-Controller output is cleared, and when the Z-position is not modulated relative to the current surface position during spectroscopy measurements. The reference plane for the image can be aligned to the surface of the sample using the slope parameters (see *Figure 4-2: Coordinate systems* (page 21).

Image offset X/Y
The center position of the measured area.

Slope X
A positive value rotates the image plane around the Y-axis counterclockwise.

Slope Y
A positive value rotates the image plane around the X-axis counterclockwise. The center position of the measured area can be changed by typing its position as well as by using the Move tool in the Imaging toolbar. The zero position corresponds to the center position of the scanner.

Adjust slope
The “Adjust slope” button will cause the control software to set appropriate values for X- and Y-slope by performing two single line scans (one in X- and one in Y-direction) and determining the respective slopes via line fitting, thus electronically compensating for these measurement plane slopes (see *Adjusting the measurement plane* (page 66) for details).

Auto slope
Automatically performs the same action as the “Adjust slope” button does. It adjusts the slopes with each new “Start” of imaging.

Imaging modes

Scan mode
This parameter defines how the images are acquired and displayed:
– **Continuous** The acquisition direction is reversed after each scan: from bottom to top and vice versa.
– **Cont. Up** The acquisition direction is always from bottom to top.
– **Cont. Down** The acquisition direction is always from top to bottom.

Measurement mode
This parameter defines how each imaging line is acquired and stored:
– **Forward** During forward scan only (left to right in the image).
– **Backward** During backward scan only (right to left in the image).

– Forw.&Backw. During both forward and backward scan.

Const. Height mode

When the Constant Height imaging mode is enabled, the Z-Controller is turned off during the scan (as a consequence, the Probe Status light will blink green). Instead, the scanner scans along a straight line, that should be parallel to the surface. The slope of the line is defined by the X- and Y-Slope parameters. The height of the line is determined at the start of each scan line: First the Z-Controller is turned on. Once the tip position is stable, the Z-Controller is turned off and the tip is moved away from the sample by the distance set by the parameter Rel.Tip-Pos.

Rel. Tip-Pos

This parameter defines the distance by which the Tip is moved towards the sample from the position that corresponds to the Setpoint. A negative setting will move the tip away from the sample.

Chart automation

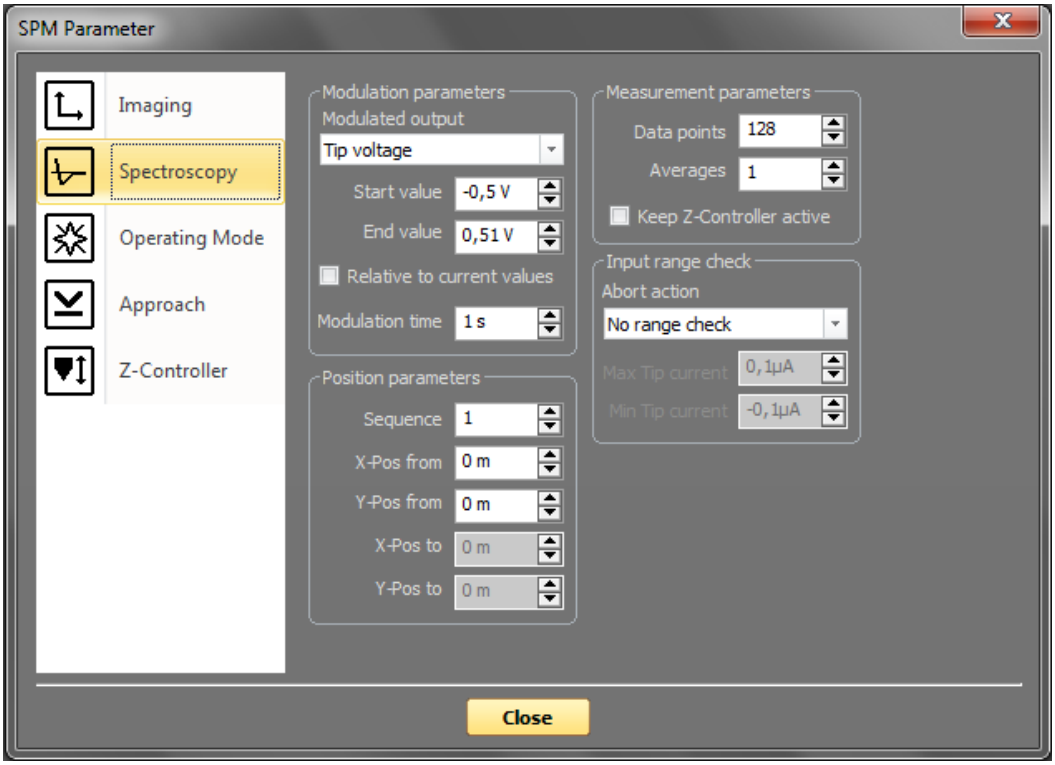
Auto. clear old chart data

Automatically clear the chart data from a measurement when a measurement is restarted (either when a scan is restarted manually, or when a previous scan has finished and measurement recommences as determined by the scan mode (see *Section 11.6: Operating modes* (page 106).

Auto chart settings

Automatically adjusts the Z-Range of all Charts to encompass the measurement data. The Z-Range will also be automatically updated during measurements.

11.5.2: Spectroscopy



Modulation parameter

Modulated output
This parameter defines the signal used to drive the spectroscopy (X-Axis). All possible signals are recorded while this modulation output signal is changing its value from “Start value” to “End value” (Y-Axis). The number of available modulated outputs depends on the scan head and the number of installed controller modules. Possible values are: “Z-Axis”, “Tip Potential” and the names of the User Outputs.

Start value / End value
The range over which the Modulated output is changed. The “Spec Forward” data is measured from the Start to the End value, the “Spec backward” data is measured in the opposite direction. The “Spec forward” data is always measured before the “Spec backward” data. For spectroscopy as a function of distance (Z-axis modulation), more negative values are further away from the sample whereas more positive values go towards (or even into) the sample.

Relative to current value
When checked, the Start and the End values are added to the value the modulated output had before starting the modulation:

- When the Tip Potential is modulated, the current value is the Tip voltage set in the Z-Controller panel.
- When the Z-Axis is modulated, the current value is the sample surface height, as measured using the Z-Controller output. Otherwise, the measurement Z-position is given by the value of the Ref. Z-Plane in the Imaging Panel.

Modulation time
The time used to change the Modulated output from the Start to the End value.

Position Parameters

Sequence
The number of Spectroscopy Measurements to be made in the sequence. If Sequence points is set to one (1), a single point spectroscopy is performed. If Sequence value is two or more, a line spectroscopy is performed. The spectroscopic measurements positions that are equally distributed over the line defined by “X/Y-Pos from” and “X/Y-Pos to”.

X-Pos from / Y-Pos from
The XY-coordinates of the measured point in a spectroscopy measurement. Sets the XY-coordinates of the starting point of the line in a spectroscopy measurement sequence.

X-Pos to / Y-Pos to
The XY-coordinates of the end-point of the line in a spectroscopy measurement sequence. The from and to coordinates are more conveniently chosen using the point and line tools in the Spectroscopy toolbar.

Measurement parameter

Data points
The number of data points measured while the Modulation output changes from Start to End value. The data points are equally distributed over the modulation range.

Averages
The number of times the modulation is repeated to obtain an averaged spectroscopic measurement. The measurement results of aborted modulations are discarded during averaging.

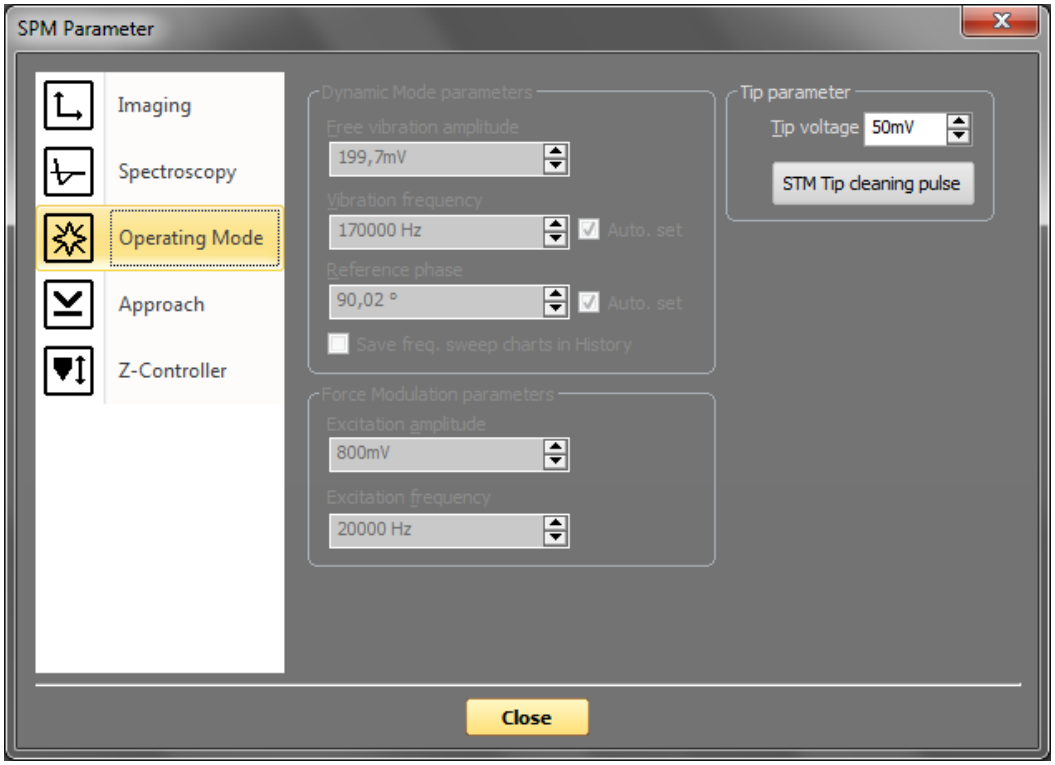
Keep Z-Controller active
When checked, the Z-Controller will continue to change the Z-position to keep the tip–sample interaction constant. This option is not available when the Modulated output is set to the Z-Axis. This setting can for example be used to measure tip current as a function of applied voltage while keeping the tip–sample force constant.

Input range check
In order to prevent tip damage due to too high tip–sample interaction, the settings below “Input range check” define a safe range of tip–sample interaction. When the interaction signal (Deflection in static modes, Amplitude in dynamic modes, Current in STM mode) leaves this safety range, the measurement is aborted. When a spectroscopy measurement has been aborted, a warning dialog is displayed. The number of aborts that occurred in a measurement is reported in the Data Info panel (see *Section 4.3.1: Data Info panel* (page 28)) as: “ModAborted = <number of aborts>”.

Abort action
Action to be performed when the measurement is aborted. Possible options are:

- **No range check** will never abort the measurement. The tip is not protected against damage due to too high tip-sample interaction. This is the default setting.
- **Abort modulation** Aborts the current modulation and continues with the next until the number of modulations in “Averages” is reached.
- **Abort measurement** Aborts the spectroscopy measurement for the current point and continues with the next point of the line, if a line spectroscopy is being performed.
- **Abort sequence** Aborts the entire spectroscopy measurement sequence (cancels all “Averages” and points).

Max / Min input value
The Minimum/maximum value that the feedback signal is allowed to have.



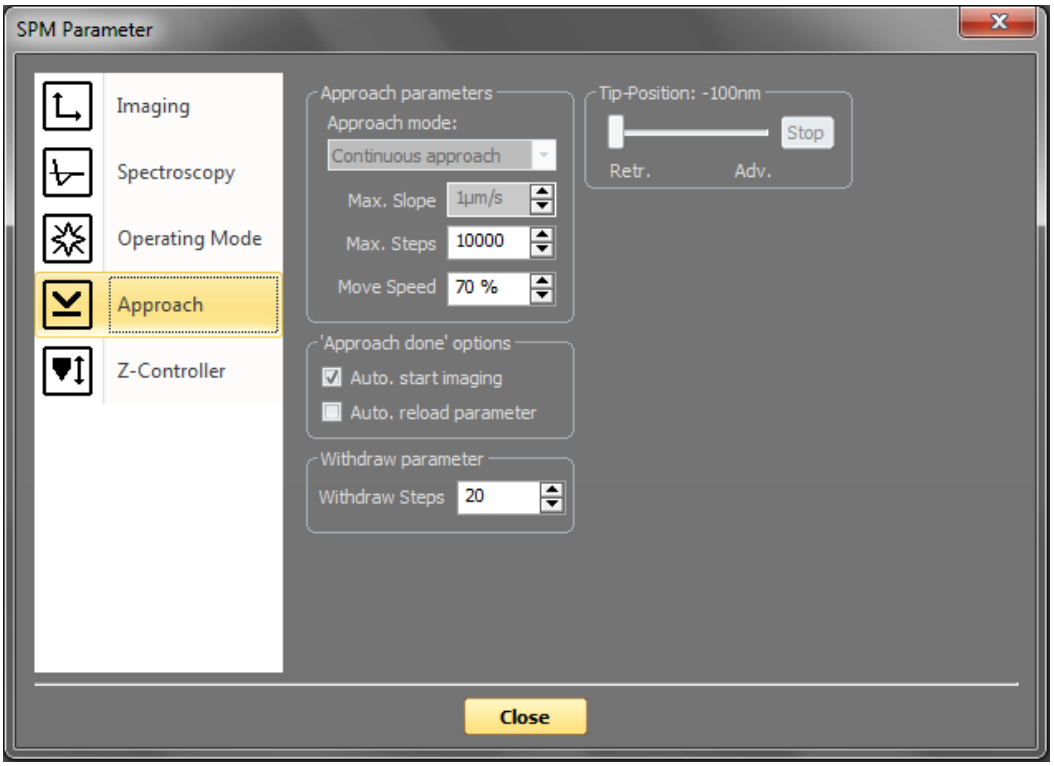
11.5.3: Operating mode

Here, for STM measurements only the tip parameter section is available.

Tip Voltage
This parameter defines the potential to be applied to the tip. The voltage that can be used lies between -10V and +10V.

Info:
With the STM scan head the sample is automatically connected to the ground of the instrument. With AFM scan heads the sample has to be electrically connected to the instrument chassis ground for accurate measurements.

STM Tip cleaning pulse
A short voltage pulse applied to the STM tip to remove material picked up by the tip during measurements. The voltage pulse is approximately 5 V in height and 100 ms in duration. It can be used to clean a dirty STM tip.



Approach parameter

Approach Mode
For STM scan heads no selection is possible (approach is always performed step-by-step through the stick-slip motion of the STM approach stage). For AFM scan heads, two approach options are available for AFM:

– Continuous approach
Approach with continuous slow motorized stage movement until surface contact point is reached. Z-Axis stays at Tip-Position during approach. This is the default approach method.

– Step-By-Step approach
Approach is performed by moving the motorized stage quickly over a distance that is less than the scanner's Z-range. During the movement of the motorized stage, the tip is fully retracted. When the motorized movement is finished, the scanner extends the tip along the Z-axis to probe for the sample surface. The approach is considered done when the Setpoint (defined in the Z-Controller page of the SPM Parameter dialog, or in the Z-Controller section of the Imaging panel of the Imaging window) has been reached. If it is not reached within the Z-range of the scanner, the tip is again fully retracted and the next motorized step is performed. This process of 'step-and-probe' is repeated until the Setpoint has been reached (and approach is done). This approach method is considered to be more 'gentle' to tip and sample and should be considered for very sharp tips and/or very soft samples. In general, it does however take more time than Continuous approach.

Max. Slope
This parameter defines the speed of extending the z-axis. This parameter is only available in Step-By-Step approach mode. Slower speeds help to preserve sharp tips.

Max. Steps
This parameter defines the maximal duration of an automatic approach:

- In Continuous approach mode it defines the maximum time.
- In Step-By-Step approach mode it defines the maximum number of cycles.

Move Speed
This parameter defines the move speed during automatic approach and withdraw:

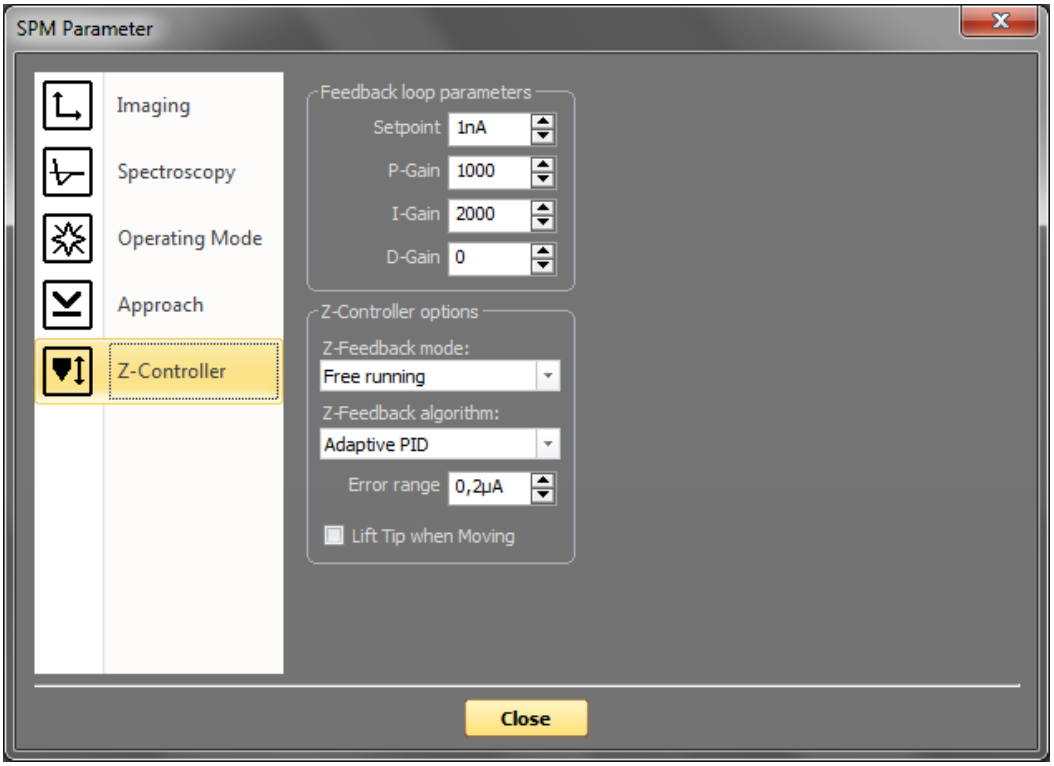
- In Continuous Approach mode this value should be small. If the approach is too fast, the tip or the sample surface can be damaged. On the other hand, the motor will not move when the move speed is too small.
- In Step-By-Step Approach mode should be around full speed. Lower values help to stop in the z-range of the scanner. On the other hand the approach time increases.

'Approach done' options

Auto. Start imaging
When selected, the system automatically starts imaging after a successful approach. Scanning automatically stops the approach motor is moved.

Auto. Reload parameter
When selected, the control software reloads the default startup parameter file for each approach.

Tip-Position
This value determines the Z-Position of the scanner when the approach motor stops. When the Tip-Position is changed when the tip is already approached to the sample, the motor will move the approach stage so, that the Z-Position of the tip becomes equal to the set Z-Position. When a high resolution scanner is used, the Tip-Position before approach is set to approximately +500 nm (advanced) by default. This compensates for the residual motion of the approach stage that occurs after the approach motor has stopped.



Feedback loop parameter

Setpoint
The working point for the Z-Controller. Depending on the operating mode, this is the tunneling current (STM mode), cantilever deflection (Static Force mode) or relative cantilever vibration amplitude (Dynamic Force mode). In the latter case, the set amplitude is relative to the operating amplitude, set in the Mode Properties Section. For example, when a Setpoint of 70% is used, the Z-Controller will move the tip closer to the sample until the vibration amplitude has decreased to 70% of the vibration amplitude far away from the sample.

P-Gain
The strength of the Z-Controller reaction that is proportional to the error signal. Increasing the P-Gain decreases the error signal.

I-Gain
The strength of the Z-Controller reaction that is proportional to the integral of the error signal. Increasing the I-Gain decreases the error signal over time. It is the least sensitive to noise, and usually the dominant contributor to the topography measurement.

D-Gain
The strength of the Z-Controller reaction that is proportional to the derivative of the error signal. Increasing the D-Gain decreases fast changes in the error signal, but also amplifies high frequency noise.

Z-Controller options
Z-Feedback Mode
The following modes are available:

- **Free Running** The Z-Controller is active.
- **Freeze Position** The Z-Controller is not active, the scanner remains in its current Z-position.

- **Stop and Clear** The Z-Controller is not active, the scanner is moved to the “Ref. Z Plane”, set in the Imaging page of the SPM Parameter dialog. The Probe Status Light will blink green as long as the Z-Controller is deactivated.

Z-Feedback algorithm
The following algorithms are available:
- **Standard PID** A standard PID controller is used for Z-Feedback.
- **Adaptive PID** A standard PID controller is used for Z-feedback. In addition, the bandwidth of the Topography measurement is adapted to the number of measured points per second. The adaptive PID controller thus reduces noise in the measurement. However, topography changes that happen faster than the time between two measured points are also lost. This makes it more difficult to detect vibrations due to instability of the feedback loop. These vibrations remain visible in the Current, Amplitude, or Deflection signal, however, so always monitor these signals when optimizing Z-Controller settings, especially when using the Adaptive PID.

Error Range
The range of the error signal used to control the Z-Position. The error signal is the difference between the signal used for topography feedback and the current Setpoint. When the value of “Error Range” is reduced, the resolution of the error signal is increased.

11.6 Operating modes

With an STM, the sample surface can be scanned in two different ways: by using *Constant Current mode* or by using *Constant Height mode*. Read the following sections for details.

Constant Current mode
In Constant Current mode, the tunneling current is kept constant by the Z-Controller. The output of the Z-Controller thus corresponds to the height of the sample surface. This output is recorded as a function of X and Y position, and is displayed as the Topography signal.

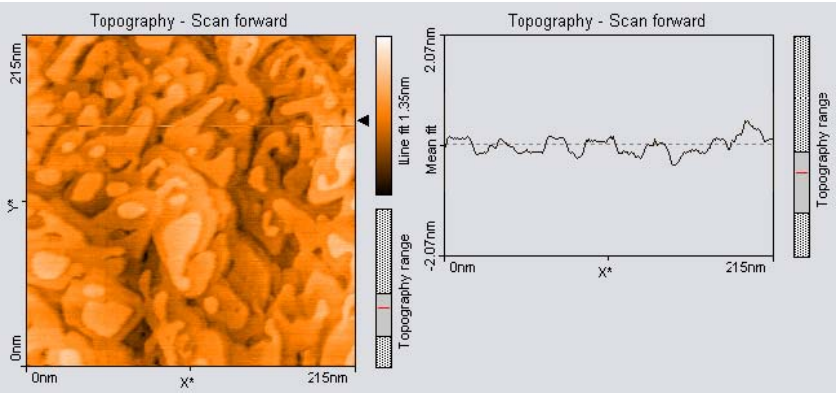
Const.-Height mode
In Constant Height mode on the other hand, the tip does not follow a surface of constant tip current. Instead the variation of the tunneling current is directly recorded as a function of the X and Y position in plane parallel to the sample surface. The PHYWE STM is normally configured to measure in the Constant current mode. To switch to the Constant height mode, you could theoretically just turn off the Z-Controller. However, several problems arise:
- The tip would crash into the slightest unevenness of the surface.
- The scan plane of the scanner must be very well adjusted to the plane of the sample.
- The thermal drift in the Z-Direction will cause the tip to rapidly move away from the sample, or even worse, to crash into it.

For a large part, these problems can be avoided by setting the Loop gain to a very low value. Thus, the feedback loop can follow the slow movement of the sample caused by thermal drift and the sample plane, but not the fast height changes due to the presence of the atoms.
To measure in Constant height mode:
1. Find a flat area of the sample by imaging it in Constant current mode, and zoom in on this area.
2. In the Z-Controller section of the Imaging tab, set I-Gain to 4.
3. Set P-Gain to 0.

To visualize the current:
> In the Color map chart that is displayed in the Imaging Window, set the Input channel to “Tip Current”. The bar next to Color map should now display the text “Tip Current” and should have the units “pA” or “nA” instead of “nm”.

11.7 Measuring Gold

Now that you are familiar with measuring the Graphite (HOPG) sample and using the PHYWE Measure Nano software you can proceed with the next challenge. It is way more difficult to obtain good images of gold. Atomic structures are difficult to observe, because the electrons on the surface are much more homogeneously distributed than in graphite. But, with some training, the mono-atomic gold steps can be observed.



Since the gold sample cannot be cleaned by simple means, it is possible that over time contaminants may prevent you from obtaining good results. If you have problems measuring the gold sample because of this issue, please order a replacement from your local PHYWE distributor (or online on the www.phywe.com homepage).

Tip

Before performing any experiments with the gold sample it is necessary to practice on the graphite sample. The graphite sample is also a good test sample to judge the quality of the installed STM tip.

To perform measurements on gold proceed as describe for the graphite sample, but with the following changes to the settings:

- Set the Tip voltage in the Z-Controller panel to 0.5 V.
- Set the Time/Line in the Imaging Panel to 0.3 s.

If you do not get stably reproduced scan lines, try to re-approach:

- Press Withdraw followed by Approach.

If that does not help to improve the image quality, retract the sample holder, rotate it a little by hand and repeat the approach.

If the image reproduces stably:

Select an Image size between 200 and 300 nm, and evaluate your measurements in the same way as you did with the graphite images.

CHAPTER 12:

Spectroscopy Mode

In a spectroscopic measurement, the input channels are measured as a function of a modulated parameter. This modulated parameter can be the Z-distance to the sample or the tip voltage. The measured parameter is the tunneling current.

The accuracy of the spectroscopic measurements can be increased by averaging the measurement results of several consecutive modulations. A spectroscopic measurement sequence consists of a number of spectroscopic measurements of the same type, measured along a user defined line in the XY-plane. A point measurement is made if the number of points is one.

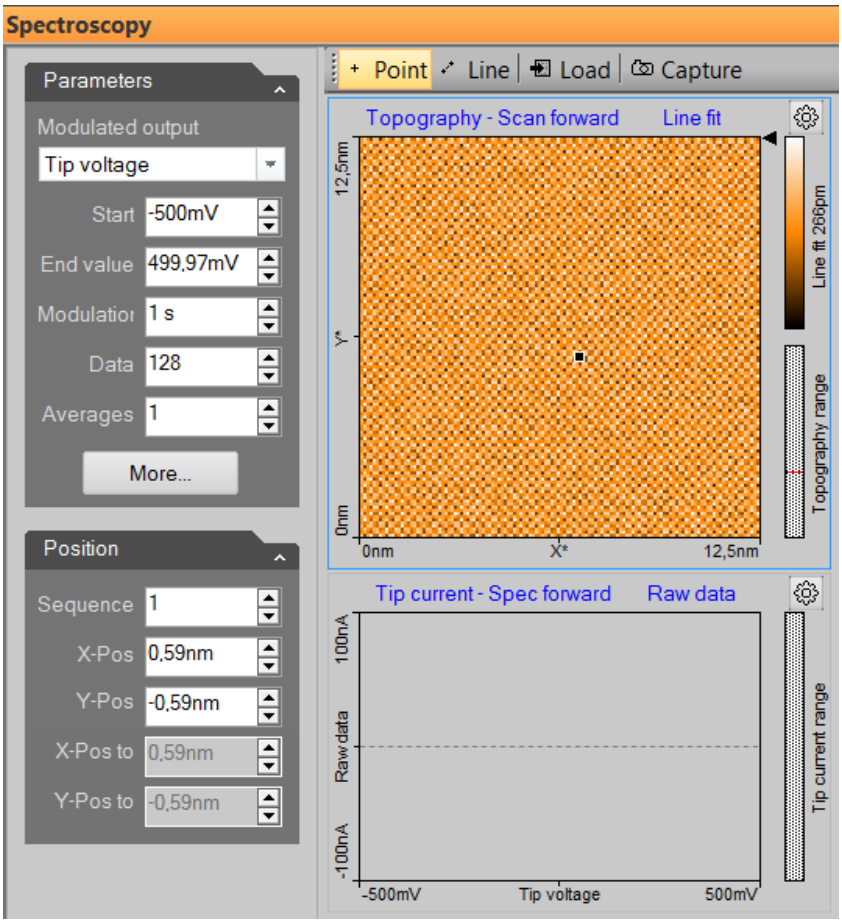


Fig. 12-1 Interface of the Spectroscopy window.

The measurement sequence is carried out as follows:

1. Move the tip to the start of the line with active Z-control.
2. Switch off the Z-Controller.
3. Record a spectroscopic measurement.
4. Turn on the Z-Controller again.
5. Move the tip to the next point on the line in the XY-plane. Steps 2–5 are repeated for all points on the line.

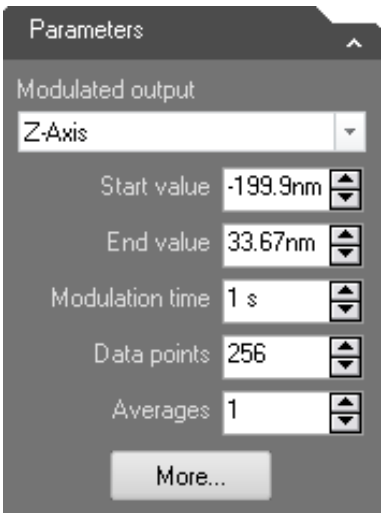
The Spectroscopy window contains the Spectroscopy toolbar, with commands that control the spectroscopy processes, and the Spectroscopy panel (parameter area), with parameters that determine how the spectroscopy measurement is performed.

The Spectroscopy tab also contains a number of charts that display the data from a previous imaging measurement and the data from the ongoing spectroscopic measurement. The Spectroscopy tab can display as many charts as the size of the window can accommodate. It is recommended to display at

least two charts, one Color map of a previous Topography measurement of the area where the spectroscopy measurement is performed, and one Line graph of the current spectroscopy measurement. For more information on adding and changing charts see *Section 4.3.2: Charts* (page 28).

12.1 Spectroscopy panel

Parameters section



Modulated output
This parameter defines the signal used to drive the spectroscopy (X-Axis). All possible signals are recorded while this modulation output signal is changing its value from “Start value” to “End value” (Y-Axis). The number of available modulated outputs depends on the scan head and the number of installed modules. Possible values are: “Z-Axis”, “Tip Potential” and the names of the User Outputs.

Start value / End value
The range over which the Modulated output is changed. The “Spec Forward” data is measured from the Start to the End value, the “Spec backward” data is measured in the opposite direction. The “Spec forward” data is always measured before the “Spec backward” data. For spectroscopy as a function of distance (Z-axis modulation), more negative values are further away from the sample whereas more positive values go towards (or even into) the sample.

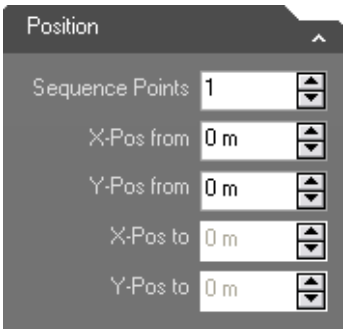
Modulation time
The time used to change the Modulated output from the Start to the End value.

Data points
The number of data points measured while the Modulation output changes from Start to End value. The data points are equally distributed over the modulation range.

Averages
The number of times the modulation is repeated to obtain an averaged spectroscopic measurement. The measurement results of aborted modulations are discarded during averaging.

“More” button
Opens up the SPM Parameter dialog on the “Spectroscopy” page for more advanced parameter settings (see *Section 11.5.2: Spectroscopy* (page 100)).

Position section



The Position parameters can be used to define a sequence of spectroscopy measurements on positions that are evenly distributed on a line.

Sequence Points
The number of Spectroscopy measurements to be performed in the sequence. If Sequence points is set to one (1), a single point spectroscopy is performed. If Sequence value is two or more, a line spectroscopy is performed. The spectroscopic measurements positions that are equally distributed over the line defined by “X/Y-Pos from” and “X/Y-Pos to”.

X-Pos from / Y-Pos from
The XY-coordinates of the measured point in a spectroscopy measurement. Sets the XY-coordinates of the starting point of the line in a spectroscopy measurement sequence.

X-Pos to / Y-Pos to
The XY-coordinates of the end-point of the line in a spectroscopy measurement sequence. The from and to coordinates are more conveniently chosen using the point and line tools in the Spectroscopy toolbar.

12.2 Spectroscopy toolbar

Point
Activates the single point spectroscopy mode. It defines the position of the spectroscopy measurement by mouse. Click in the topography Color Map chart at the position where the spectroscopy measurement should take place. A small black dot appears at this position. The positions coordinate is transferred to the “X/Y-Pos From” parameters in the Position section. The value in “Sequence” defines the number of spectroscopy measurements measured at these point.

Line
Activates the line spectroscopy mode. Defines the start and end position of the spectroscopy measurement by mouse. Click and hold the left mouse button in the topography Color Map chart at the position where the spectroscopy measurement should start. Move the mouse to the end position and release the left mouse button. A line is drawn between these two points. The positions coordinate is transferred to the “X/Y-Pos From” and X/Y-Pos To” parameters in the Position section. The value in “Sequence” defines the number of spectroscopy measurements measured along these line.

Load

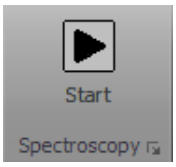
Fills the Topography Color Map chart in the Spectroscopy window with the current measurement of the Imaging window. Selection of “point” or “line” measurement position can be done in this chart.

Capture
A click on “Capture” saves the current spectroscopy measurement data to the History page of the Gallery panel, even when the measurement(s) have not been completed yet. The spectroscopy data are stored as a new document and remain open in the Document space of the SPM software.

12.3 Acquisition tab

During spectroscopy, all groups of the Acquisition tab are identical to those during imaging of the sample, with exception of the Imaging group, which is replaced by the Spectroscopy group.

12.3.1 Spectroscopy group



Start
Clicking “Start” starts a spectroscopy measurement sequence and changes the button to “Stop” until the measurement sequence is finished. Clicking “Stop” aborts the measurement sequence as soon as the current modulation period is finished.

Launcher icon
More advanced settings are available through the “Dialog Launcher” icon (at the bottom right corner of the Spectroscopy group), which opens up the SPM Parameter dialog on the Spectroscopy page (see *Section 11.5.2: Spectroscopy* (page 100).

12.4 Current-Distance-Spectroscopy

In order to perform spectroscopy measurements you have to pin down values of your measurement parameters accurately. If you experience problems in measuring the desired behavior, try to adjust your parameter values. Follow these step by step instructions to run your first spectroscopy measurement. Here you measure the behavior of the current in dependence of the distance to the sample (z).

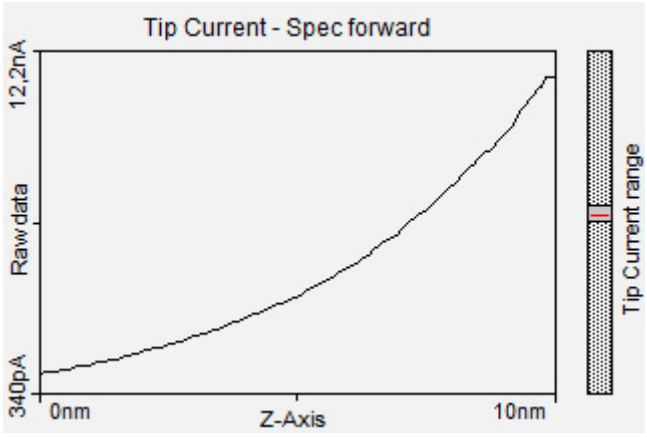
This plan is an example for a current-distance measurement:

1. Approach your sample with standard parameters set.
2. Start a surface scan of your sample in the “Imaging” window.
3. End the running measurement by pressing the “Finish” button.
4. Change to the “Spectroscopy” window and transfer the current image using the “Load” button.
5. Change the “Modulated Output” to “Z-Axis” if it is not selected yet.
6. Set an appropriate value for “Data Points”, for example set it to 128.
7. Now set the “Start Value“ to 0 to start the measurement at the sample surface.
8. Set the “End Value” to a value of your choice, you get good results with values from 5 to 25 nm.
9. Set the “Modulation Time” to 0.15 s (higher values, can smooth the data, lower values are more effected from noise).

10. Set a good value for “Averages”, for example a minimum of 5. Increase the value up to 200 to get a smooth measurement curve. (20 is a good choice and doesn’t take too long to gather the data).
11. For your first measurement you can set the “Input Range Check” to “No abort action” (to exclude false measurements you can set range values later on).
12. Prepare the measurement by clicking on “Point” and putting the cursor on your surface of your scanned image where you want to take the spectroscopy data.
13. Now press the “Start” button.

After a few seconds the end of the measurement is indicated when, the “Stop” button changes back to “Start” and when your data curve doesn’t change anymore. The “Probe Status” should be green during the whole measurement. If the “Probe Status” changes to orange switch to the “Imaging” window approach the sample again and take a new surface scan, afterwards switch back to the “Spectroscopy” window.

With these example values you can get a data curve like this below. You see an exponential dependence of current to distance.



The curve has been taken with the following values:

Star Value:	0 nm
End Value:	10 nm
Modulation Time:	0.15 s
Data Points:	128
Averages:	128
Sequence:	1, denoting it is a point-measurement.

12.5 Current-Voltage-Spectroscopy

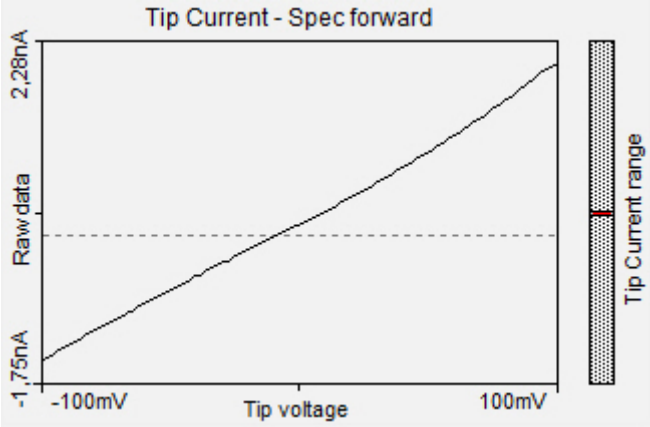
To measure the behavior of the tunneling current in dependence of the applied voltage follow this step by step introduction. If you experience problems in measuring the desired behavior, try to adjust your parameter values.

This plan is an example for a current-voltage measurement:

1. Approach your sample with standard parameters set.
2. Start a surface scan of your sample in the “Imaging” window.

3. End the running measurement by pressing the “Finish” button.
4. Change to the “Spectroscopy” window and transfer the current image using the “Load” button.
5. Change the “Modulated Output” to “Tip Voltage” if it is not selected yet.
6. Set an appropriate value for “Data Points”, for example set it to 128 (higher values can smooth the data curve, but data acquisition takes longer).
7. Now set the “Start Value“ to “-0.1 V”.
8. Set the “End Value” to “+0.1 V”.
9. Set the “Modulation Time” to 0.15 s (higher values, can smooth the data, lower values are more effected from noise).
10. Set a good value for “Averages”, for example a minimum of 5. Increase the value up to 200 to get a smooth measurement curve. (20 is a good choice and doesn’t take too long to gather the data).
11. For your first measurement you can set the “Input Range Check” to “No abort action” (to exclude false measurements you can set range values later on).
12. Prepare the measurement by clicking on “Point” and putting the cursor on your surface of your scanned image where you want to take the spectroscopy data.
13. Now press the “Start” button.

After a few seconds the end of the measurement is indicated when, the “Stop” button changes back to “Start” and when your data curve doesn’t change anymore. The “Probe Status” should be green during the whole measurement. If the “Probe Status” changes to orange switch to the “Imaging” window approach the sample again and take a new surface scan, afterwards switch back to the “Spectroscopy” window.



With these example values you can get a data curve like this below. In the example you see gold showing a linear dependence of current and voltage, following Ohm`s Law (U=R*I).The curve has been taken with the following values:

Star Value:	-0.1 V
End Value:	+0.1 V
Modulation Time:	0.15 s
Data Points:	128
Averages:	80
Sequence:	1, denoting it is a point-measurement.

CHAPTER 13:

Technical Data

13.1 Electronics

Power supply:	100–240 V AC, 50/60 Hz
Computer interface:	USB 2.0
Scan generator:	16 bit D/A converter for all axes
Scan drive signals:	±10 V, no high voltage
Scan speed:	Up to 60 ms/line at 128 data points/line
Measurement channels:	Up to seven measurement channels through 16 bit A/D converter
Scan area and data points:	Individual width/height, up to 2048×2048 points
Scan image rotation:	0–360°
Sample tilt compensation	Hardware X/Y-slope compensation
Spectroscopy modes:	Single point measurement or multiple measurements along a vector
Spectroscopy data points:	Up to 2048
Spectroscopy measurement averaging:	Up to 1024 times

13.2 Software

Simultaneous display of data in charts types:	Line graph, Color map, 3D view, …
User profiles:	Customizable display and parameter settings
Online processing functions:	Mean fit, Polynomial fit, Derived data, …
Quick evaluation functions:	distance, angle, cross section, roughness, …
Data export:	TIFF, PNG, BMP, ASCII, CSV, …

13.3 Computer Requirements

Operating system:	Windows 2000 / XP / Vista
Required hardware:	USB 2.0 connector
Recommended hardware:	Pentium 4/M or AMD Athlon (or better), 256 MB RAM, True color >1024×786 resolution video card, Hardware Open GL accelerator

13.4 The STM Scan Head

Maximum Scan range:	approx. 500 nm x 500 nm
Maximum Z-range:	200 nm
Drive resolution Z:	~ 3 pm
Drive resolution XY:	~ 8 pm
Current Set point:	0.1-100 nA in steps of 25 pA

13.5 Operating Modes

Imaging modes:	Constant Current (Topography), Constant Height (Current)
Spectroscopy modes:	Current-Voltage, Current-Distance
Tip voltage:	±10 V in 5 mV steps
Tip–sample approach:	Stick-slip piezo motor
Sample size:	max. 10 mm diameter

CHAPTER 14:

FAQ

CHAPTER 15:

Experiments

In the following some experiments are shown which can be done using the PHYWE Scanning Tunnel Microscope. These experiments are building onto the basic shown in the previous chapters of this manual. Therefore, it is recommended to read these chapters first. Furthermore, you should be familiar with the preparation and scanning procedures e.g. from measuring graphite (chapter 6) before advancing to the below experiments. Some of the experiments will give fast results but often you will have to be patient to get good results. The given collection summarizes the content of detailed described TESS expert experiments available from PHYWE. The detailed descriptions are available at phywe.com (→ www.phywe.com/'article number'). For this please use the corresponding article numbers given below.

15.1 Surface and defect analysis – atomic resolution

References:

- ***P2532000 "Atomic Resolution of the graphite surface by STM"***
- ***P2532500 "Investigate in surface atomic structures and defects of different samples by STM"***

Task: Investigate the topography of HOPG, TaS_2 and MoS_2 with respect to crystal irregularities and analyze their atomic structure.

We will start over with the measurements on HOPG. This is the easiest sample to handle. To activate the full measurement-range of your device click the Full button in the imaging window. When you are lucky you find a terrace, which is a line defect on the sample surface, in your first measurement. If that is not the case you can either use the Move tool in the imaging window to start the measurement at a different location on the surface or retract the sample from the tip and carefully turn the sample holder with the black plastic handle. Afterwards approach the sample again. For good results image sizes of about 0.2 µm are recommended.

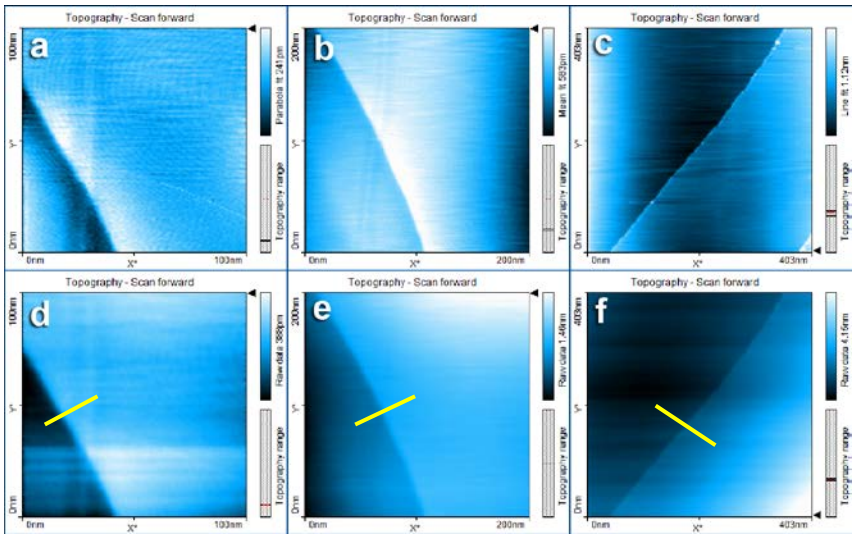


Fig. 15.1-1 Original images of different atomic terraces (a, b, c) and their processed images (d, e, f). Yellow bars indicating the place of the cross sectioning (see below)

In figure 15.1-1 you can find examples of terraces at different spots on the HOPG sample surface. Make sure you adjust the parameters of the feedback loop to achieve good image quality. Too high values in the P-and I-gain will be noticeable in the line graph as very high peaks and a very rough line structure. Adjust the gain values to smooth the line graph and your image respectively. When getting a good scan apply some filters to reduce noise, adjust scan line levels and remove a background distortion.
The recommended procedure would be:

- Apply glitch filter.
- Apply noise filter.
- Do a background substraction and/or correct scan line levels.

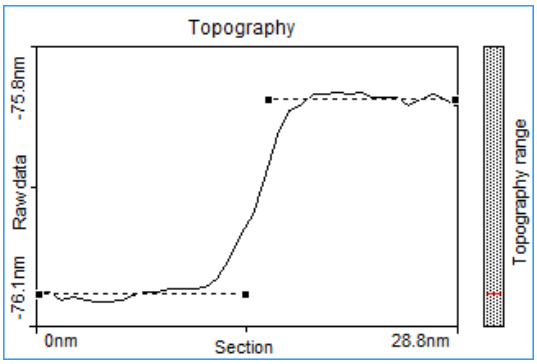


Fig. 15.1-2 Cross section of image d (see above). Distance: Δz=332.2 pm.

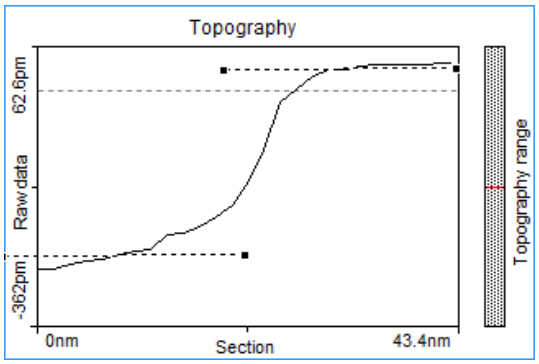


Fig. 15.1-3 Cross section of image e (see above). Distance: Δz=333.9 pm.

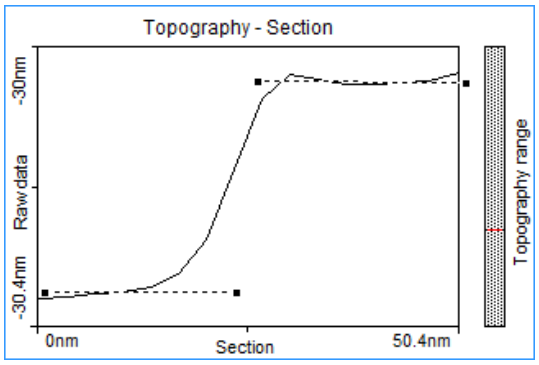


Fig. 15.1-4 Cross section of image f (see above). Distance: Δz=686.1 pm.

If you are not sure that the corrected image is the better one, go a step backwards and start over again. Your corrected image should be almost free of strong gradients and one plane of the sample surface should have the same color in every point.

Estimating the step height of terraces

Next, you can start to analyze your data. At first use the "Create Cross section" tool. Place a line on the image you want to analyze. If you want to estimate the step height of terraces you should make sure the line is perpendicular to the edge of the terrace. Click "Cut out line". This will create a new graph image with the Z-information corresponding to the cross section you have drawn (Fig. 15.1-2 to 15.1-4). Avoid creating cross sections from spots with scan or correcting artifacts (strange gradients or other jumps in the coloring of the surface). Now you can use the „Measure Distance" tool. It allows you to draw two parallel lines between which the distance is measured. The advantage of using the line measurement instead of a point measurement is, that it is possible to reduce the influence of the surface roughness or noisy data by placing the lines between the lowest and the highes values of each terrace.
When you do measurements keep in mind that you always have errors influencing your results. Errors arise from the scanning itself (temperature dependence of the piezo electric devices) but even more due to a bad Z-leveling or background correction. Your results should not exceed a relative error of about 5% to literature values (the lower the better). When you have completed some measurements it is recommended to calculate the mean value of your data for each proposed step size.

The mean value of the shown measurements is 3.364 Å which is very close to the theoretical step size 3.35Å which corresponds to a deviation of 0.5%.

Achieving atomic resolution

To achieve atomic resolution zoom into a clean terrace. Therefore

- Click the color map chart to activate it. A blue square is now drawn around the color map chart.
- Click “Zoom” in the upper tool bar, move the mouse cursor to a flat region (similar color) in the color map and click on it. The software will now draw a square that indicates the new scan range. The size of the new scan range is displayed in the “Tool Results” panel.
- Change the size of the new scan range to about 30–50 nm by clicking and dragging a corner of the square with the mouse cursor.
- Double-click the color map when the new scan area is set as you want it (or press “Zoom” in the “Tools Result” panel). The imaging settings are now set in such a manner that the new measurement will correspond to the area that was indicated by the square you have set. Let the topography reproduce stably again.

Because one nanometer is the diameter of between four and eight atoms, atomic arrangements can normally be recognized at an image size of about 10 - 3 nm (Fig. 15.1-5). Therefore: Set the image size in the imaging panel to 3 nm or use the “Zoom” option on your last image.

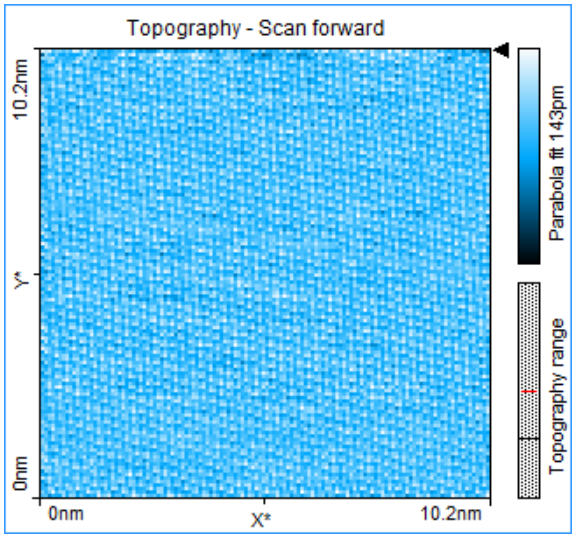


Fig. 15.1-5 Atomic resolution on HOPG

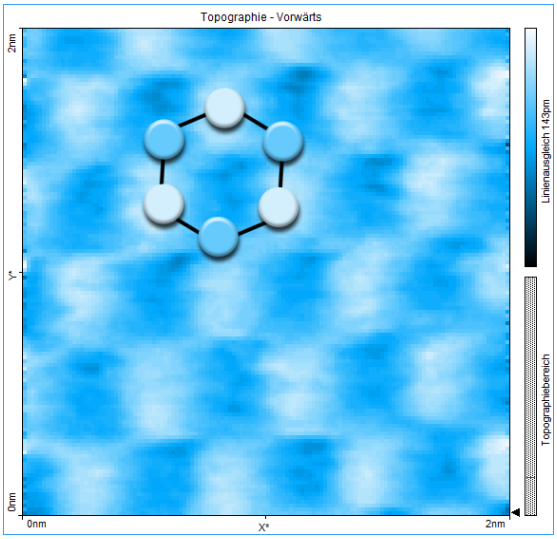


Fig. 15.1-6 Hexagonal pattern reveals electronic and topographic structure of surface atoms

Hexagonal Structures

The lattice structure of graphite is the so called hexagonal-closest-packing (h.c.p) with an “ABA” pattern. The hexagonally arranged carbon atoms in the surface layer which have a neighbor in the layer below appear darker as those without a direct neighbor which appear brighter due to their free bonds. Image 15.1-6 shows the atomic resolution on HOPG. Additionally, one hexagonal carbon ring is marked.

One can measure the lattice period by using the “measure length” tool. The mean lattice parameter comes out at $a \approx 2.193\,\text{Å}$. The theoretical lattice parameter is $a \approx 2.450\,\text{Å}$.

3D representation of data

When you have finished your measurements you also have the possibility to present your data in 3D (Fig. 15.1-7) (see page 35). Select “3D View” in the “Select Chart Type” drop down menu. Then adjust the appearance until you are satisfied with the look:

Always click and hold the left mouse button on the 3D view chart while changing the 3D view. The surface is reduced in feature complexity as long as the left mouse button is pressed.

Press the following additional keys/buttons to determine what chart property is changed:

- Surface rotation mouse left/right
- Surface tilt mouse up/down.
- Size displayed surface “Ctrl”- key + mouse up/down
- Surface position “Shift”-key + mouse up/down/left/right Z-scale magnification left mouse button + right mouse button + mouse up/down
- Light source direction (360°) “Shift”+“Ctrl”-key + mouse left/right
- Light source height (0°–90°) “Shift”+“Ctrl”-key + mouse up/down

The scanning parameters for HOPG were: set point: 1.2 nA, tip voltage: 50 mV, P-gain: 1000-1200, I-gain: 1200-1500.

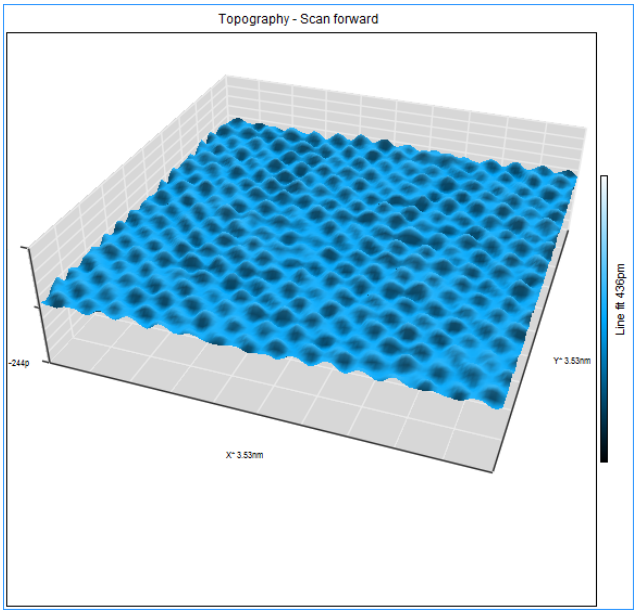


Fig. 15.1-7 3D representation of constant current data on HOPG

Investigation of TaS_2 and MoS_2

The measurements done on HOPG can be transferred to TaS_2 and MoS_2 analogously although it is much more difficult to get good results. To achieve atomic resolution you will need to zoom in step for step into the plainest region of the sample.

Figure 15.1-8 shows the surface structure of TaS_2. The measured step height $11.67\,\text{Å}$ from the cross-section in Fig. 15.1-9 which is across two layers giving a mean layer height of 5.835Å which is good according to the theoretical value of $5.853\,\text{Å}$.

Furthermore, in figure 15.1-10 one can see atomic resolution on TaS_2 and measure the period of the lattice. The mean (vertical) lattice parameter comes out at $a \approx 3.433\,\text{Å}$. This value is pretty close to the theoretical lattice parameter $a \approx 3.346\,\text{Å}$. Furthermore, one sees a super-lattice with period $\sqrt{13}a$ caused by charge density waves (CDW). CDW are an electronic state certain materials

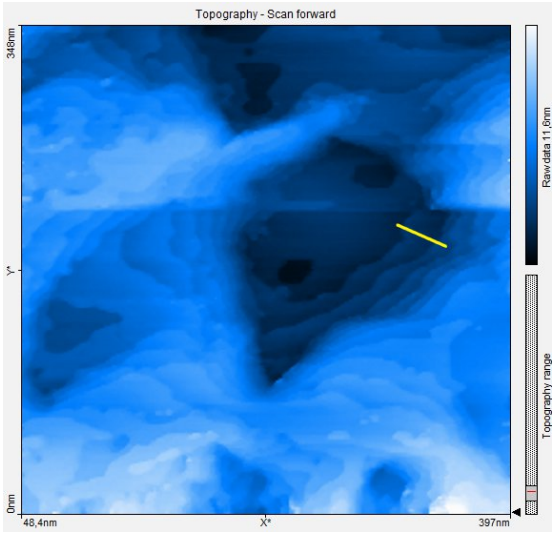


Fig. 15.1-8 Terraces on TaS_2 with indicated cross-section (Fig. 15.1-9)

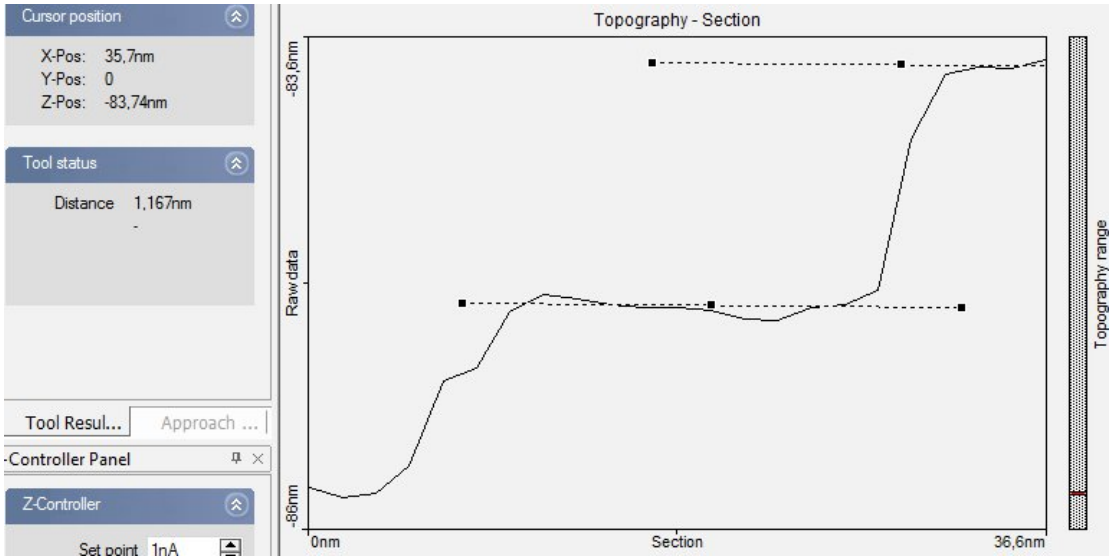


Fig. 15.1-9 Cross-section on TaS_2 from Fig. 15.1.8

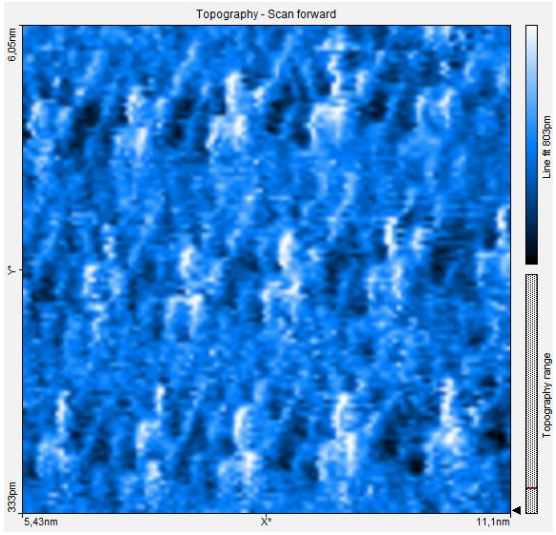


Fig. 15.1-10 Atomic resolution on TaS_2

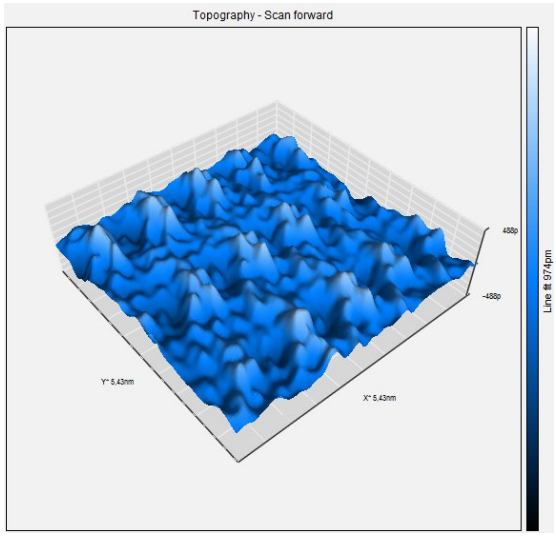


Fig. 15.1-11 3D representation of figure 15.1-10 (TaS_2)

can undergo a phase transition to. The scanning parameters for TaS_2 were: set point: 30 nA, tip voltage 30 mV, P-gain: 0 and I-gain: 60

Figure 15.1-12 shows the surface measurement on MoS_2. Again we create a cross-section to determine the step height (Fig. 15.1-14). The estimated step height for one MoS_2 layer is $0.616\ \text{Å}$. Apparently the measured step in Fig. 15.1-14 consists of 8 layers, resulting in a mean layer height of $0.598\ \text{Å}$.

In Figure 15.1-13 one can identify the atomic structure of MoS_2. The mean lattice parameter comes out at $a \approx 3.78\ \text{Å}$. The theoretical value $a \approx 3.148\ \text{Å}$ differs quite a bit from our result which probably is due to the bad image quality or thermal drift.

The scanning parameters for MoS_2 were: set point 1.5 nA tip voltage 0.4 V, P-gain 1000 and I-gain 2000.

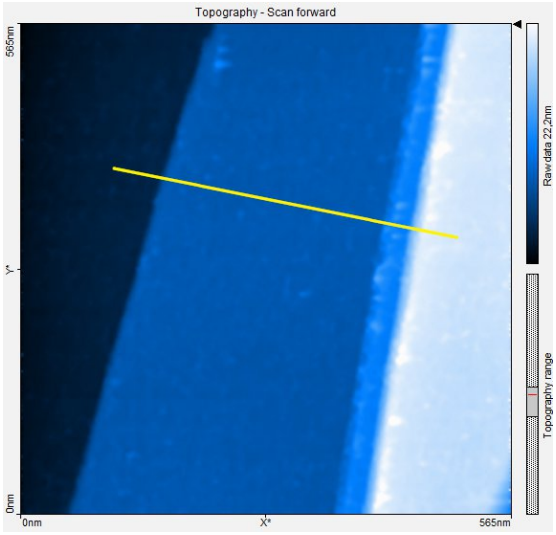


Fig. 15.1-12 Terraces on MoS_2 with indicated cross-section (Fig. 25)

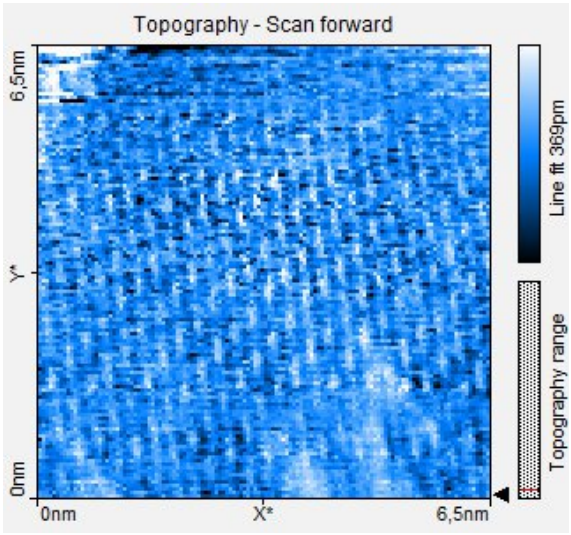


Fig. 15.1-13 Atomic resolution on MoS_2

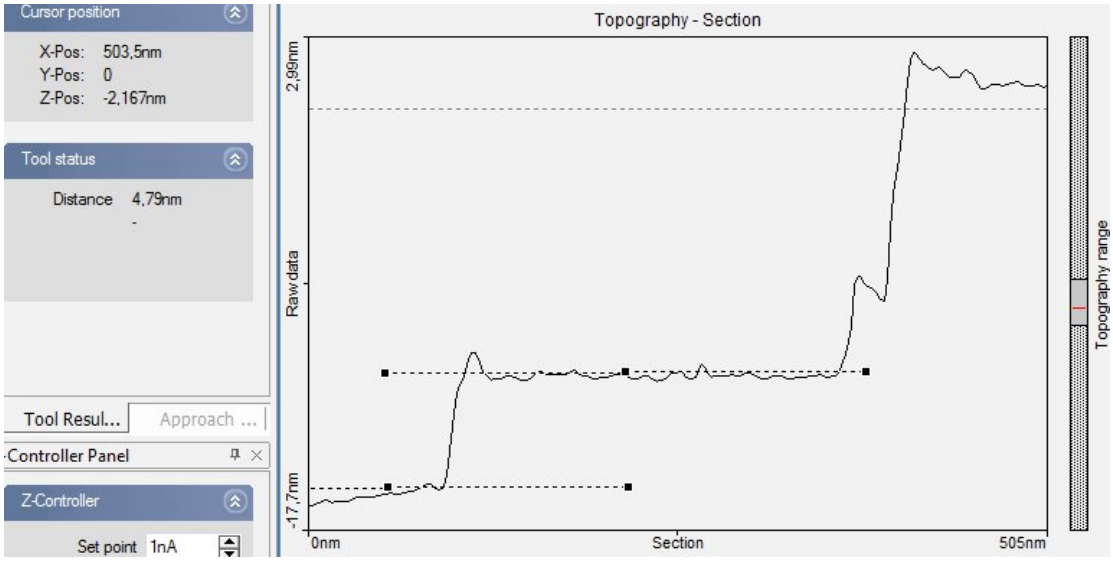


Fig. 15.1-14 Cross-section on MoS_2 from Fig. 19

An interesting observation you might come across when scanning MoS_2 is the replacement of single surface atoms. This results in single smaller or bigger atoms in atomic resolution scans. Such replacements affect the reactivity of the sample greatly and e.g. are used to control the resistivity in MoS_2-nano-tubes.

2D-FFT of atomic resolution

Using a SPM-analysis software such as the open source tool “Gwyddion” one can do a Fourier Transformation of the scanned images, providing information such as periodicity without the need to measure distances of atoms in the images by yourself.

The background for this is the reciprocal lattice. The reciprocal lattice reflects all the properties of the original lattice but is set in the k-space. It can be constructed from the original lattice as shown in Fig. 15.1-15.[1]

Furthermore the Fourier Transformation (FT) of a lattice is just equivalent to an elementary cell of the corresponding reciprocal lattice and therefore one can read all the properties of the original lattice from the FT of a scanned lattice.

The three samples we investigated all are arranged in hexagonal lattices with lattice constant *a*. The Fourier Transformation of a hexagonal lattice is hexagonal itself but with reciprocal lattice constant a^{-1}.

Figures 15.1-17 to 15.1-19 show the atomic resolution scans from above with the corresponding 2D-FFT (Fast FT). From the peaks in these Images one can read the reciprocal lattice by connecting corresponding peaks. The peak height gives the amplitude of the periodic structure.

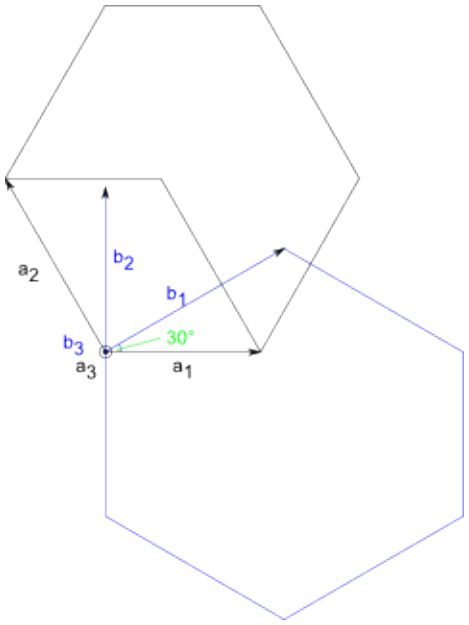


Fig. 15.1-15 Construction of reciprocal lattice. The a_i span the real lattice, the b_i the reciprocal

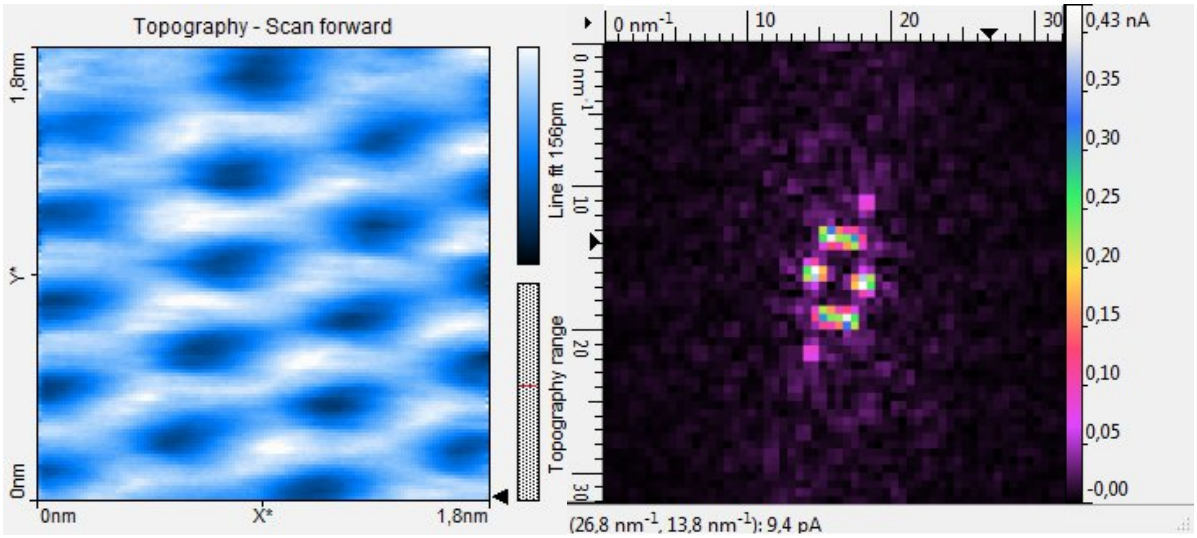


Fig. 15.1-17 Original scan and FFT of HOPG

[1] For the exact construction rules look up an introduction to solid state physics book of your choice

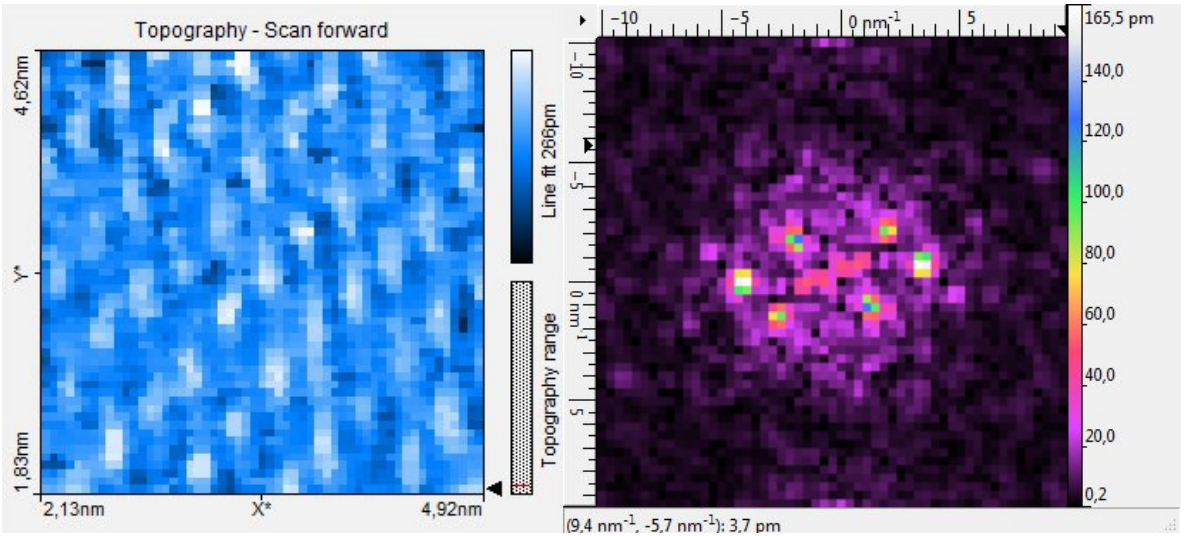


Fig. 15.1-18 Original scan and FFT of MoS_2

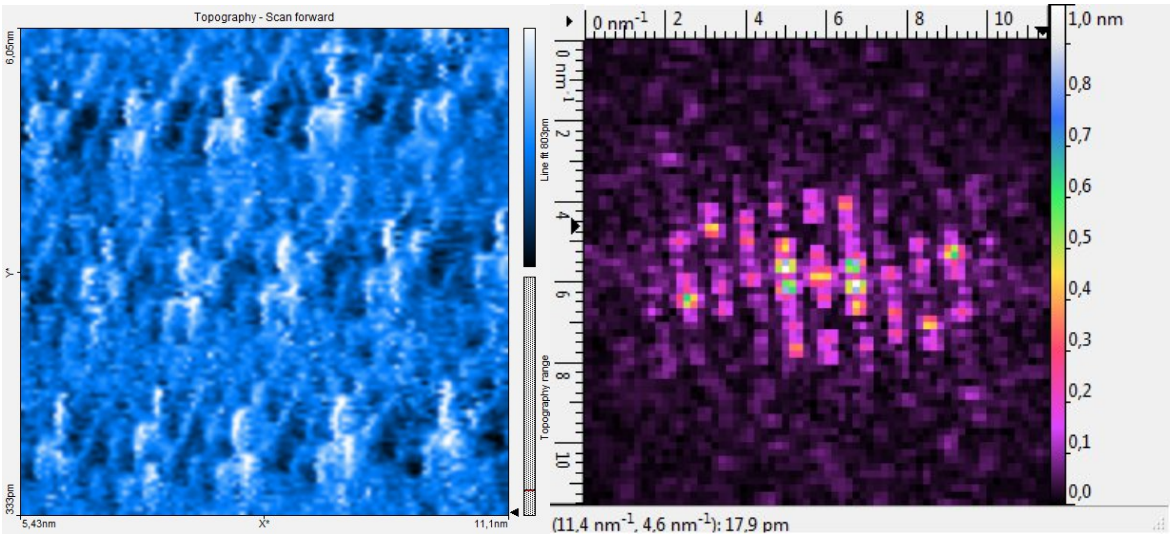


Fig. 15.1-19 Original scan and FFT of TaS_2

In the FFT of TaS_2 (Fig. 15.1-19) on can identify an inner hexagonal shape, representing the CDW and an outer hexagonal shape representing the atomic lattice. Image 15.1-20 shows the same images as before with the indicated structures and the corresponding lengths.

One sees that the reciprocal lattice is the original lattice rotated by 30° and the length of the sides inverted. The inner hexagonal shape in k-space has the inverted side lengths of the CDW structure in position space (yellow). The outer side length of 2.9 nm^{-1} (green) corresponds to the vertical lattice period 3.433 Å we determined before.

For the other scans one can determine the periodicity of the lattice from the reciprocal lattice in the same way.

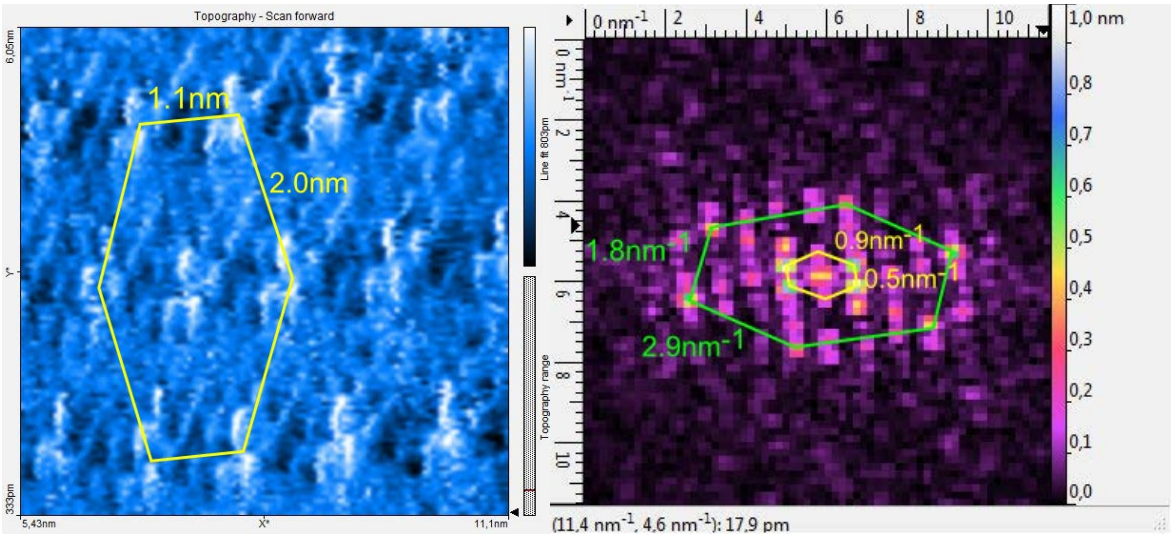


Fig. 15.1-20 Original scan and FFT of TaS_2

A further very useful application of the FT is frequency filtering: First one applies the FT to a signal. Then only certain frequency intervals in the transformed signal are selected, representing the structure one wants to investigate. Afterwards the inverse FT is applied to the selected periods resulting in an image mostly free from noise. Examples are shown in Image 15.1-21 and 15.1-22.

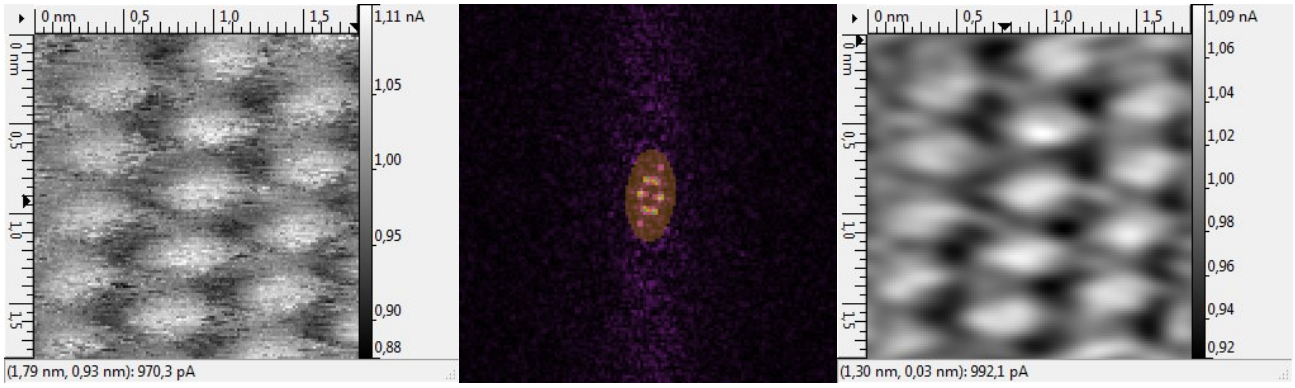


Fig. 15.1-21 (HOPG) Original Image - k-space with selected periods – retransformed Image

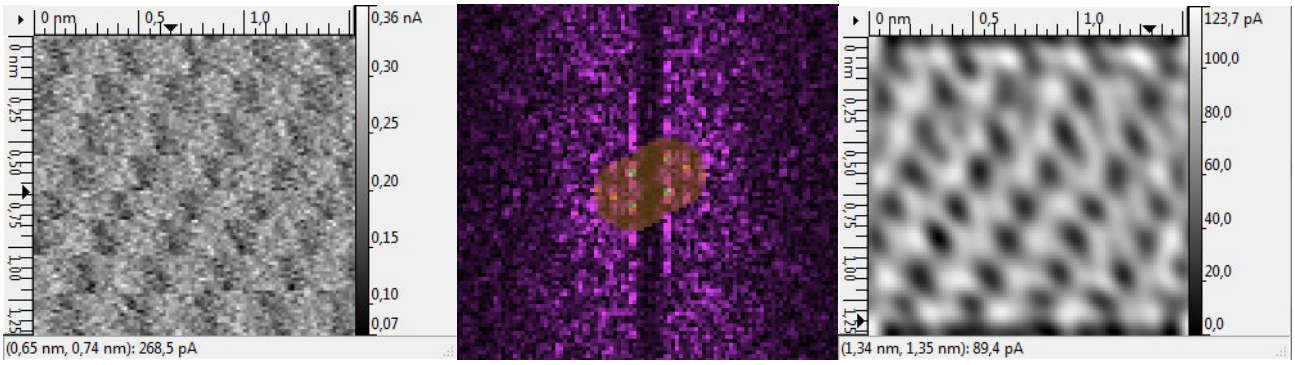


Fig. 15.1-22 (WSe_2) Original Image - k-space with selected periods – retransformed Image

2D Autocorrelation

Another possibility to improve scans is autocorrelation. This is a mathematical tool for finding repeating patterns, such as a periodic signal buried under noise. The autocorrelation function is define as

$$R(\tau) = \int_{-\infty}^{\infty} f(t)\bar{f}(t-\tau)dt.$$

Image 15.1.23 shows the original scan from Image 15.1-21 and the corresponding image improved by autocorrelation. Furthermore Image 15.1-25 shows the autocorrelation of the surface of WSe_2.

Conclusion

For all three samples one can achieve good scan quality although with different difficulty.

For the measured step heights we notice that MoS_2 obviously tends to form multi-layer steps while HOPG and TaS_2 more likely form single or double layer steps. All measured layer heights were pretty close to the theoretical values. The main error sources here are the imprecise layer edges.

To reach atomic resolution with HOPG is not very difficult. However it is difficult for MoS_2 and TaS_2. The measured lattice periods on atomic resolution tends to deviate from the expected values quite a bit. The

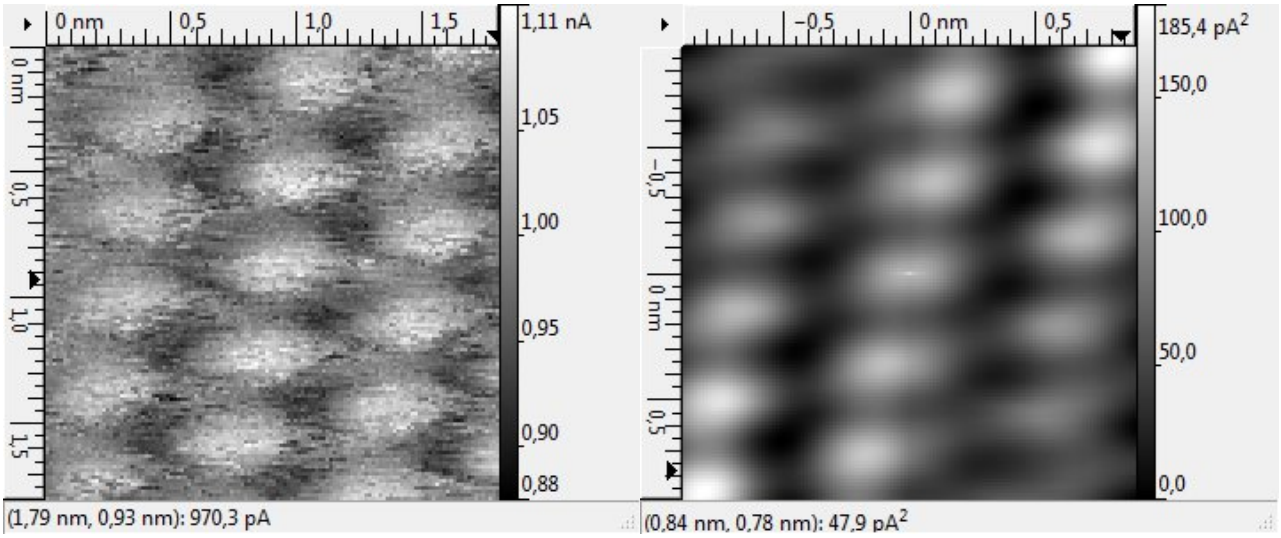


Fig. 15.1-23 (HOPG) Original Image – Image improved by autocorrelation

reason for this could be a not perfectly shaped tip, the thermal drift of the sample, which plays an important role at atomic resolution, creep or piezo non-linearity. The reason for the last point is that piezo crystals only move slowly into an equilibrium position after their elongation has been changed.

The FT provides information about the lattice parameter and structure of the sample quickly and more precise than measurements in position space do, because here the noise plays a less important role. Furthermore scanned images can be enhanced greatly by frequency filtering enabling a closer investigation than before. Therefore, the FT is a very powerful tool when investigating periodic structures.

Autocorrelation is a fast tool to improve noisy scans but has no adjustable parameters. In contrast, the FT provides the possibility to try out different frequency filters to receive the best result which is more time intensive though.

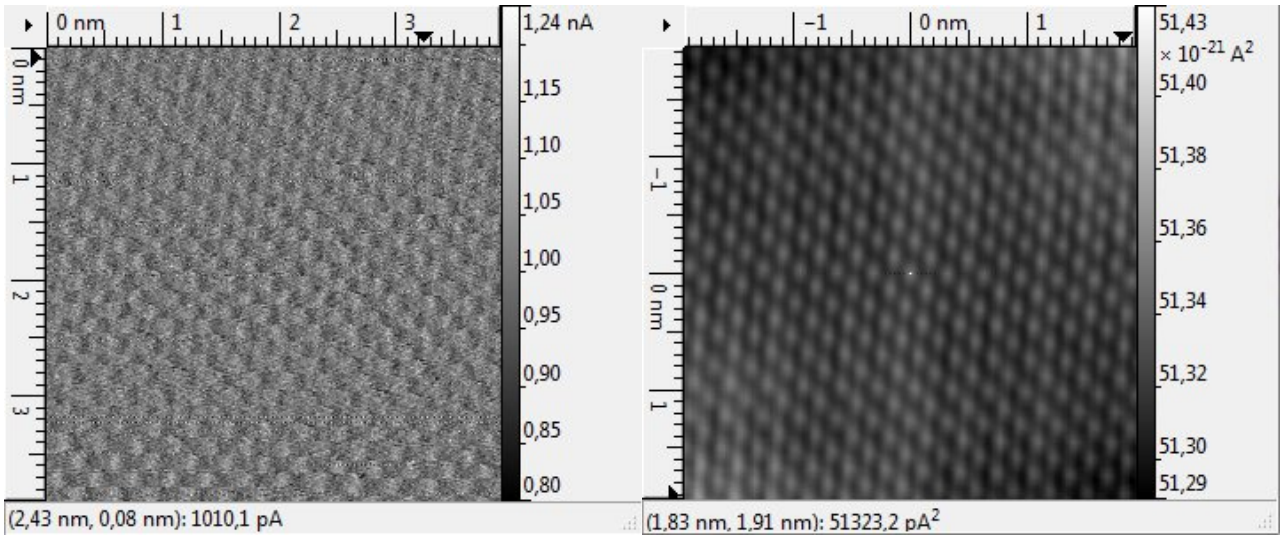


Fig. 15.1-25 (WSe_2) Original Image – Image improved by autocorrelation

15.2 Surface structure characterization – roughness and nano morphology

References:

- ***P2537000 "Investigation in roughness and nano morphology of different conductive samples by STM"***

Task: Investigate the topography of several samples with respect to their roughness.

The difficulty in scanning extremely rough surfaces as we will do in this experiment is that the piezo crystals have a limited speed and range of operation. Therefore extreme height differences in a small area can lead to scanning artifacts or even crashing the tip into the surface.
One way to prevent this from happening and achieving good images is to decrease the scanning speed meaning to increase the time per line of scanning. Furthermore it's recommended to start scanning just a small area of few 100 nm² slowly increasing the image size as desired.
When receiving artifacts one can try to furthermore decrease the image size or scanning again with a higher tip-surface distance i.e. increasing the tip voltage or decreasing the set point followed by withdrawing and re-approaching the sample.
To receive good scans of large areas of a rough sample the sequence of procedure should be as followed:

1. Prepare a tip and do a surface scan on HOPG to check if the tip is good by resolving single atoms
2. Change to the rough sample and start scanning with a low image size (e.g. 10 nm x 10 nm)
3. If you receive artificial structures in the scanned images try increasing the time / line or if this won't help increase the tip voltage and/or lower the set point, withdraw the sample and re-approach it
4. If still receiving artifacts repeat 3. until you receive good scans. If not receiving good scans even with tip voltage > 5V and set point < 0.5nA withdraw the sample and try scanning the surface at a different location
5. Increase the scanning area.
6. If receiving good scans repeat 5. Otherwise start over at 3.
7. When reaching the desired image size adjust the gain values to smooth the image. You can also try to increase the points / line for a better scan quality.
8. If you receive a statisfactory scan apply some filters to reduce noise, adjust scan line levels and remove a background distortion. The recommended procedure would be:

- Apply glitch filter.

- Apply noise filter.
- Do a background substraction and/or correct scan line levels.

Your corrected image should be almost free of strong gradients, one plane of the sample surface should have the same color in every point

After scanning a sample you can use PHYWE Measure Nano's integrated function *Calculate Area Roughness* to determine the nanoscopic roughness of the sample. This will result in an output as shown in figure 15.2-1. You can look up the meaning of the single values in *chapter 4.3.4.3: Roughness group* (page 45).
We are mostly interested in S_y which is the maximum height difference of any two points on the surface because it reflects the roughness of the sample with respect to step heights.

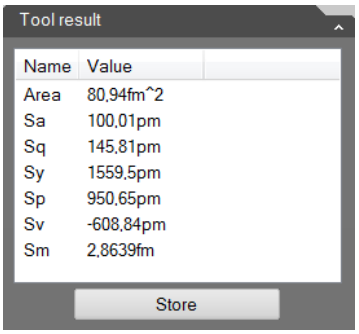


Fig. 15.2-1 Area Roughness measurement

Comparison of Surfaces
Due to the different procedures of manufacturing of several samples we expect the surfaces to have characteristic structures. In the following you can see pictures taken from grown, rolled and coined surfaces. Some of the images are displayed as shaded map (*chapter 4.3.2.3 Color map options* (page 34)).

Tantalum sheets are usually rolled and have a shiny and a dark side. Images 15.2-2 and 15.2-3 show the two surfaces of such a sheet in nanoscopic solution. One sees the shiny side consists of longish structures whereas the dark side more likely has island-like structures.
Figure 15.2-4 shows the surface of a Copper sheet, which has also been rolled but its two surfaces do not differ. One immediately sees the similarities in figure 15.2-2 and 15.2-4.
Fig. 15.2-5 shows a coined copper surface of a European 1 cent coin. The structures are round and chaotic. Figure 15.2-6 shows HOPG which is grown and consist of single layers which are strongly bonded within themselves but only bonded among each other by the weak van-der-Waals force. Furthermore the Gold in Fig. 15.2-7 which is also grown shows atomic steps. Tantalum disulfide (TaS_2, Fig. 15.2-8) which is also grown consists of layers like HOPG but is much more brittle. This means the single layers are not as strong bonded within themselves as those of HOPG resulting in smaller flat terraces (comp. Fig. 15.2-9).

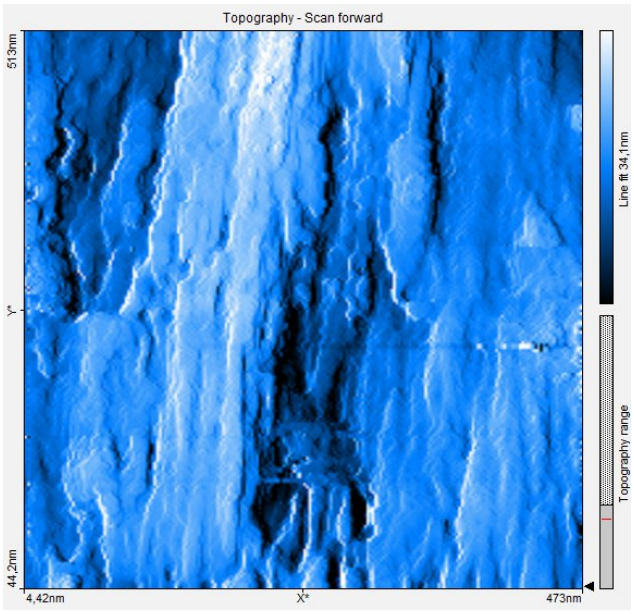


Fig. 15.2-2 Ta shiny side

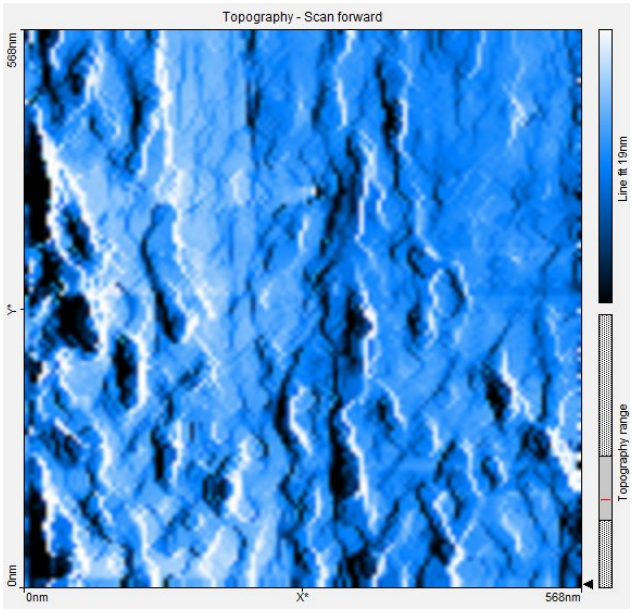


Fig. 15.2-3 Ta dark side

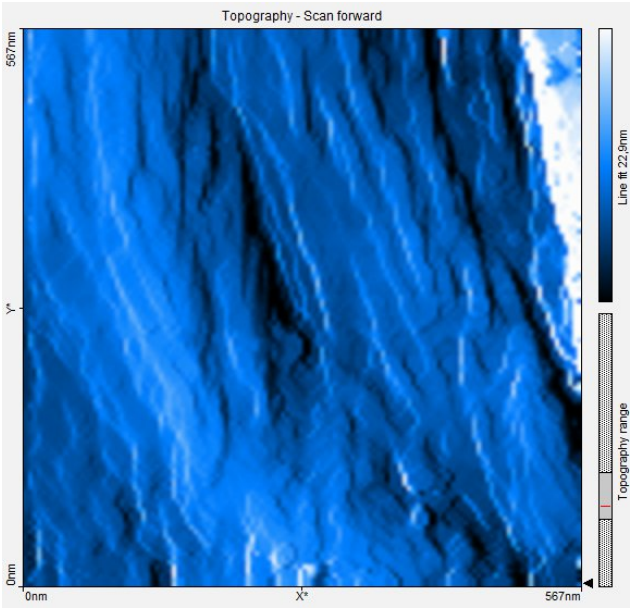


Fig. 15.2-4 Cu

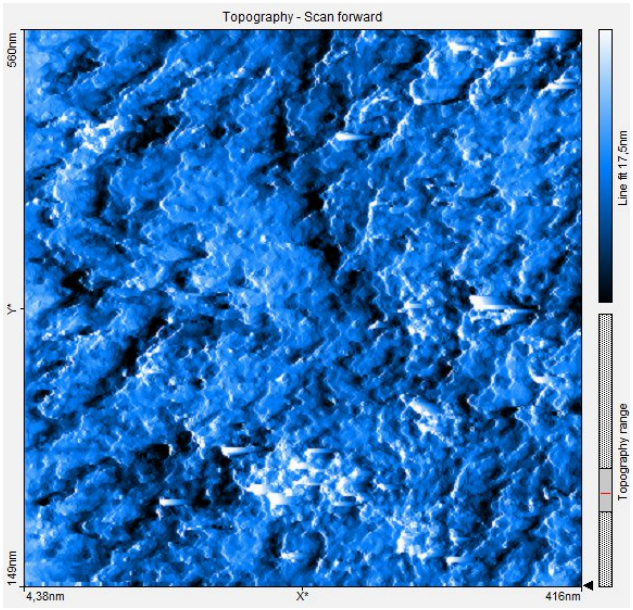


Fig. 15.2-5 Copper Coin

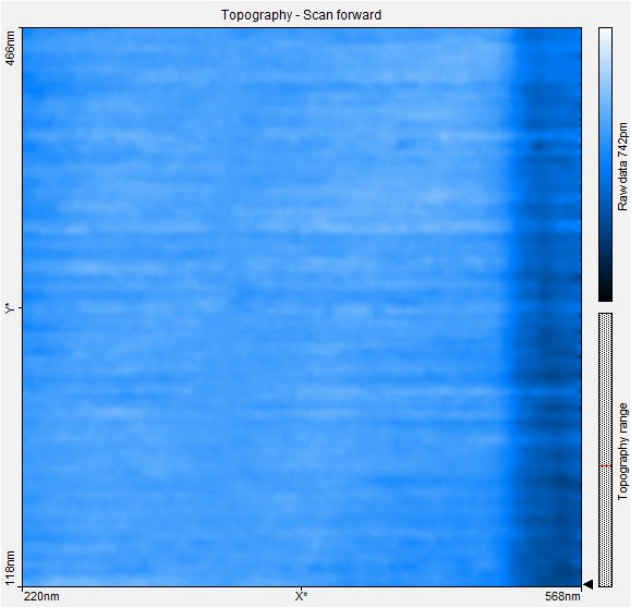


Fig. 15.2-6 HOPG

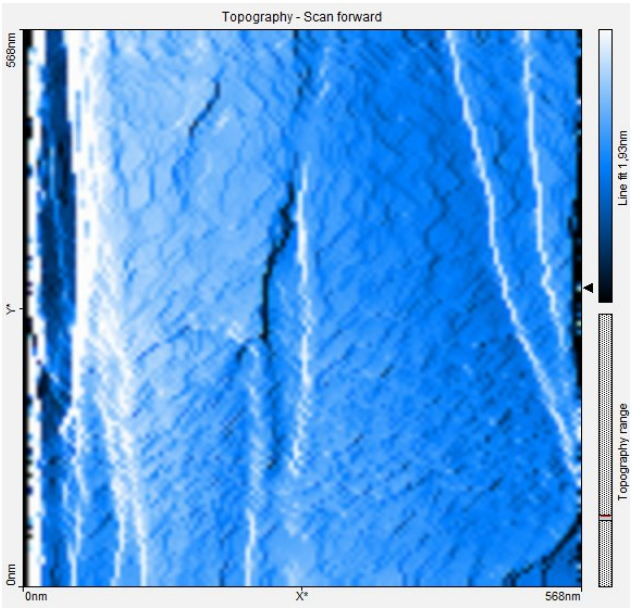


Fig. 15.2-7 Gold

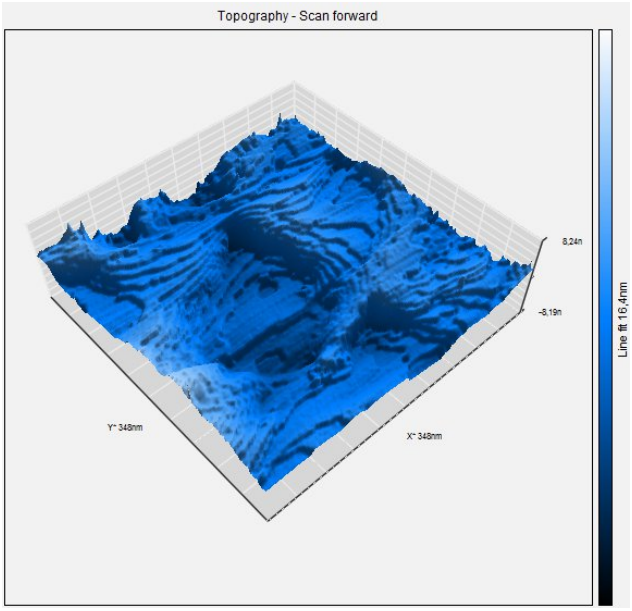


Fig. 15.2-8 TaS_2. Size 348 nm x 348 nm x 16.43 nm

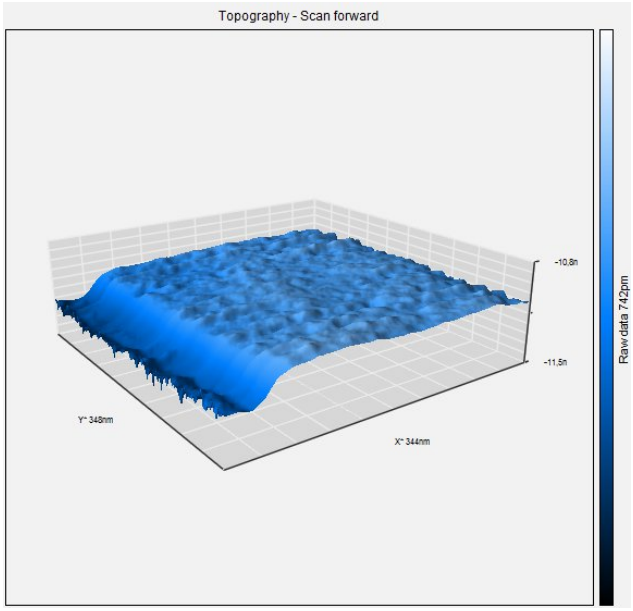


Fig. 15.2-9 HOPG. Size 348 nm x 344 nm x 0.7 nm

Material	Cu	Ta shiny	Ta dark	HOPG	Gold	Coin	Sn	TaS_2
Sy [nm]	33.3	38.2	56.1	0.4	4.4	31.3	133.0	14.0

Tab. 1 Area Roughness

Table 1 shows the outputs of S_y when applying the area roughness tool on the scans shown above. One sees the surfaces can be categorized by their roughness as done in table 2.
Regarding the manufacturing procedures of the surfaces in table 2 the rolled and coined materials are rough and the grown materials are plain which corresponds with expectations.

Conclusion
The longish nanoscopic structure of rolled materials can be explained by the stretching process the materials experience when they are flattened by rolling.

The chaotic structures of the coin surface are reasoned in the fast coining process during which the surface is exposed to high forces and immediately cools down afterwards.

The plain surfaces of the grown materials are rooted in the growing process in which the single atoms lay down onto the surface slowly after each other. Because the smallest possible surface generally is energetically favorable the material will form plain structures.

An important result of our measurements is that the surface roughness of coined materials does not differ from rolled but when looking at the to-

Rough	Plain
Cu	HOPG
Ta shiny	Gold
Ta dark	TaS_2
Coin	
Sn	

Tab. 2 Materials categorized by roughness

pography one can identify the coined material by the round, chaotic structures whereas rolled materials have longish structures.

Furthermore we have seen that macroscopically shiny materials are not necessary plain on the nanoscopic scale and vice versa.

15.3 Differential conductivity – nanoscale electrical characteristics

References:

- ***P2533500 "Nanoscale electrical charakteristics of different samples by STS"***

Task: Investigate the differential conductivity of HOPG, gold and MoS_2 by I-U tunneling spectroscopy.

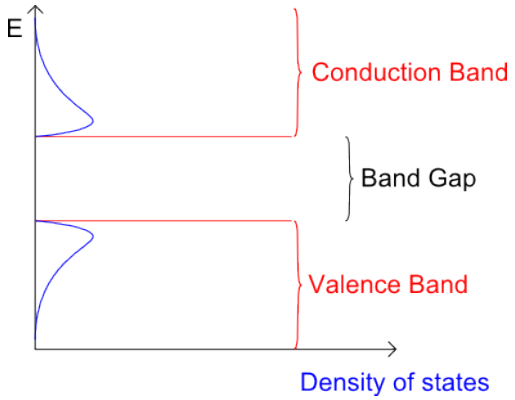


Fig. 15.3-1 Sketch of the band structure of an insulator/semiconductor

The electrons of a single atom can occupy discrete energy levels forming atomic orbitals. Several atoms together into a molecule will form molecular orbitals where the number of molecular orbitals is proportional to the number of atoms is the molecule.
When large numbers of atoms (>10^{20}) are brought together to form a solid there are large numbers of energy levels occupied by electrons and the difference between them becomes very small, forming energy bands. Hereby, the density of states (DOS) gives the number of electrons which can occupy certain energy levels. Depending on the atoms forming the solid there are energy intervals which cannot be occupied by electrons no matter how many atoms are aggregated (i.e. the DOS vanishes). Such intervals are called band gaps (Fig. 15.3-1). Band gaps only occur in semiconductors (small band gap < 3 eV) and insulator (large band gap > 3 eV). Solids which have no band gap, meaning Conduction band and Valance band overlap, are called conductors.

Regarded in detail, the electronic band structure of a solid is very complex. It depends on the properties of the underlying crystal lattice and is described by graphs as shown in Fig. 15.3-2. Here the energy is plotted as a function of the wave vector k, which describes the motion of the electrons in the crystal lattice. The wave vector k takes on values within the Brillouin Zone, which is a model to describe unit cells, corresponding to the crystal lattice. Particular directions/points in the Brillouin Zone are assigned conventional names like Γ, Δ, Λ, Σ, *etc.* The areas, electrons are allowed to be are highlighted grey.

Regarding the possible configurations in Fig. 15.3-3 one can see the difference between the different types of conductors. The materials used in our measurements are a conductor/metal (gold), a semiconductor (MoS_2) and a semi metal (HOPG).

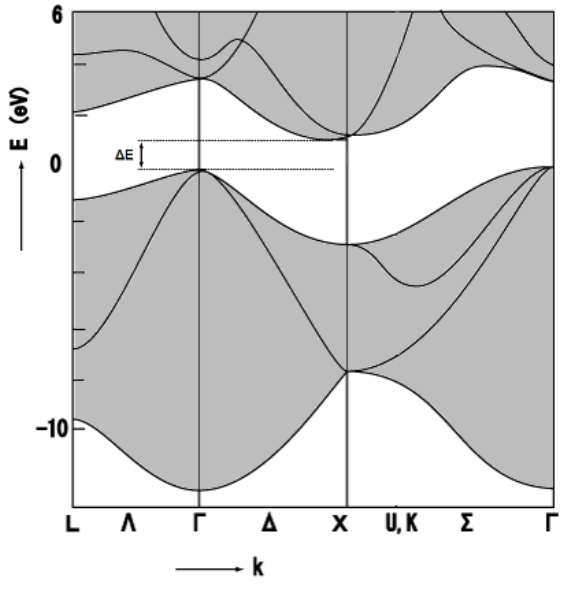


Fig. 15.3-2 Reduced band structure of Si with indicated bandgap ΔE

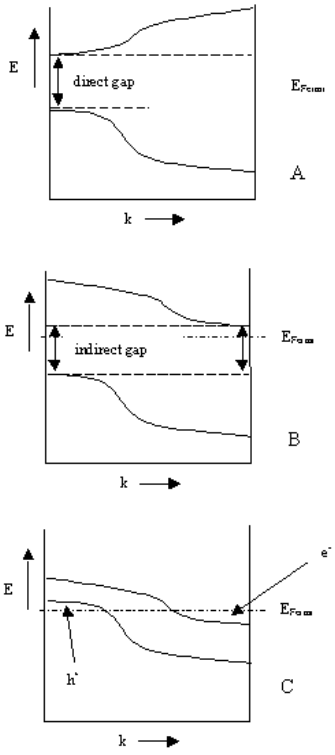


Fig. 15.3-3

A: Direct band gap semi conductor

B: Indirect bandgap semi conductor

C: Semi metal

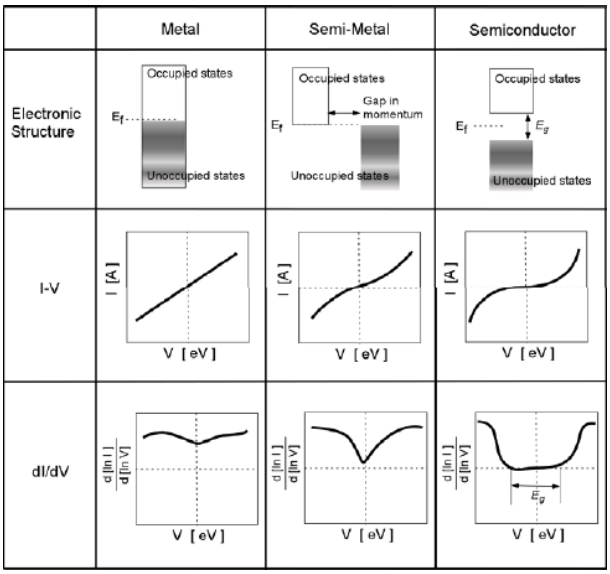


Fig. 15.3-4

Properties of different kinds of conductors

The LDOS (Local DOS) is a space-resolved description of the number of states at each energy level which are available to be occupied in a system. The LDOS is proportional to the slope of the characteristic curve received from I(U)-Spectroscopy (dI/dU) when regarding the tip's density of states to be with no structure. We will use this to image the band structure of (semi-)conductors. The measured curves are an overlay of the bands in the corresponding band scheme. The expected results are shown in Fig. 15.3-4.

Always keep in mind to do measurements preferably at the same room temperature. Temperature dependencies create uncertainty and errors in the expected values of your measurements.

Investigation of band structure

The procedure imaging the I(U)-spectroscopy curve is the same for all three samples. After you approached the sample click the Full button in the imaging window to activate the full measurement range. Now zoom in to atomic resolution to check the tip. Adjust the parameters so that you can see a detailed picture of the surface. Now, you can apply some filters to reduce noise, adjust scan line levels and remove a background distortion.

The recommended procedure would be:

- Apply glitch filter.
- Apply noise filter.
- Do a background correction and/or correct scan line levels.

The samples surfaces are shown in Fig. 15.3-5, 15.3-7 and 15.3-9. Furthermore the corresponding reduced band structures are shown in Fig. 15.3-6, 15.3-8 and 15.3-10.

Change back to full measurement range and switch to the spectroscopy mode. Choose “Tip voltage” as modulated output to record an I(U)-curve by using the “Tip voltage” spectroscopy mode in a plane region. Pin down values of your measurement parameters accurately. If you experience problems in measuring the desired behavior, try to adjust your parameter values. For example you can start experimenting with the following set of parameters:

- Set point 1.0 nA
- P-Gain 1000
- I-Gain 2000
- Tip voltage 0.05 V
- Start value -0.5 V
- End Value 0.5 V
- Modulation time 0.2 s
- Data points 128
- Averages 16

By using this set of parameters, the device will divide the interval from -0.5 V to 0.5 V into 128 points and measure the tunnel current at each of them. The time used for one series of measurements is 0.2 s. This process will be repeated 16 times. The resulting curve is the average of the 16 measurements.

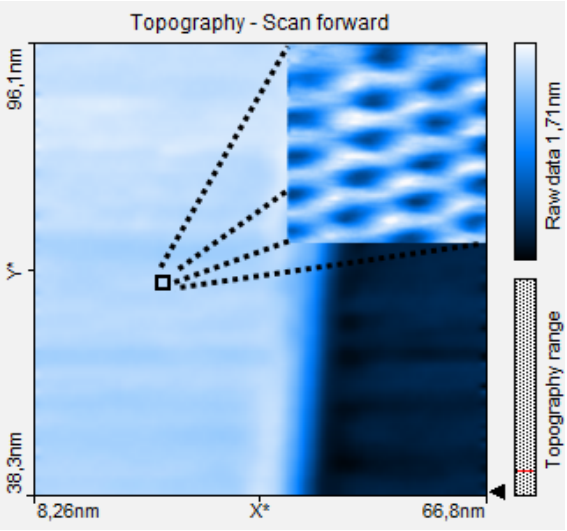


Fig. 15.3-5 Topography of HOPG

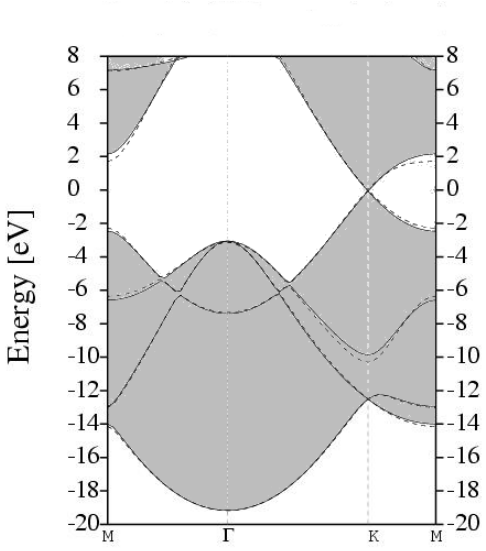


Fig. 15.3-6 Reduced band structure of a single layer of HOPG

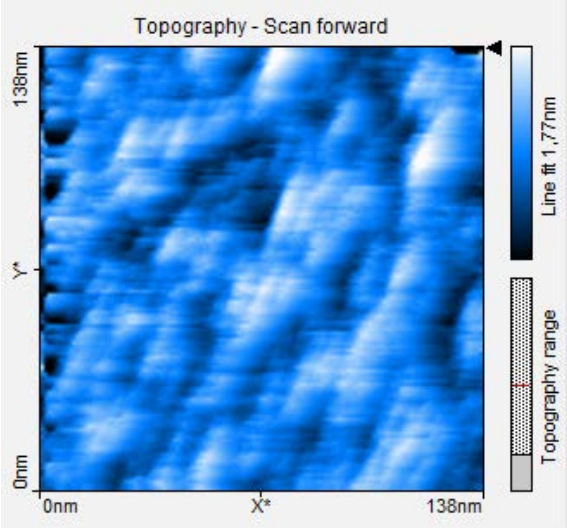


Fig. 15.3-9 Topography of MoS_2

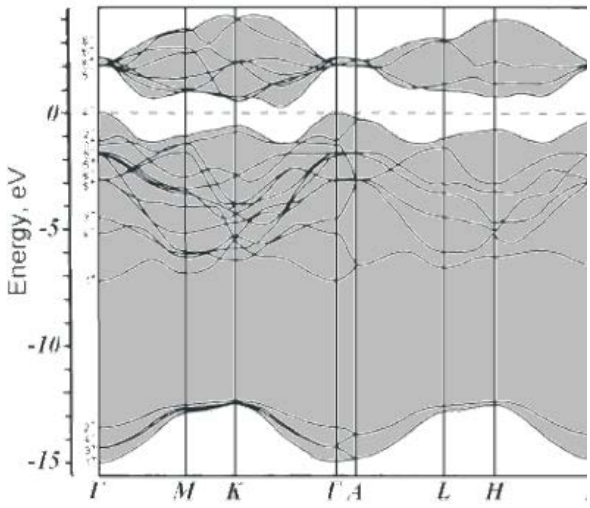


Fig. 15.3-10 Reduced band structure of MoS_2

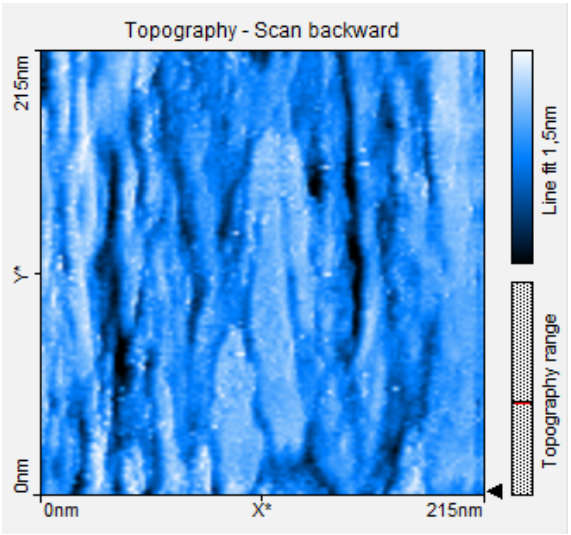


Fig. 15.3-7 Topography of gold

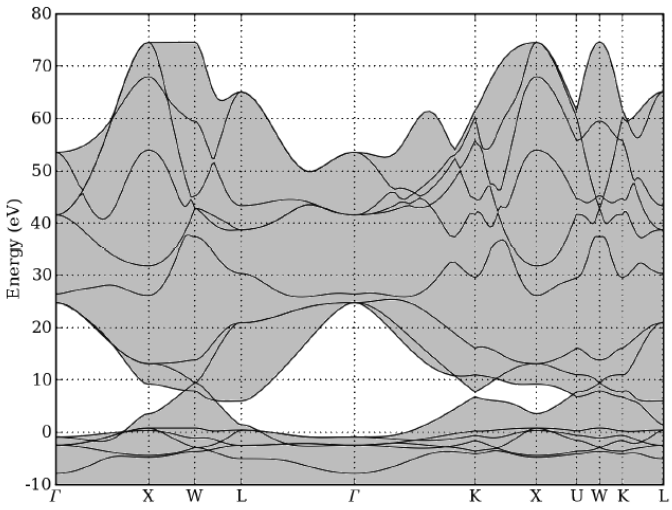


Fig. 15.3-8 Reduced band structure of gold

Notice:

- The device may cut off the graphs due to high tunnel currents. In this case adjust your Parameters to decrease the current (e.g. lower set point)
- High tunnel currents may also influence the structure of the tip and/or the surface. Therefore try choosing a lower set point or lower start and end values if the curve looks not as expected
- As long as you are in the spectroscopy mode the piezo controllers are inactive. Because of thermal drift you might receive different results when measuring the same point multiple times, especially when using high resolutions. Also modifications of the local tip geometry can occur during the measuring procedure indicated by noisy and “jumpy” spectra. Therefore check the topography after every spectroscopic measurement to evaluate the results.

Prepare a measurement by clicking on “Point” and putting the cursor on the surface of your scanned image where you want to take the spectroscopy data. Press the “Start” button. After a few seconds the end of the measurement is indicated when the “Stop” button changes back to “Start” and when your data

curve doesn't change anymore. The "Probe Status" should be green during the whole measurement. If the "Probe Status" changes to orange switch to the "Imaging Window" approach the sample again and take a new surface scan, afterwards switch back to the "Spectroscopy" window.

After the measurement is finished you can see the I(U)-curve in the line graph. If your curve does not look as desired repeat the measurement by pressing "start" again. If you still don't get the desired curves switch back to the imaging mode and repeat the process. To analyze the current-voltage curves right-click the line graph and click "Copy data to clipboard". Now open the analyzing software PHYWE *measure* and paste the data by pressing ctrl+v or using "Measurement" → "Import Data". You should receive a graph as shown in Fig. 15.3-9.

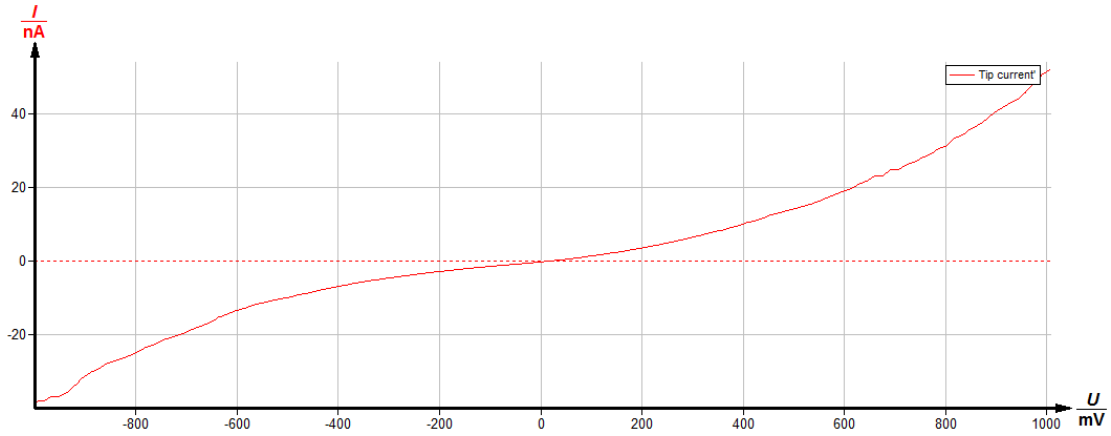


Fig. 15.3-9 I(U)-spectroscopy curve of HOPG

The derivate of the I(U) curve is called specific resistivity and is proportional to the LDOS. To analyze the derivate it is recommended to smooth the curve first. Therefore go to "Analysis" → "Smooth". Choose the strongest smoothing and overwrite the existent curve. Repeat the smoothing until you receive an appropriate curve as shown in the graphs. Now go to "Analysis" → "Channel Modification" and choose "differentiate" (Fig. 15.3-10). Now you can see the corresponding curve proportional to the LDOS.

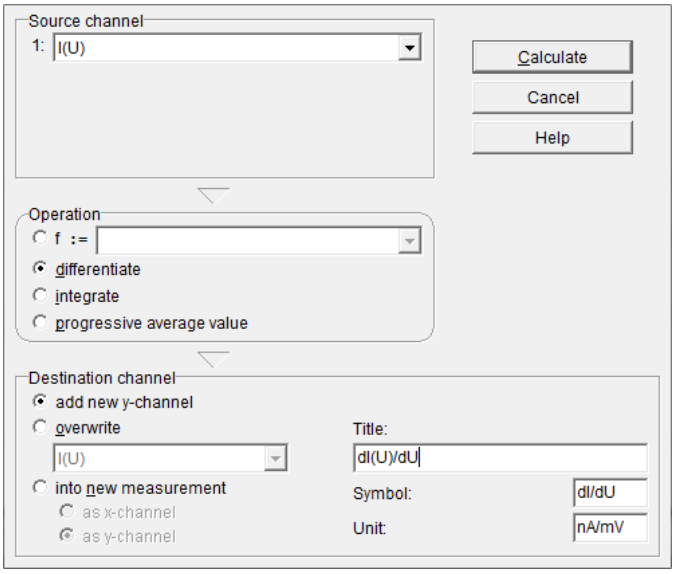


Fig. 15.3-10 Channel modification window.

Apply the spectroscopy mode on the other samples in the same way and compare the results. The resulting curves are shown in Fig. 15.3-11 to 15.3-13.

Note

The X-axis are zoomed in a bit (use mouse wheel on X-axis) because the smoothing process distorts the data at the edges.

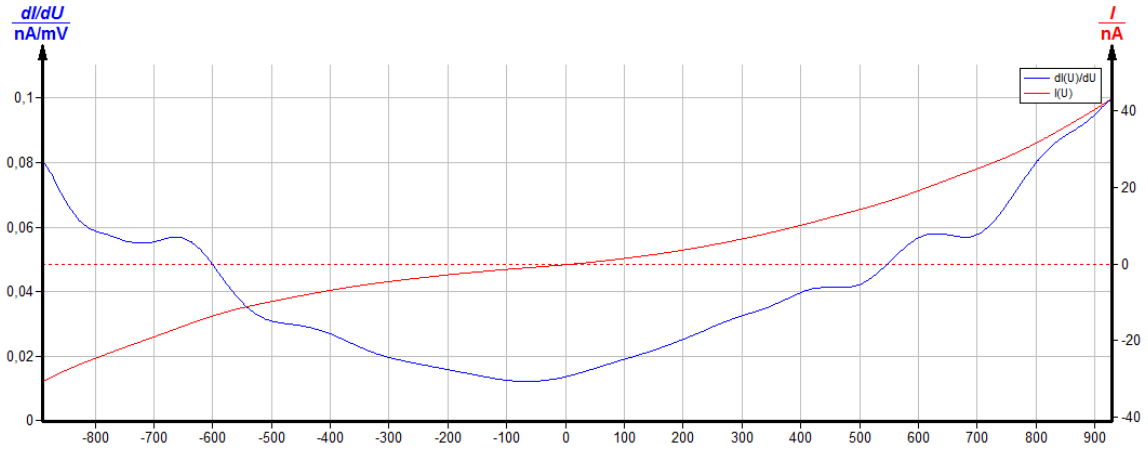


Fig. 15.3-11 Specific resistivity of HOPG

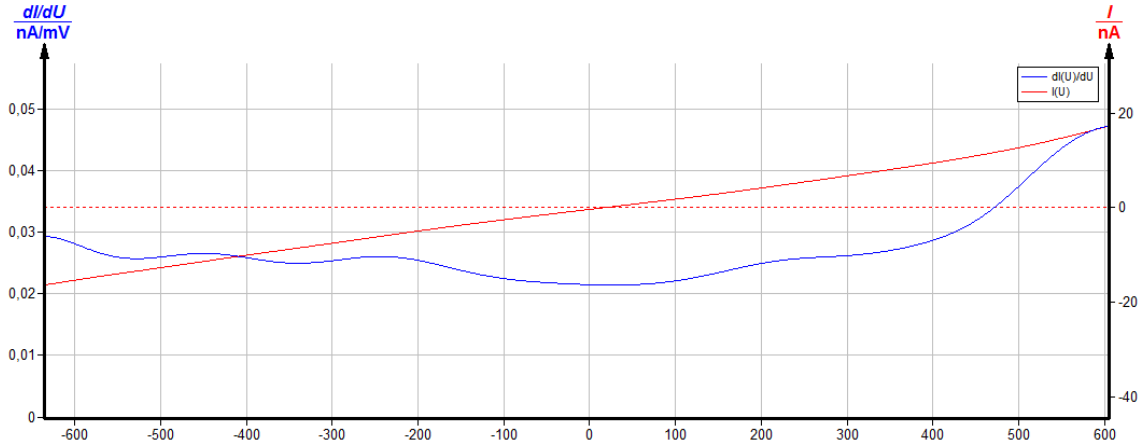


Fig. 15.3-12 Specific resistivity of gold

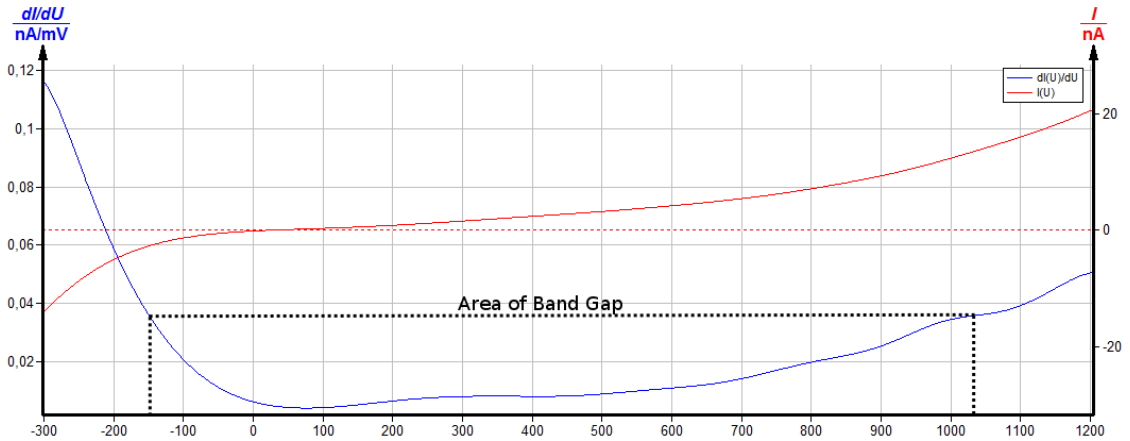


Fig. 15.3-13 Specific resistivity of MoS_2 with marked band gap

Conclusion

The first thing we see is that the LDOS of gold is described by a linear curve, while HOPG and MoS_2 are not.

This is the result of gold being a conductor. This means it has no band gap due to the valence and conduction band overlap, meaning electrons can move freely along the metal. The I(U)-spectroscopy-curve therefore images Ohm's law U=RI.

Regarding the band structure of HOPG and MoS_2 one sees there are regions where the LDOS is near zero. These regions are where the band gaps are. Theoretically we would expect the LDOS to be exactly zero, but impurities and the non-ideal measurement environment (air, high temperature) influences the recorded data here.

Furthermore one sees the characteristics of the two curves (e.g. extrema) differ. Especially remarkable is that the curve of Graphite is approximately symmetric to 0 V, whereas the curve of MoS_2 is not symmetric at all. This comes from the underlying band structure.

Our results correspond to the typical properties of (semi-)conductors from figure 15.3-4. Therefore we can determine the kind of a material of a sample by just seeing it's I(U)-spectroscopy-curve.

The theoretical band gap of MoS_2 is 1.29 V wide and is indicated in Fig. 15.3-13.

15.4 Charge density waves (CDW) – quantum mechanics

References:

- ***P2535000 "Quantum Mechanics by STM - Tunneling Effect and Charge Density Waves"***

Task: Characterize the Charge Density Waves on the TaS_2 surface and investigate empty and filled states.

Low Dimensional metals (quasi-1D or 2D) can undergo a phase transition involving electron phonon coupling. Hereby the atoms of the lattice change their equilibrium position. This is only possible if the cost of elastic energy needed for this deformation is compensated by a gain of electronic energy.

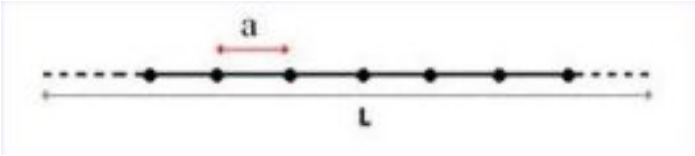


Fig. 15.4-3 Lattice with Period a

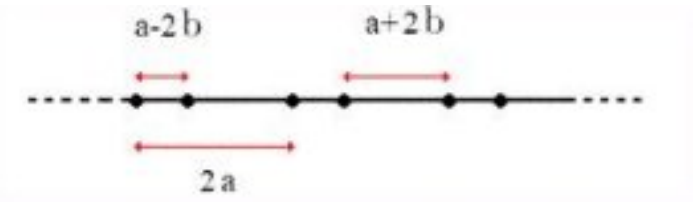


Fig. 15.4-4 Lattice after displacement of atoms

The phase transition results in a new electronic band structure and periodicity of the lattice. Charge density waves (CDW) are interesting for a couple of reasons including the prediction that such a state could lead to superconductivity and a special AC/DC response.

Fig. 15.4-3 shows a 1D-lattice with lattice parameter a. A possible second state is shown in Fig. 15.4-4. Here the atoms are successively displaced left or right by b<<a. The new Lattice Parameter is 2a. Other displacements with different lattice parameter are possible, too. Eventual, the lattice will configure in the energetically most favorable setting. That is also the reason why only certain materials are observed to form CDW.

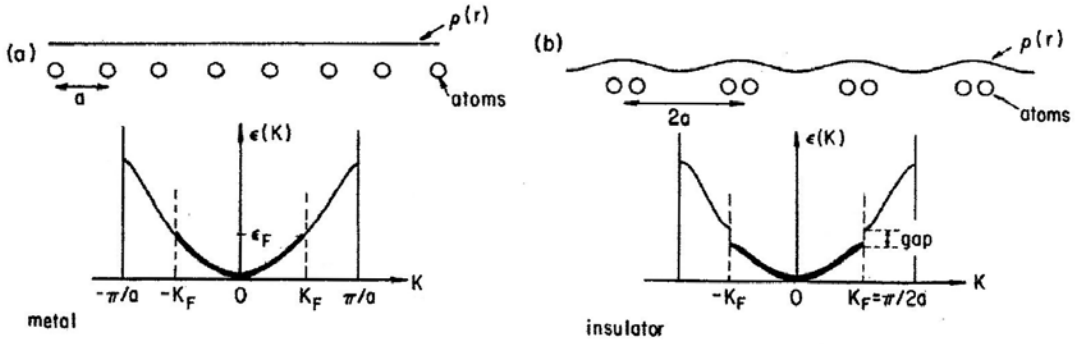


Fig. 15.4-5 Band structure of a 1D-lattice with Period a

Rudolf Peierls was the first one to explain this effect: In one dimension the periodicity of the crystal creates energy band gaps in the E(k) diagram at multiples of the value k = π/a. In the model the ions each contribute one electron, then the band will be filled up to the Fermi-energy E_F i.e. up to values of k_F = ±π/2a in the ground state as shown in figure 15.4-5 (a).

If the lattice period changes to 2a by lattice distortions as in figure 15.4-5 (b), this has the effect of introducing new band gaps at k = π/2a. This causes the electrons to be at lower energy than in the original lattice. Hence, this lattice distortion becomes energetically favorable when the energy savings because of the new band gaps is larger than the elastic energy cost of the lattice deformation. This effect will only be noticeable when the electrons are arranged in states close to the ground state meaning the lattice needs to be under a characteristic temperature, the Peierls temperature.

As we can see also from figure 15.4-5 the CDW state leads to a transition from a conductor to a semiconductor (insulator) because of the new band gap.

Although CDW typically are a one dimensional effect they can occur in higher dimensions. However, the theoretical background is much more complex.

TaS_2 is a transition metal chalcogenide in the 1T phase. This means each layer of Ta is packed between layers of S as shown in figure 15.4-6. The weak van-der Waals bonding between the single layers are the reason for CDW in the two dimensional layers.

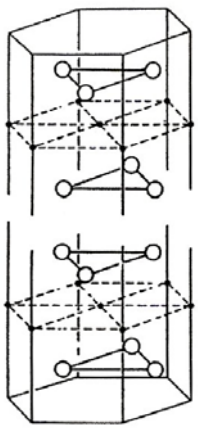


Fig. 15.4-6 Layer packing of samples
O: S, ⋅: Ta

The CDW can form a commensurable or incommensurable superlattice. Commensurable means the ratio between CDW period and atomic lattice period is rational whereas it is incommensurable when it's irrational. Furthermore semi-commensurable phases are possible in which there are areas with commensurable period as well as incommensurable.

Below 183K TaS_2 forms a commensurable phase. Between 183 K and 353 K TaS_2 forms a commensurable phase and an incommensurable $\sqrt{13}$ x $\sqrt{13}$ super-lattice. This nearly commensurable phase is what we will observe and is shown in Fig. 15.4-7. Above 353 K TaS_2 forms an incommensurable phase.

The new setting of the electrons can be directly investigated by STM. In the scans the CDW superimposes the atomic structure. To see both structures it is needed to bring the tip very close to the sample surface.

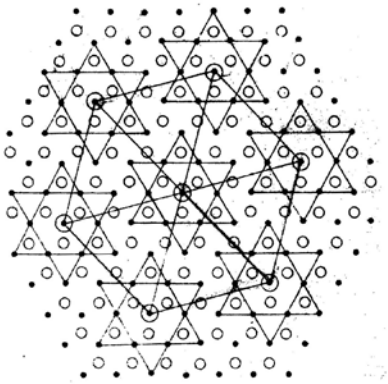


Fig. 15.4-7: Nearly commensurable phase CDW on TaS_2
O: S
.: Ta

The recommended procedure to achieve good scans is:

1. Prepare a tip and do a surface scan on HOPG to check if the tip is good by resolving single atoms (set point=1 nA; tip voltage=50 mV)
2. Change to the TaS_2 sample
3. Set image size to approximately 100 nm, tip voltage to 20 mV, set point to 4 nA and approach the sample
4. You should see terrace like structures (comp. fig. 15.4-8), if not try using the “cleaning pulse” button or withdraw the sample and re-approach a different spot on the sample
5. Zooming into a clean terrace, you should be able to see the CDW at a resolution of about (30 nm)² (fig. 15.4-9)
6. Continue zooming into an area with high periodicity until you receive the desired resolution
7. To obtain atomic resolution it is needed to increase the set point up to about 30 nA (withdraw and re-approach the sample). When increasing the set point remember decreasing the I- and P-gain respectively.
8. When obtaining the desired image adjust the gain values to smooth the image. You can also try to increase the points / line for even better scan quality.
9. Next, you can apply some filters to reduce noise, adjust scan line levels and remove a background distortion. The recommended procedure would be:

- Apply glitch filter.
- Apply noise filter.
- Do a background substraction and/or correct scan line levels

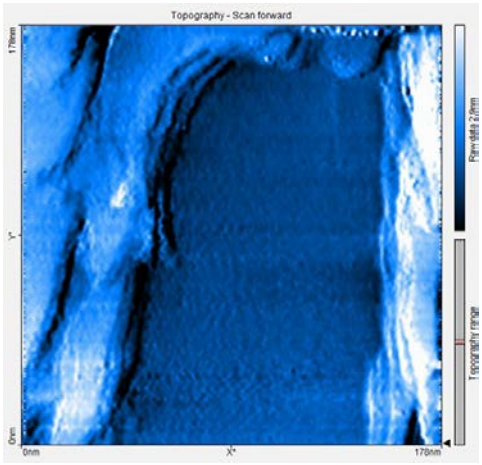


Fig. 15.4-8 Surface of TaS_2

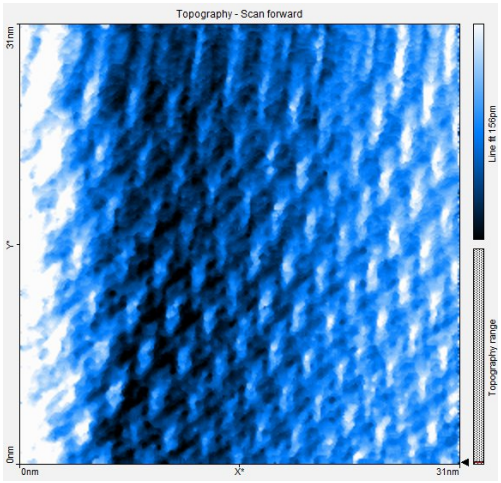


Fig. 15.4-9 CDW on TaS_2 at (31 nm)²

In theory the lattice parameter of TaS_2 is a= 3.346 Å. From the scan shown in fig. 15.4-10 one can determine the lattice parameter and period of CDW e.g. by using the *Measure Length* tool .
As shown in figure 15.4-11 we receive a mean horizontal period of $a_h = 3.433$ Å and a vertical period of $a_v = 4.633$Å. The mean period of the CDW maxima is 1.216 nm horizontally and 2,078 nm vertically leading to lattice periods $a_h' = 1.216\ \text{nm}/\sqrt{13} = 3{,}377$ Å and $a_v' = 2.078\ \text{nm}/\sqrt{13} = 5.772$ Å.

Furthermore we can measure the angles of the structure using the *Measure Angle* tool as in figure 15.4-12. We receive an angle of 109.5° between the maxima of the CDW. From figure 15.4-7 we know the angle between the hexagonal arranged CDW maxima is 120°.

Both, lattice period and angle measurement indicate that our scan is deformed. From the lattice period we know that the scan is ok in horizontal orientation but not in vertical. This could be a reasoned by a not perfectly shaped tip, the thermal drift of the sample, which plays an important role at atomic resolution, creep or piezo non-linearity. The reason for the last point is that piezo crystals only move slowly into an equilibrium position after their elongation has been changed.

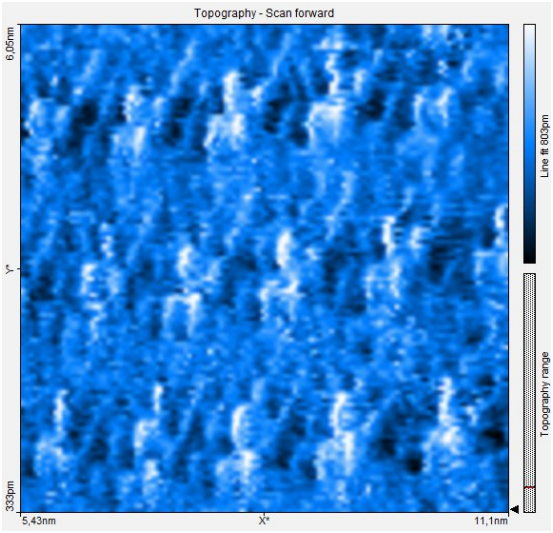


Fig. 15.4-10 CDW on TaS_2

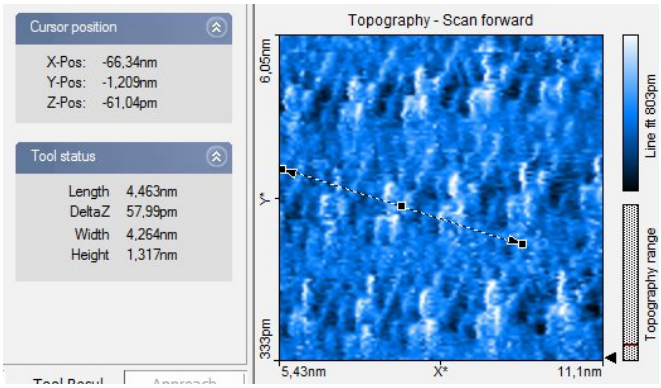


Fig. 15.4-11 Length measurement spanning 13a

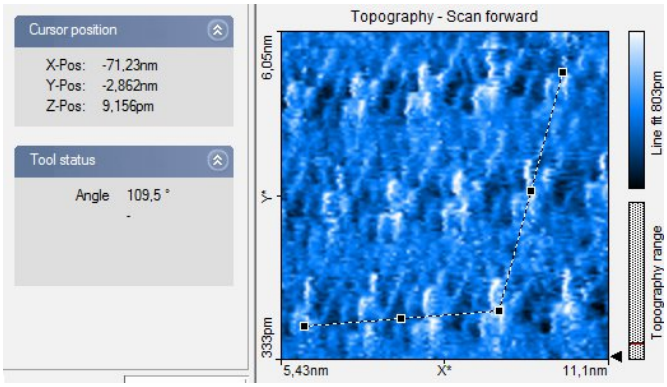


Fig. 15.4-12 Angle measurements of CDW maxima

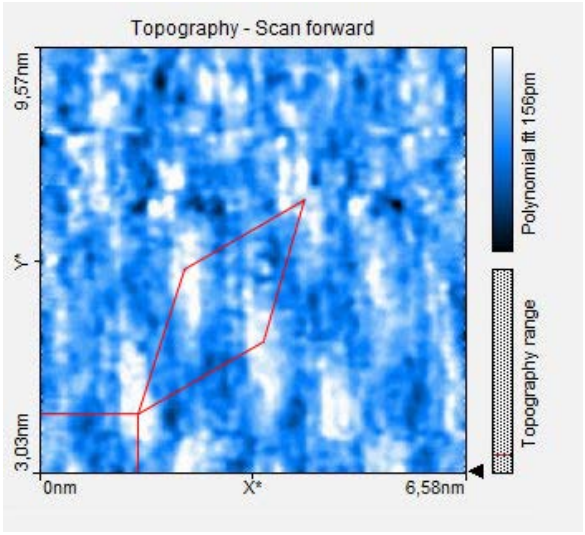


Fig. 15.4-13 (a) CDW at +30 mV tip voltage

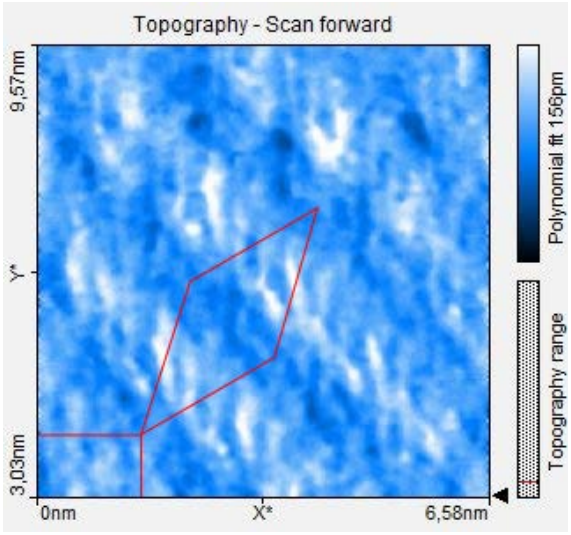


Fig. 15.4-13 (b) CDW at -30 mV tip voltage at same location

In the theory the band structure of the two dimensional sample is split up into three different bands instead of two as in the one dimensional case. This has the effect that the DOS is not very symmetrical around E_F. Only for higher energies the DOS becomes nearly symmetrical and is mainly leading to a contrast inversion when the bias voltage is inverted as shown in figure 15.4-13. Here the positive tip voltage images the filled states whereas the negative tip voltage images the empty states.
For lower bias voltages the CDW can appear similar to 15.4-13 (a) or (b), or even just as an array of dark spots.

Conclusion
All our measurements reflect the theory very good. Mainly image deformations lead to deviations in the atomic lattice and CDW periods.

All measurements we did on TaS_2 can be done on other CDW forming materials such as WSe_2, another transition metal chalcogenide, analogously.

15.5 Nanoscale work function

References:

- ***P2533000 "Nanoscale workfunction measurements by scanning tunneling spectroscopy"***

Task: Investigate the nano-scale work function of different surface sites by distance-current tunneling spectroscopy.

The strength of the tunneling current I depends exponentially on the distance between the tip and the sample (d_z), usually referred to as Z-distance and the applied bias (U). From Schrödinger's equation one can find that

$$I = f(U)\exp\left(-A\sqrt{\phi}d_z\right) \qquad [1]$$

where

$$A = 2\sqrt{\frac{2m_e}{\hbar^2}} = 10.25 nm^{-1}eV^{-0.5}.$$

Here ϕ is the effective work function to which the actual work function of the sample and the tip contribute making us only able to make relative conclusions, not absolute. Furthermore, it is influenced by surface adsorbates and the high electrical field density at the tip.
The extreme dependence on the distance makes it possible to measure the tip–sample movement very precisely. By keeping the tip voltage constant and measuring the tunnel current I in dependency of d_z the effective work function can be determined either by fitting an exponential function to the measured tunnel current [1] or by applying the logarithm to [1] which leads to

$$\ln(I) = -A\sqrt{\phi}d_z + \ln\left(f(U)\right).$$

This is a linear function of d_z so ϕ can be determined from the slope of the expected straight line by a linear fit.
By the combination of the high resolution of the tunnel microscope and the spectroscopy function it is possible to determine work functions at nano-scale.

Always keep in mind to do measurements preferably at the same room temperature. Temperature dependencies create uncertainty and errors in the expected values of your measurements.

Finding atomic terraces on the sample surface

It is recommended to start over with the graphite sample to practice before using the gold sample. To activate the full measurement-range of your device click the Full button in the imaging window. For good results you can use image sizes of about 0.2 µm. When you are lucky you find a terrace in your first measurement. If that is not the case you can either use the Move tool in the imaging window to start the measurement at a different spot on the surface or you retract the sample from the tip and carefully turn the sample holder with the black plastic handle. Afterwards, approach the sample again.

In figure 15.5-1 you can find examples of terrace structures at different spots on the HOPG and gold sample surface. Make sure you adjust the parameters of the feedback loop to achieve good image quality. Too high values in the P-and I-gain will be noticeable in the line graph as very high peaks and a very rough line structure. Adjust the gain values to smooth the line graph and your image respectively. Next, you can apply some filters to reduce noise, adjust scan line levels and remove a background distortion. The recommended procedure would be:

- Apply glitch filter.
- Apply noise filter.
- Do a background correction and/or correct scan line levels.

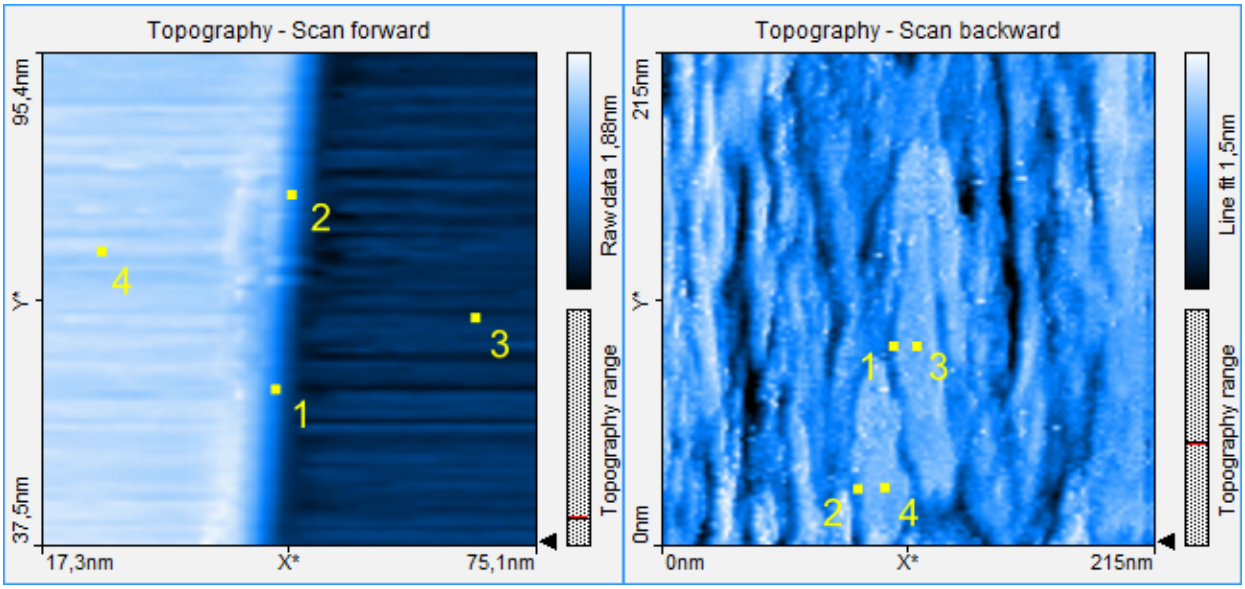


Fig. 15.5-1 Examples of terraces on HOPG (left) and gold (right) recorded at tunnel current 0.5 nA, tip voltage 0.1 V.

Determination of work function

After grabbing a good image switch to the Spectroscopy mode and load the scan. In the spectroscopic measurement, the tunneling current is measured as a function of either the Z-distance or the tip voltage. To determine the work function select “Z-Axis” as modulated output. Pin down values of your measurement parameters accurately. If you experience problems in measuring the desired behavior, try to adjust your parameter values. For example you can start experimenting with the following set of parameters for HOPG:

- Set point 0.5 nA
- P-Gain 500
- I-Gain 500
- Tip voltage 0.1 V
- Start value 0 nm
- End Value -5 nm
- Modulation time 0.2 s
- Data points 128
- Averages 16

By using this set of parameters, the device will divide the distance from 0 nm to -5 nm from the current tip position into 128 points and measure the tunnel current at each of them. The time used for one series of measurements is 0.2 s. This process will be repeated 16 times. The resulting curve is the average of the 16 measurements.

Notice:
- For gold you will need higher gain values than for HOPG to be able to see the step structure like in figure 15.5-1.
- Positive Start and End values will bring the tip closer to the surface: 1 nm will change the tunnel current about 1 magnitude!
- High tunnel currents may influence the structure of the tip and/or the surface. Therefore try choosing a lower set point or lower start and end values if you receive high currents (>20 nA) and the curve is not exponential.
- As long as you are in the spectroscopy mode the piezo controllers are inactive. Because of thermal drift you might receive different results when measuring the same point multiple times, especially when using high resolutions. Also modifications of the local tip geometry can occur during the measuring procedure indicated by noisy and “jumpy” spectra. Therefore check the topography after the spectroscopic measurements to evaluate the results.

Before you start measuring switch to a dual-line graph first (”show reverse line” in window options, *chapter 4.3.2.2: Chart Properties dialog* (page 31)). Then prepare a measurement by clicking on “Point” and putting the cursor on the surface of your scanned image where you want to take the spectroscopy data. Press the “Start” button. After a few seconds the end of the measurement is indicated when the “Stop” button changes back to “Start” and when your data curve doesn’t change anymore. The “Probe Status” should be green during the whole measurement. If the “Probe Status” changes to orange switch to the “Imaging Window” approach the sample again and take a new surface scan, afterwards switch back to the “Spectroscopy” window.

After the measurement is finished you should be able to see two curves in the dual-line graph like in figure 15.5-2. One represents the forward measurement and one for the backward measurement. You should see an exponential dependence of current to distance for both of them. If not, repeat the same measurement pressing “Start” again. If you still don’t get the desired curves switch back to the imaging mode and start over.

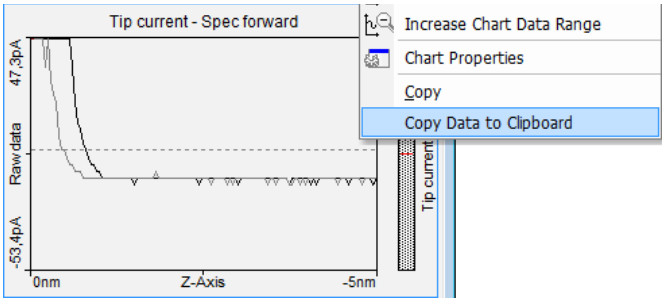


Fig. 15.5-2 Sketch of the dual-line graph

To analyze the current-distance curves right-click the dual-line graph and click “Copy Data to Clipboard” (Fig. 13). Now open the analyzing software PHYWE *measure* and paste the data by pressing ctrl+v or using “Measurement” → “Import Data”. A dialog will pop up. Select “Sort data. Ask again if any x-values occur twice”. You should receive a graph as shown in figure 15.5-3. Now go back to PHYWE Measure Nano and switch the spectroscopy signal of the dual-line graph by right-click → “Signal”. Export this curve to PHYWE *measure* in the same way. Now compare the curves for forward and backward measurement.

You will see the both curves to be different because of surface adsorbates which impact the tunnel current. When retracting the tip from the surface these adsorbates will provide a better connection between tip and surface, decreasing the actual work function. Therefore, we will only analyze the backward spec data which are less affected by this effect and leave the forward spec data aside.

To determine the effective work function we need to fit an exponential function. Therefore go to “Analysis” → “Function fitting” in PHYWE *measure* and select “exponential function” in the uppermost drop-down dialog. After clicking “calculate” you will obtain the fit parameters. By clicking “Add new curve” the fit curve will be drawn into the diagram, so you can visually evaluate the quality of the fit (Fig. 15.5-3).

From [1] we know for our fit parameter $b = A\sqrt{\phi}$ and therefore

$$\phi = \frac{b^2}{A^2}.$$

To obtain correct values for the work function ϕ, it is important to take the units of *b* into account.

Now that you are familiar with the procedure, apply the spectroscopy mode on some points on clean terraces and defects like step edges or holes on HOPG and gold and compare the measured work functions. The measured data will vary for each tip so in order to compare results you should use the same tip for the different measurements.

For example the measurement on a clean terrace on the HOPG sample (Fig. 15.5-1, point #3) gives the curve in Fig. 15.5-2. From the fit we know that

$$b = 8.78\ nm^{-1}$$

so

$$\phi = \frac{b^2}{A^2} = \frac{8.78^2}{10.25^2}\ eV = 0.73\ eV.$$

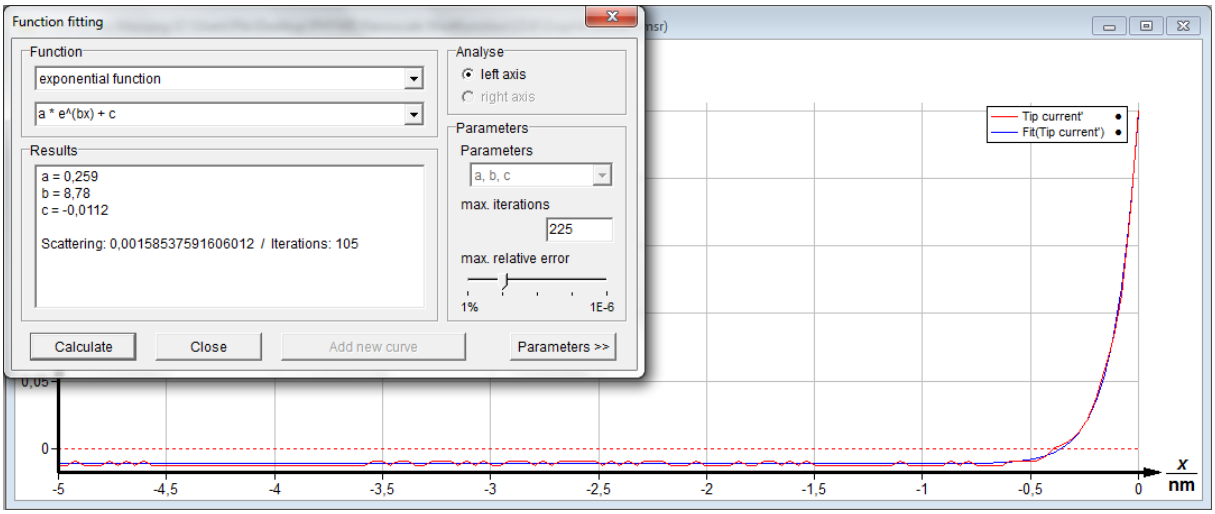


Fig. 15.5-3 Function fitting window in PHYWE *measure*

For the other points in Fig. 15.5-1 one can calculate the work function in the same way. The results are

Point #	HOPG	Gold
1	0.27 eV	0.11 eV
2	0.26 eV	0.12 eV
3	0.73 eV	0.21 eV
4	0.64 eV	0.18 eV

You can see that the measured effective work functions for gold are lower than for graphite, although the actual work function should be higher for gold. This comes from the different outside influences mentioned above and dif-

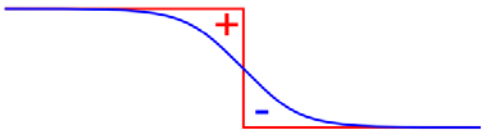


Fig. 15.5-4 Sketch of the charge distribution at a surface defect

ferent tips which have been used for the different samples.
One can see that for similar regions on one sample the work functions will not scatter a lot. Yet more important is the observation that the work function is generally smaller at defects than on clean terraces.

This can be explained by Smoluchowski's model. In this model the valence electrons in a metallic (metal like) solid are almost free and flow along the atomic cores. Defects in the solid's surface result in the expose of atomic cores while the electrons flow smooth across the defect (Fig. 15.5-4). This results in an abundance of electrons in certain areas (=negative charged regions with higher potential energies of electrons) for example the lower edge of step edges which then can be extracted easier.

Conclusion
The result of a smaller work function is a higher reactivity of a solid surface and therefore defects in the surface make solids more chemically active.
One application of this effect is to increase the effectiveness in catalysts by using materials with rough surfaces, where rough means the surface has a high density of defects.

We have seen that with the aid of STM one can determine work functions of nano structures. This ability can be used for many purposes e.g. measuring the work function of optically stimulated surfaces or nanostructures.

15.6 Arachin acid – self-assembled molecular network

References:

- ***P2534000 "Investigate in self-assembled molecular networks of arachin acid on HOPG by STM"***

Task: Investigate the topography of adsorbed arachidic acid molecules on the HOPG surface.

Preparation of Molecules
In this experiment we will investigate the adsorbation of arachidic acid on HOPG. For this one needs to set up a solution of arachidic acid in Phenyloctane.
First prepare a saturated stock solution: Solve 20 mg of the arachidic acid salt in 0.5ml of Phenylocatane, then take 0.2 ml of the stock solution and dilute with 20 µl Phenyloctane. The resulting solution is which we will use in the experiment.

Applying the molecules

Fig. 15.6-1 A drop adheres to the tip of the syringe

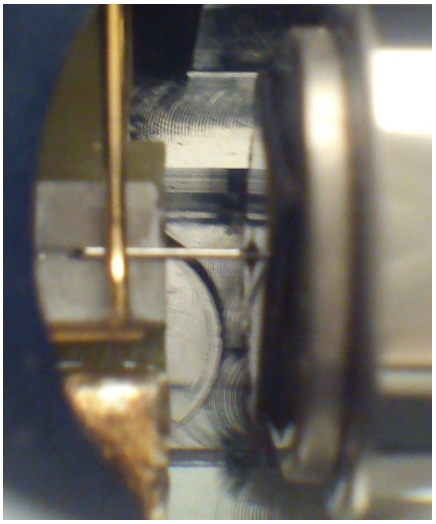


Fig. 15.6-2 The liquid meniscus surrounding the tip

To apply the molecules to the surface first stop the measurement on HOPG when seeing single atoms and withdraw the sample 2-3 times via the “withdraw” button. Remove the Cover from the STM carefully.
Now use the syringe with the smallest (brown plastic top) needle to aspirate a small drop of the thinned down arachidic acid solution. Withdraw the syringe from the solution and push its piston a bit, such that a drop of the solution adheres to the top of the needle. Now touch the HOPG surface near the needle with the drop carefully (figure 15.6-1). The drop will come undone the needle and disperse across the sample resulting in a liquid meniscus between the scanning tip and the sample as shown in figure 15.6-2. If there won’t be a meniscus repeat the process.

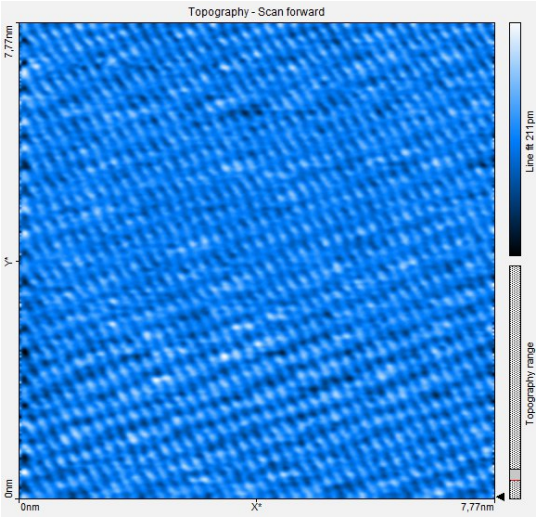


Fig. 15.6-3 HOPG lattice after applying the arachidic acid drop to the surface

Now place the cover carefully on the STM again. The status light should be orange all the time. After approaching the sample you should be able to see the atomic resolution of HOPG as before. If not, try scanning a different area of the surface (lower resolution -> move -> increase resolution on flat terrace).
If you are not able to receive atomic resolution, you probably have crashed the tip into the sample when applying the solution. Hence you will have to prepare a new tip and start over again.
For tip voltages of few 10mV the electrons will mainly tunnel from the orbitals of the HOPG (Fig. 15.6-3). When changing the voltage stepwise to values larger than 1V and adjusting the tunnel current respectively you should be able to see the molecules adsorbed on the surface. You will need to be patient in this step because the molecules need to assemble to the surface first. It often is useful to use cleaning pulses to help the molecules aligning on the surface. Typical scanning parameters to see the molecules are tip voltage 1.3 V, set point 0.6 nA, P-gain 1000, I-gain 1000.

Moiré patterns

Moiré patterns are beats of the lattice of the adsorbate and the substrate. As shown in figure 15.6-4 long-chain alkanes form a simple commensurable assembly on HOPG, i.e. the distance between the single molecules is an integer multiple of the HOPG lattice period.

However, Arachidic acid doesn’t form a simple commensurable assembly (3d>ΔA>2d) due to its functional acid group at one end. Hence neighbored arachidic acid molecules cannot occupy identical surface sites. This results in a modulated tunneling contrast with the moiré period ΔM (Fig. 15.6-6). It holds

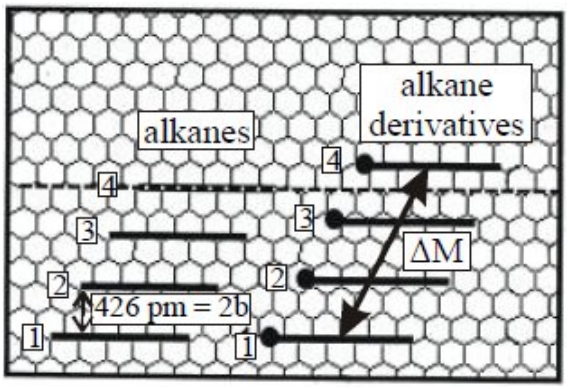


Fig. 15.6-4 Simple commensurable and not simple commensurable assembly on HOPG

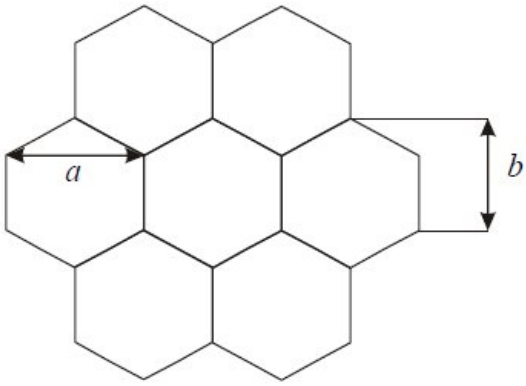


Fig. 15.6-5 HOPG lattice with $a = 24$pm and $b = \frac{\sqrt{3}}{2} \times 24_{\text{nm}}$

$$\Delta A = \frac{b(2x+1)}{x}$$

where x is the number of molecular chains in ΔM and $b = \frac{\sqrt{3}}{2} \times 246$ nm (Fig. 15.6-5).

One can measure ΔA in fig. 15.6-7, coming out at $\Delta A = 447.5$ nm. Obviously in on Moiré period in figure 15.6-8/15.6-9 there are $x = 5$ molecules, resulting in $\Delta A = 468.7$ nm. Moiré pattern can be investigated best at an intermediate tip voltage

1 V and 0.6 nA tunneling current.

By determining the angle between the “gaps” and the molecules via the measure angle tool in figure 15.6-7 one finds the length of a single molecule is $l \approx 3.6$ nm. The gap itself has a width of approximately 364 pm.

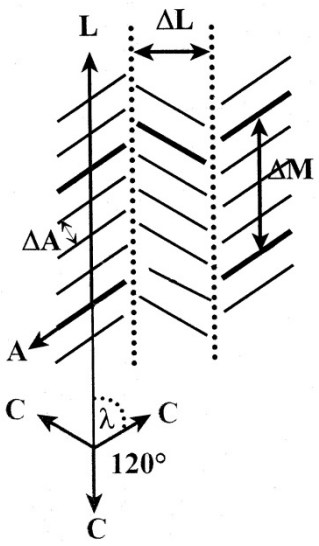


Fig. 15.6-6 Moiré patterns

A: main axis
L: Lamellar axis
C: HOPG axis
ΔA: Intra molecular distance
ΔL: Lamellar width
ΔM: Moiré period
λ: Angle between L and C

Conclusion

Arachidic acid molecules form a monolayer of parallel aligned molecules when brought onto the HOPG surface. The dominant effects in this self-organization are the dipole-dipole interactions between the carboxyl end groups (figure 15.6-10) and the side-by-side van der Waals interactions between the aliphatic chains.

Furthermore the molecules theoretically align to the substrate as shown in figure 15.6-11. This is due to the carbon atoms in the chain have practically the same array as the HOPG carbon

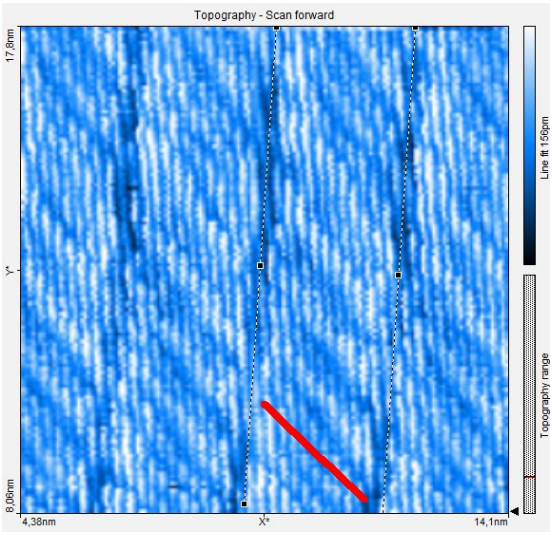


Fig. 15.6-7 Arachidic acid molecules adsorbated on the surface of HOPG. The line distance is 2.761nm. The position of a single molecule is indicated in red

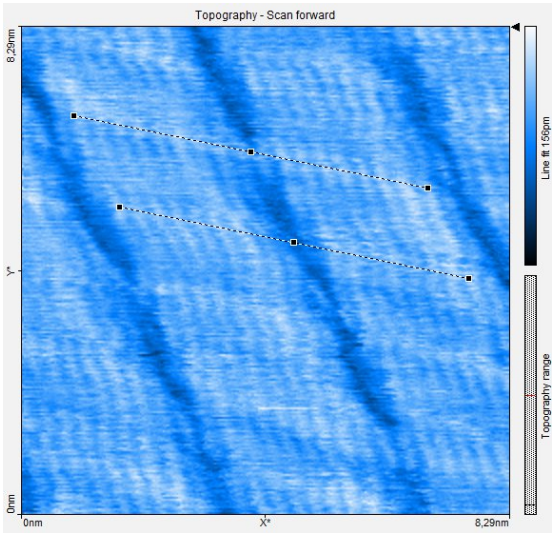


Fig. 15.6-8 Moiré pattern. The line distance is 1,361nm

atoms and bond to the π-orbitals of the HOPG substrate, but the carboxyl end group of arachidic acid needs more space resulting in a not simple commensurable assembly.

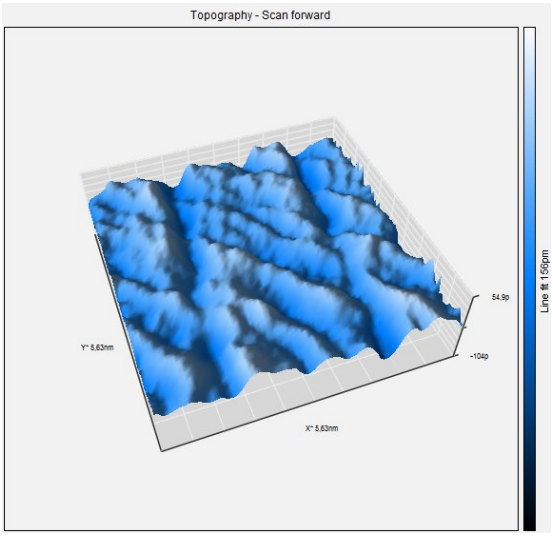


Fig. 15.6-9 3D view of the arachidic acid molecules on HOPG (Fig. 15.6-8)

The measured tunneling current for tip voltages >1V is dominated by the molecules aligned to the surface modulated with a Moiré pattern, making it difficult to achieve atomic resolution on the molecules as one sees in figure 15.6-7. However one can measure certain characteristics of the molecules: The measured molecule length of $l \approx 3.6$ nm approximately matches the theoretical model of image 15.6-11 regarding the length of the aliphatic chain of 2.59 nm, the carboxyl end group and the space between single molecules due to bonding. So the measured length is approximately the actual molecular length (length from first to last atom) with contributions from the bonding.

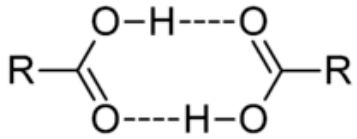


Fig. 15.6-10 A dimer of two carboxylic acids

The lamellar distance of 468 pm is larger than the expected from simple side by side van der Waals bonding (van der Waals radius of H is 118 pm, the covalent radius of C is ~70 pm). This is due to the alignment on the substrate and the carboxyl end groups, which reject neighbored molecules because of partial charges.

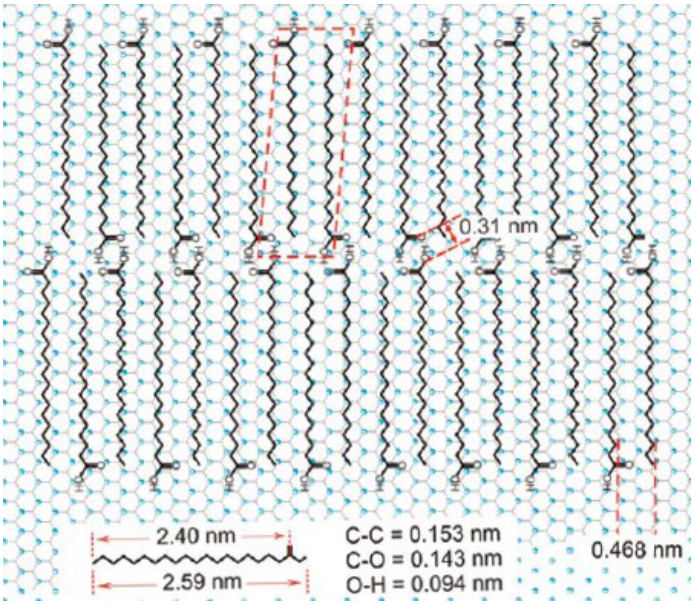


Fig. 15.6-11 Schematic alignment of arachidic acid on HOPG. The blue spots indicate the carbon p orbitals.[2]

On the basis of the two dominant interactions (dipole-dipole and van der Waals) one can predict the behavior of molecules on HOPG similar to arachidic acid, e.g. other fatty acids or aliphatic chains with other end groups with similar self-interactions.

Yet more complicated is the transfer of these concepts to other substrates, due to different lattice parameters and surface states.

For further readings see Thomas et al.: Monolayer Structure of Arachidic Acid on Graphite; *J. Phys. Chem. C, Vol. 114, No. 44, 2010*

[2] From Thomas et al.: Monolayer Structure of Arachidic Acid on Graphite; *J. Phys. Chem. C, Vol. 114, No. 44, 2010*

15.7 Hexadecanol and TMA – different phases of self-assembled molecular networks

References:

- ***P2534500 "Investigate in different phases of multicomponent self-assembled molecular networks of TMA and hexadeconal molecules on HOPG by STM"***

Task: Investigate the topography of adsorbed hexadecanol and trimesic acid molecules on the HOPG surface.

Applying the molecules

To apply the molecules to the surface first stop the measurement of HOPG when seeing single atoms and withdraw the sample 2-3 times via the "withdraw" button. Remove the Cover from the STM carefully. Now use the syringe to aspirate a small drop of one of the solutions. Withdraw the syringe from the solution and push its piston a bit, such that a drop of the solution adheres to the tip of the needle. Now touch the HOPG surface with the drop carefully (Fig. 15.7-1). The drop will come undone the needle and disperse across the sample resulting in a liquid meniscus between the scanning tip and the sample as shown in figure 15.7-2. If there won't be a meniscus repeat the process.

Now carefully place the cover on the STM again. The status light should be orange all the time. After re-approaching the sample you should be able to see the atomic resolution of HOPG as before. If not, try scanning a different area of the surface (lower resolution -> move -> increase resolution on flat terrace).

If you are not able to receive atomic resolution, you probably have crashed the tip when applying the solution. If so, you will have to prepare a new tip and start over again. However you may also try to continue the measurements first.

For tip voltages of few 10mV the electrons will mainly tunnel from the orbitals of the HOPG. When changing the voltage to values of about 1V and adjusting the tunnel current respectively you should be able to see the molecules adsorbed on the surface. You maybe will need to be patient in this step because the molecules need to assemble to the surface first. It often is useful to use cleaning pulses to help the molecules aligning on the surface. Typical scanning parameters to see the molecules are tip voltage 0.9 V, set point 0.3 nA, P-gain 1000, I-gain 2000.

Figure 15.7-3 and 15.7-4 show the structure of the investigated molecules which we are expecting to find in the scans. In figure 15.7-5 and 15.7-6 one sees the corresponding alignment of the molecules in the solutions of only TMA/hexadecanol: The hexadecanol molecules are aligned aside each other. This is due to side-by-side van der Waals binding of the aliphatic chains. The alignment of the TMA molecules is a results from dipole forces resulting from the partial charges of the carboxyl functional groups.

Fig. 15.7-1 A drop adheres to the tip of the syringe

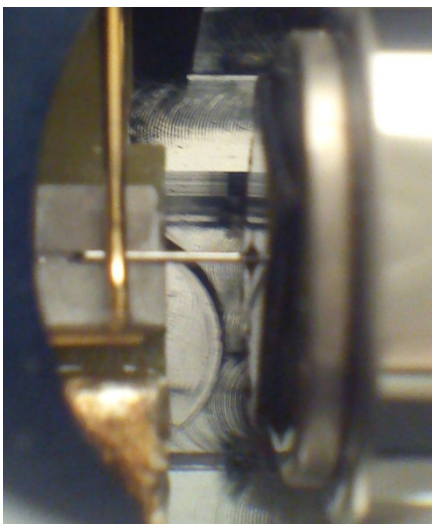


Fig. 15.7-2 The liquid meniscus surrounding the tip

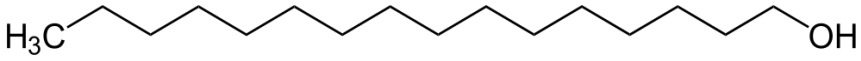


Fig. 15.7-3 Hexadecanol

In figure 15.7-7 the mixture of TMA/hexadecanol molecules is shown. One sees domains with differently aligned molecules. A zoom in such a domain border is shown in figure 15.7-8 (in phenyloctane) and 15.7-9 (in toluene). In the right of image 15.7-8 and in image 15.7-9 there are hexadecanol molecules side by side and TMA molecules between lines of hexadecanol as visible in image 15.7-10. Here the TMA molecules are aligned in between the hexadecanol molecules as seen in figure 15.7-11. Furthermore figure 15.7-12 shows hexadecanol molecules aligned side by side as in the left of figure 15.7-8. One sees that across one “stip” of the structure there are two hexadecanol molecules aligned in an angle of 180° to each other. Additionally the 3D representation of figure 15.7-10 is shown in Fig. 20.

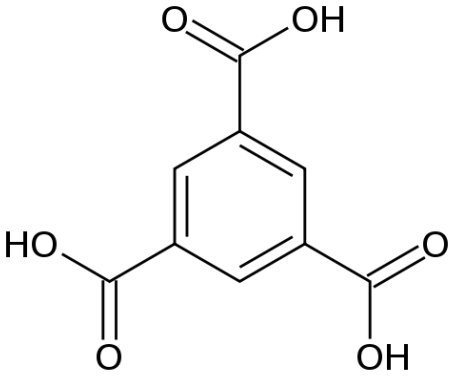


Fig. 15.7-4 Trimesic acid

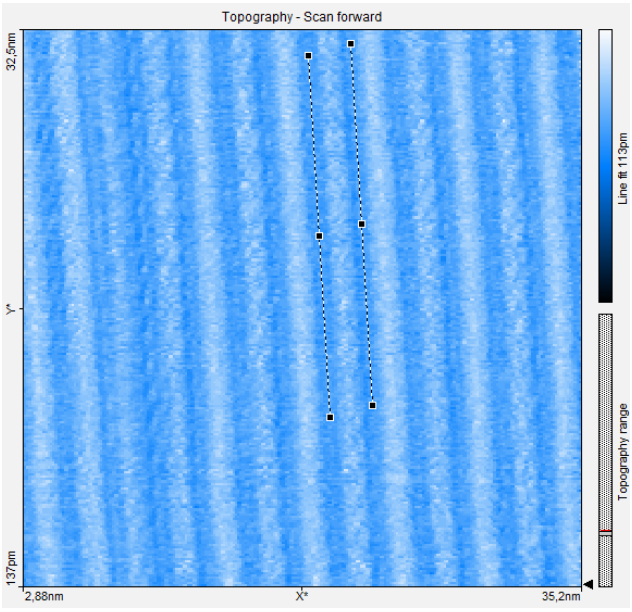


Fig. 15.7-5 Hexadecanol at the pheyloctane interface. The distance between the lines is 2,5nm

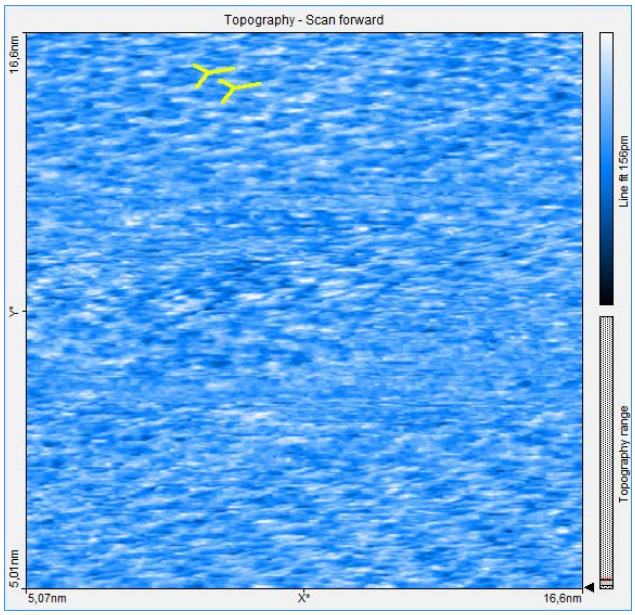


Fig. 15.7-6 TMA molecules at the toluene interface

A phenomenon you might come across are Moiré patterns. These are beats of the lattice of the adsorbate and the substrate. The results is a modulated tunneling contrast with the moiré period ΔM. For further readings on this topic see the experiment P2534000 "Investigate in self-assembled molecular networks of arachin acid on HOPG by STM" in this same series of experiments. In figure 15.7-10 one can see a slight modulation in the contrast of the hexadecanol molecules. The period is approximately 2 nm which is in correspondence with literature.

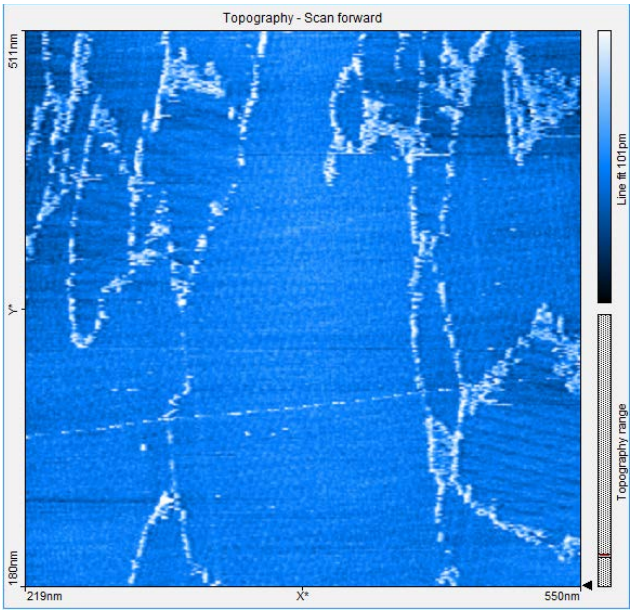


Fig. 15.7-7 Domains with different molecule alignments at the phenyloctane interface

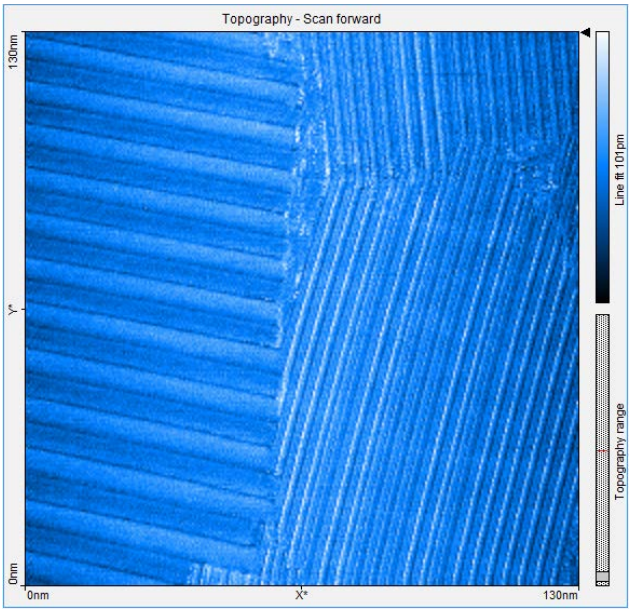


Fig. 15.7-8 TMA and hexadecanol at the phenyloctane interface. Left: pure hexadecanol, right: alternating hexadecanol and TMA

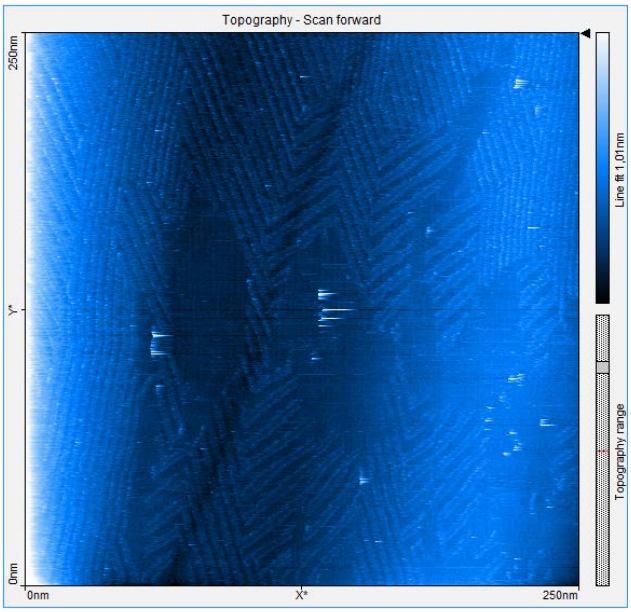


Fig. 15.7-9 TMA and hexadecanol alignments at the toluene interface

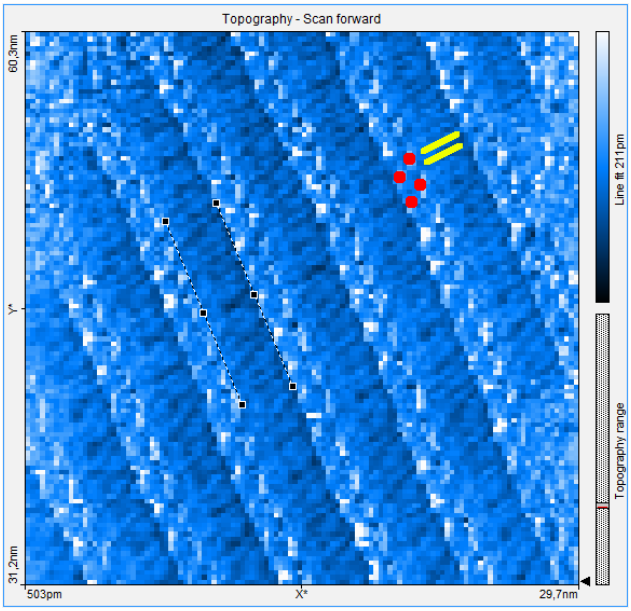


Fig. 15.7-10 Alternating hexadecanol (indicated yellow) and TMA (indicated red) at the toluene interface. The distance between the dashed lines is 2.9nm

Conclusion

Hexadecanol molecules form a monolayer of parallel aligned molecules in the liquid meniscus on the HOPG surface. The dominant effects in this self-organization are the dipole-dipole interactions between the hydroxyl end groups and the side-by-side van der Waals interactions between the aliphatic chains. The alignment on the HOPG substrate is due to emerging p-orbitals: The carbon atoms in the chain have practically the same array as the HOPG carbon atoms and bond to these orbitals of the HOPG surface. However the hydroxyl end group of hexadecanol needs space resulting in an incommensurate alignment and Moiré patterns.

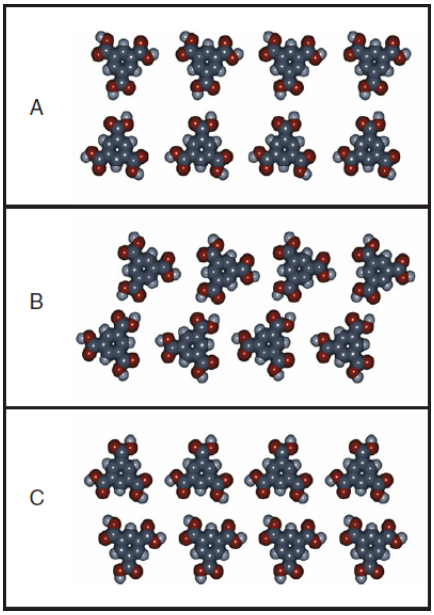


Fig. 15.7-11 Dimers of two carboxylic acids. Grey: carbon, red: oxygen, white: hydrogen[3]

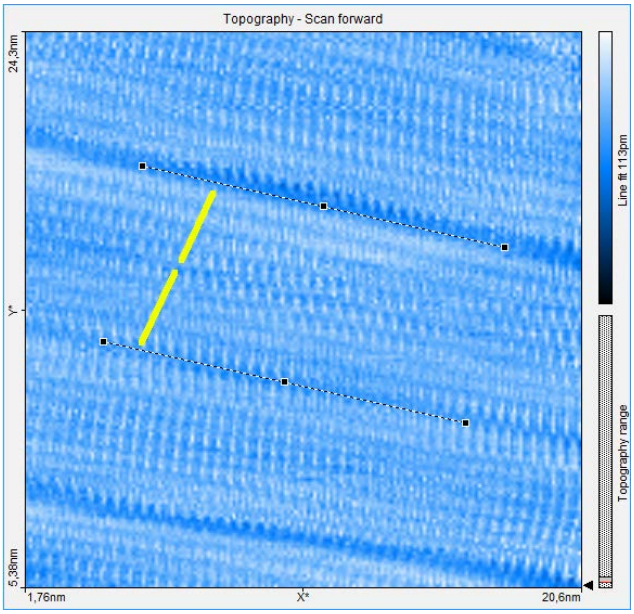


Fig. 15.7-11 Zoom into the left of image 15: Hexadecanol at the phenyloctane interface. The distance between the lines is 6.1nm. Two molecules are indicated in yellow

The TMA also bonds to the p-orbitals of the HOPG substrate. The benzene ring in the molecular structure of TMA basically is identical with the carbon hexagons in HOPG. However the carboxyl groups surrounding the benzene ring need space resulting in a different periodicity than the carbon hexagons in the HOPG. The carboxyl groups also result in partial charges in the molecules which results in the present alignment.

If hexadecanol and TMA are brought together on the HOPG surface there domains with different alignments. In some of them there are exclusively hexadecanol or TMA molecules in others the both align alternately: A strip of hexadecanol is followed by a strip of TMA molecules and vice versa. In such areas the strip of TMA mostly consists of 2 rows of molecules, whereas that of hexadecanol consists of one row of molecules aligned side by side and might be modulated by Moiré patterns.

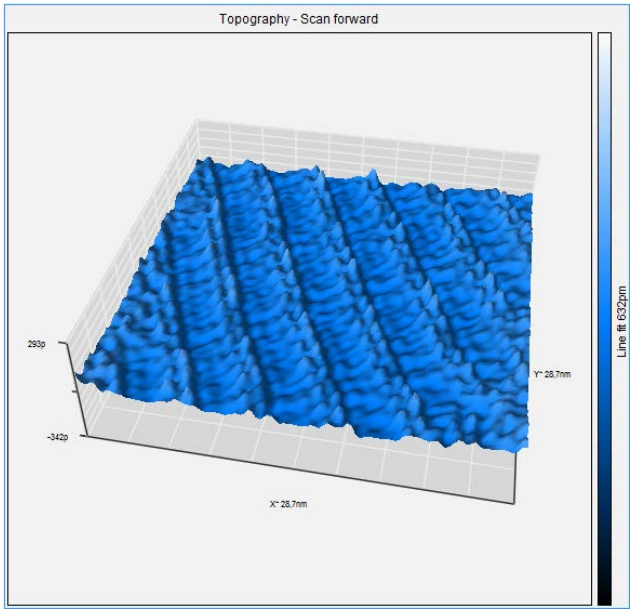


Fig. 15.7-13 3D view of figure 15.7-10

The resolution of the solutions differs when using phenyloctane or toluene: In toluene the single molecules can be identified clearly, but the structures drift relatively fast resulting in a bad scan resolution due to the required scan speed. The scans in phenyloctane can be done using a lower scan speed enabling high scan resolutions, however it is difficult to see single molecules in the scans.

[3] From J. M. MacLeod et al.: Stabilization of exotic minority phases in a multicomponent self-assembled molecular network. Nanotechnology 18 (2007) 424031 (9pp)

The measured size of a hexadecanol molecule is approximately 2.7nm which is in correspondence with literature.

Using the spare test-tubes and syringes you can also try to investigate TMA in the phenyloctane interface and hexadecanol in the toluene interface. However, it's generally more difficult to receive good results using this combinations of molecules and solvents.

For further readings see J. M. MacLeod et al.: **Stabilization of exotic minority phases in a multicomponent self-assembled molecular network.**; Nanotechnology **18** (2007) 424031 (9pp)

15.8 Carbon nano tubes (CNT)

References:

- ***P2536000 "Investigate in carbon nanostructures by Scanning Tunneling Microscopy and Spectroscopy"***

Task: Investigate the topography of the carbon nanostructures on HOPG in constant-current mode. Switch to spectroscopy mode. Measure and compare images recorded on different locations in Tip-voltage mode (I-U spectroscopy).

Electronic properties of carbon nanotubes

Single walled carbon nanotubes (SWCNT, Fig. 15.8-1) can be described as rolled up single sheets of Graphite. Depending on their structure nanotubes show different electronic and thermal properties. Some of the possible pattern are shown in figure 15.8-2[4]. One can find out about the electronic characteristics of an imaged nanotube by local I(U)-scanning tunnel spectroscopy.

The typical dimensions of simple SWCNTs like zigzag- and armchair-nanotubes are ≈1 nm in diameter and up to microns in length.

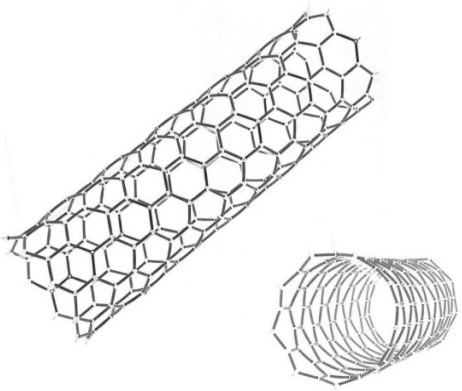


Fig. 15.8-1 Sketch of a carbon nanotube

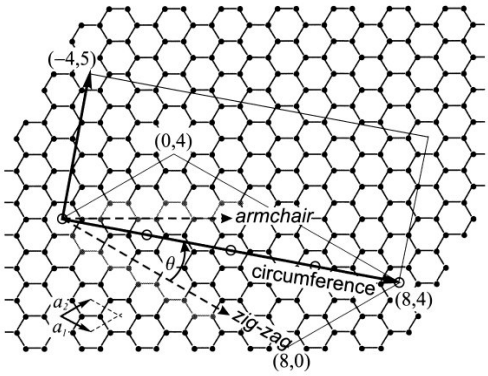


Fig. 15.8-2 Sketch of possible nanotube configurations

In I(U)-spectroscopy the distance between tip and sample is constant. Only the applied voltage is changed. From Ohm's law one would expect a linear dependency of voltage and current from the spectroscopic measurement. However, the results differ because of the band structure of the investigated materials.

[4] *Lauffer, Peter (2009): Rastertunnelmikroskopie und Rastertunnelspektroskopie an Kohlenstoffnanoröhren und epitaktischem Graphen. Dissertation, Uni Erlangen*

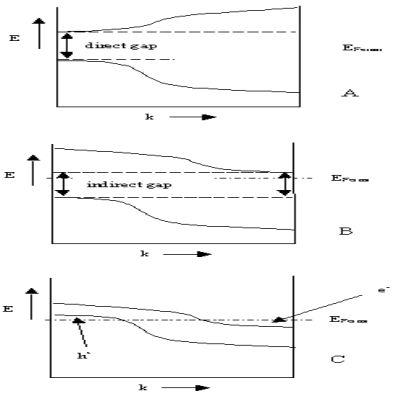


Fig. 15.8-3 Sketch of the band structure of an insulator/semiconductor

The electrons of a single atom can occupy discrete energy levels forming atomic orbitals. Several atoms together into a molecule will form molecular orbitals. The number of molecular orbitals is proportional to the number of atoms is the molecule.
When large numbers of atoms (>10^{20}) are brought together to form a solid there are large numbers of energy levels occupied by electrons and the difference between them becomes very small, forming continuous energy bands. However depending on the atoms forming the solid there are energy intervals which cannot be occupied by electrons no matter how many atoms are aggregated. Such intervals are called band gaps (Fig. 15.8-3). Band gaps only occur at semiconductors (small band gap < 3 eV) and insulator (larger band gap). Solids which have no band gap, meaning Conduction band and Valance band overlap, are called metals.

Regarded in detail, the electronic band structure of a solid is very complex. It depends on the properties of the underlying crystal lattice and is described by graphs as shown in figure 15.8-4.
Here the energy is plotted as a function of the wave vector k, which describes the motion of the electrons in the lattice. The wave vector k takes on values within the Brillouin Zone, which is a model to describe unit cells, corresponding to the crystal lattice. Particular directions/points in the Brillouin Zone are assigned conventional names like Γ, Δ, Λ, Σ, *etc.* The areas, electrons are allowed to be are highlighted grey.

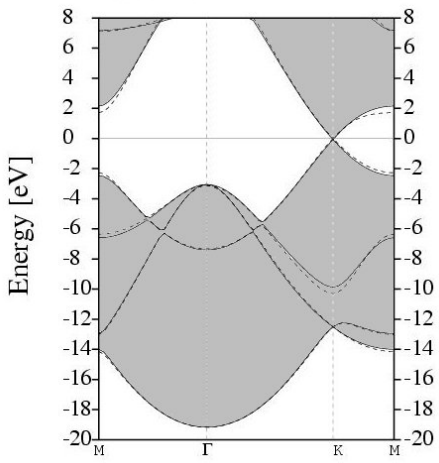


Fig. 15.8-4 Reduced band structure of HOPG

The allowed local electron distribution among the energies is determined by the local density of states (LDOS). The LDOS is a space-resolved description of the number of states at each energy level which are available to be occupied in an electronic system. The LDOS is proportional to the slope of the characteristic curve received from I(U)-Spectroscopy, which is called specific resistivity, when regarding the tip's density of states to be with no structure. The measured curves are an overlay of the bands in the corresponding band schematic. The expected results are shown in figure 15.8-5. It follows that the interesting electronic characteristics (metallic, semiconducting, etc.) are localized within a small tunneling voltage interval around U=0 V. The properties for higher voltages are more detailed material characteristics and of lesser interest for our measurements.

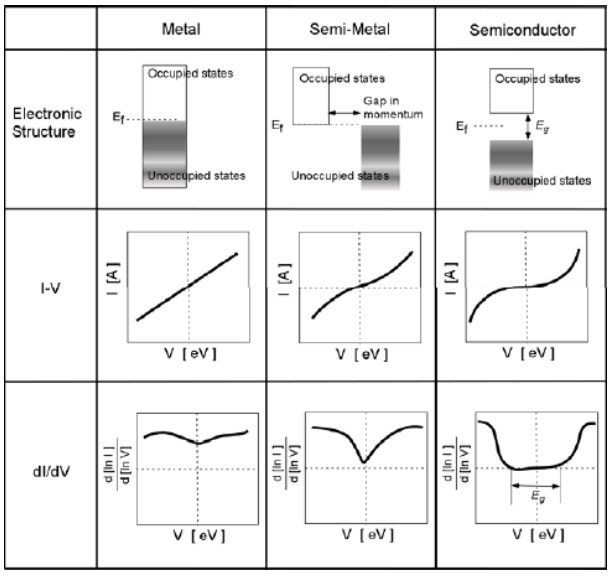


Fig. 15.8-5 Properties of different kinds of conductors

The HOPG substrate we use for our measurements is a zero band gap semiconductor and therefore falls into the category of semimetals. Carbon nanotubes can be either metallic or semiconducting.
For the armchair (metallic) and zigzag (semi conducting) configuration of carbon nanotubes the DOS is shown in figure 15.8-6[5]. The sharp peaks in the curves are van Hove singularities (VHS) which come from the quasi-one-dimensional properties of the nanotubes. In measurement we expect the peaks not to be as sharp, but they can be visible when using the right parameters.
Always keep in mind to do measurements preferably at the same room temperature. Temperature dependencies create uncertainty and errors in the expected values of your measurements.

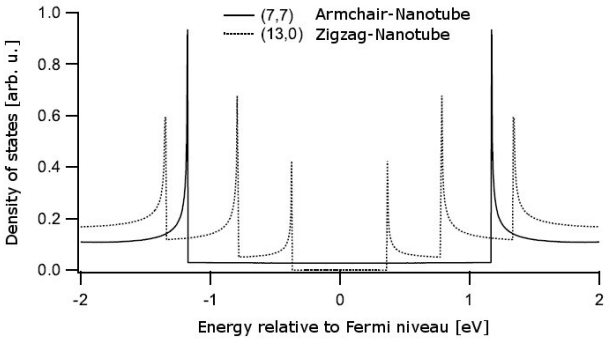


Fig. 15.8-6 Sketch of the DOS of an armchair-/zigzag-nanotube

Investigation of carbon nanotubes

After you approached the sample prepared with nanotubes click the Full button in the imaging window to activate the full measurement range. Now Zoom in to atomic resolution on a clean terrace to check the tip again.
Change back to full measurement range to investigate carbon nanotubes. If you are lucky you might find some of them in your first scan, if not withdraw the sample and re-approach another surface site. Figures 15.8-7 and 15.8-8 show bulks of carbon nanotubes on the HOPG surface. From the diameter of the structures in these images one sees that several parallel nanotubes stick together.
However, we want to measure single nanotubes, which can be found at surface line defects at times but preferably on clean terraces (Fig. 15.8-9 and 15.8-10). If you only find bulks of nanotubes you can disperse the solution in an optionally supported ultrasonic cleaner prior applying it to the HOPG sample. The aim is to image single nanotubes an apply I(U)-spectroscopy to it and in the same measurement to a clean terrace near the nanotube which is important to verify the results from the nanotube and to make sure one does not only measure a nanotube adhered to the tip.

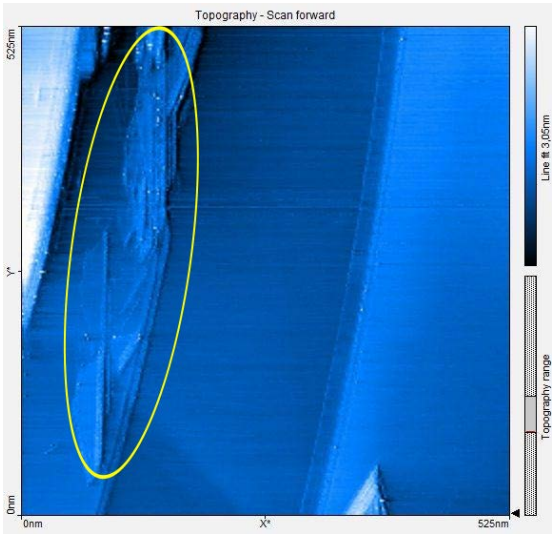


Fig. 15.8-7 Steps and nanotubes (circled) on the HOPG surface

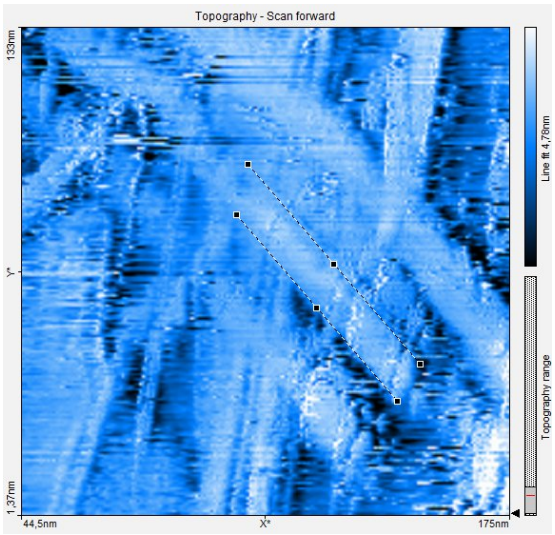


Fig. 15.8-8 A bulk of nanotubes. The distance of the lines is 11 nm

[5] *Ibid.*

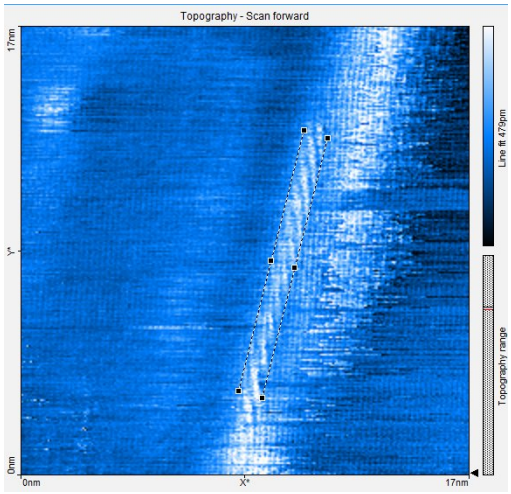


Fig. 15.8-9 A single Nanotube on the HOPG surface. The distance between the lines is 982 pm

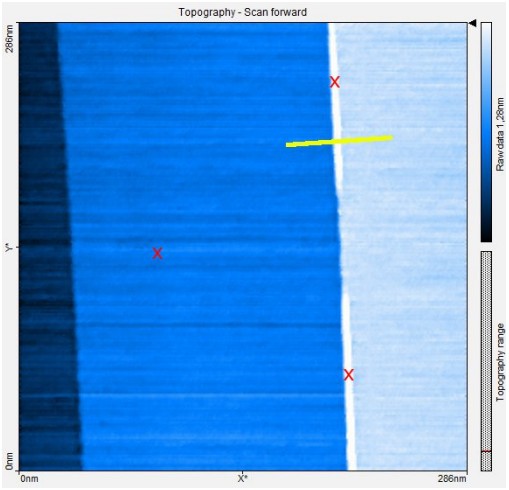


Fig. 15.8-10 Nanotubes at line defects on the HOPG surface. Three I(U) measurement positions and the cross-section in Fig. 15.8-11 are indicated.

When you found a good surface location switch to the spectroscopy mode. Choose “Tip voltage” as modulated output to record an I(U)-curve of a nanotube. Pin down values of your measurement parameters accurately. If you experience problems in measuring the desired behavior, try to adjust your parameter values.

For example you can start experimenting with the following set of parameters:

- Set point 1.0 nA
- P-Gain 1000
- I-Gain 2000
- Tip voltage 1 V
- Start value -1 V
- End Value 1 V
- Modulation time 0.2 s
- Data points 128
- Averages 8

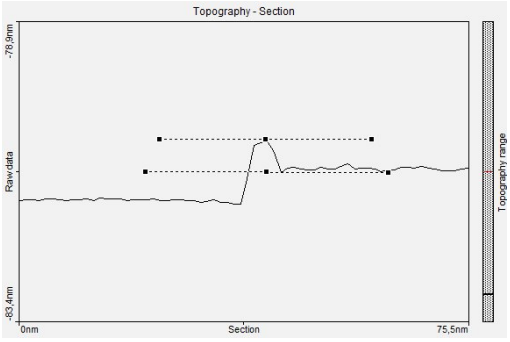


Fig. 15.8-11 The cross section from figure 15.8-10. The distance between the lines in 493pm

By using this set of parameters, the device will divide the interval from -1 V to 1 V into 128 points and measure the tunnel current at each of them. The time used for one series of measurements is 0.2 s. This process will be repeated 8 times. The resulting curve is the average of the 8 measurements.

Notice:

- The device may cut off the graphs due to high tunnel currents. In this case adjust your Parameters to decrease the current (e.g. lower set point)
- High tunnel currents may also influence the structure of the tip and/or the surface. Therefore try choosing a lower set point or lower start and end values if the curve looks not as expected

- As long as you are in the spectroscopy mode the piezo controllers are inactive. Because of thermal drift you might receive different results when measuring the same point multiple times, especially when using high resolutions. Also modifications of the local tip geometry can occur during the measuring procedure indicated by noisy and “jumpy” spectra. Therefore check the topography after every spectroscopic measurements to evaluate the results.

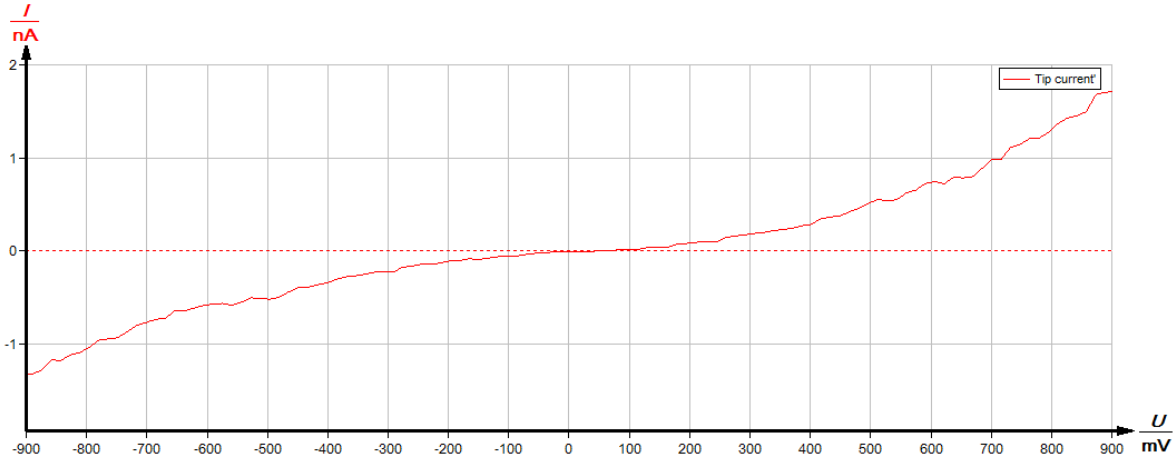


Fig. 15.8-12 I(U)-spectroscopy curve of HOPG

Prepare a measurement by clicking on “Point” and putting the cursor on the surface of your scanned image where you want to take the spectroscopy data. Press the “Start” button. After a few seconds the end of the measurement is indicated when the “Stop” button changes back to “Start” and when your data curve doesn’t change anymore. The “Probe Status” should be green during the whole measurement. If the “Probe Status” changes to orange switch to the “Imaging Window” approach the sample again and take a new surface scan, afterwards switch back to the “Spectroscopy” window.

After the measurement is finished you can see the I(U)-curve in the line graph. If your curve does not look as desired repeat the measurement by pressing “start” again. If you still don’t get the desired curves switch back to the imaging mode and repeat the process. To analyze the current-voltage curves right-click the line graph and click “Copy data to clipboard”. Now open the analyzing software PHYWE *measure* and paste the data by pressing ctrl+v or using “Measurement” → “Import Data”. You should receive a graph as shown in figure 15.8-12. Apply the spectroscopy mode to other nanotubes in the same way and compare the results. The resulting curves for a semiconducting and a metallic nanotube are shown in figure 15.8-13. The curve of HOPG is also plotted in this graph.

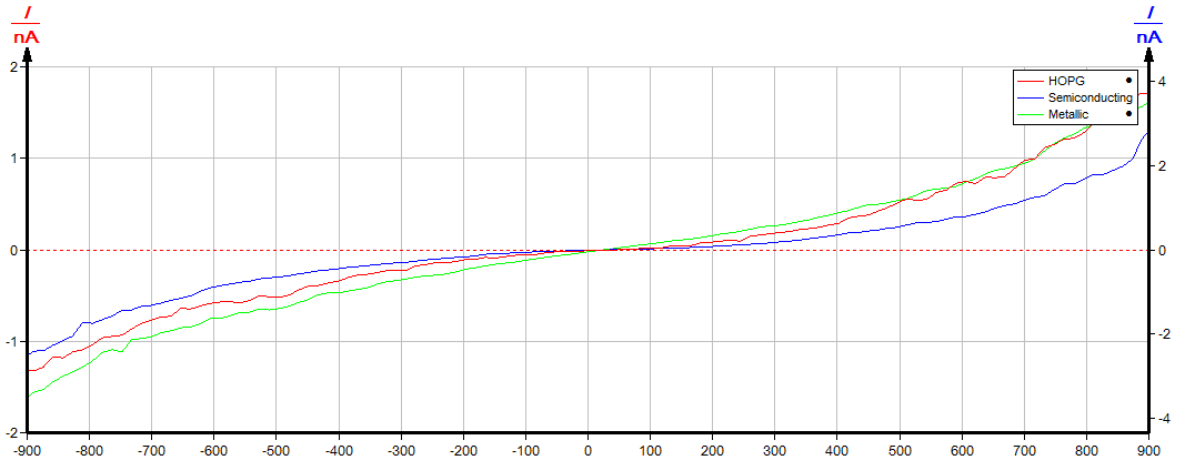


Fig. 15.8-13 I(U)-spectroscopy curve of HOPG and a semiconducting (metallic) carbon nanotube as shown in figure 15.8-10. Scales for HOPG and metallic are identical

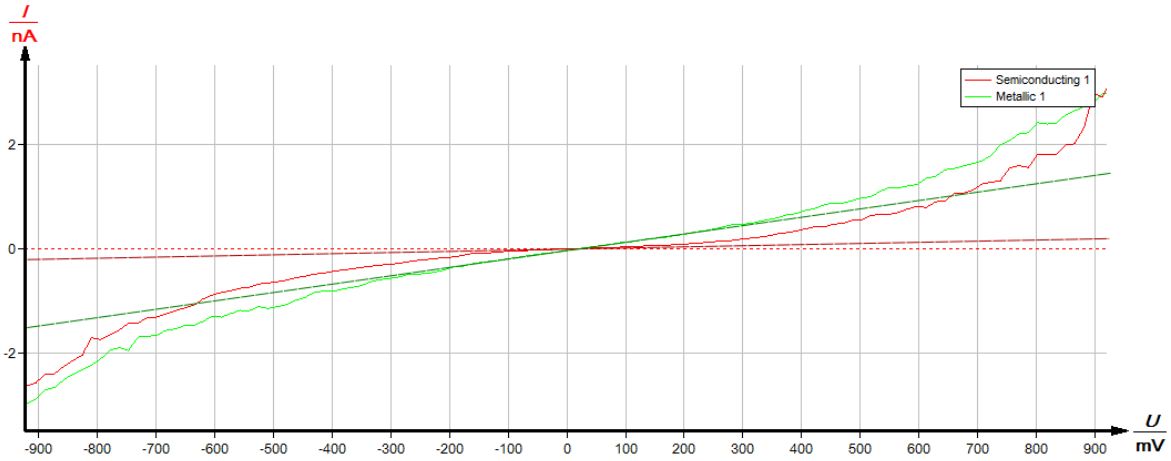


Fig. 15.8-14 I(U)-spectroscopy curve of semiconducting and metallic nanotube and the corresponding tangents in U=0V

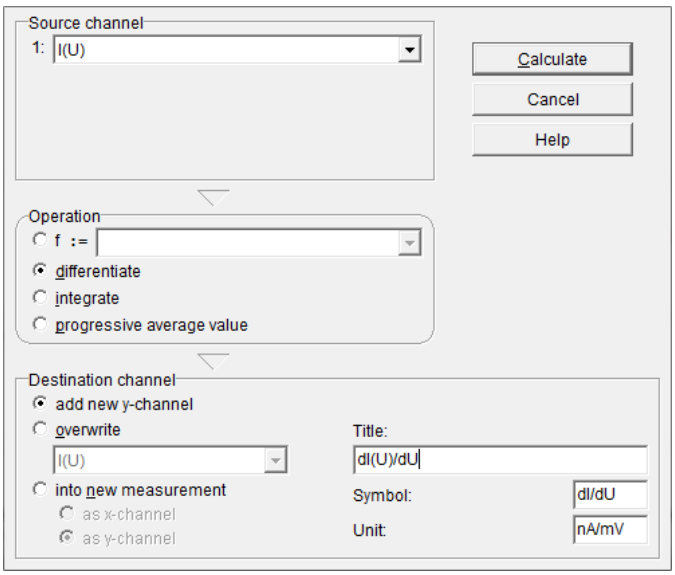


Fig. 15.8-15 Channel modification window

In order to compare the spectroscopy curves one needs to adapt the tunneling current by matching it to each other at the maximum tunneling voltage. Figure 15.8-15 shows the matched curve for an semiconducting and a metallic carbon nanotube. Furthermore the slopes of the two curves at U=0 V are indicated by dashed lines each in the corresponding color.

To analyze the derivative of the I(U) curve it is recommended to smooth the curve first. Therefore go to “Analysis” → “Smooth”. Choose the strongest smoothing and overwrite the existent curve. Repeat the smoothing until you receive an appropriate curve as shown in the graphs. Now go to “Analysis” → “Channel Modification” and choose “differentiate” (Fig. 15.8-15). Now you can see the corresponding curve proportional to the LDOS. Images 15.8-16 to 15.8-18 show the results from the measurements in figure 15.8-13.

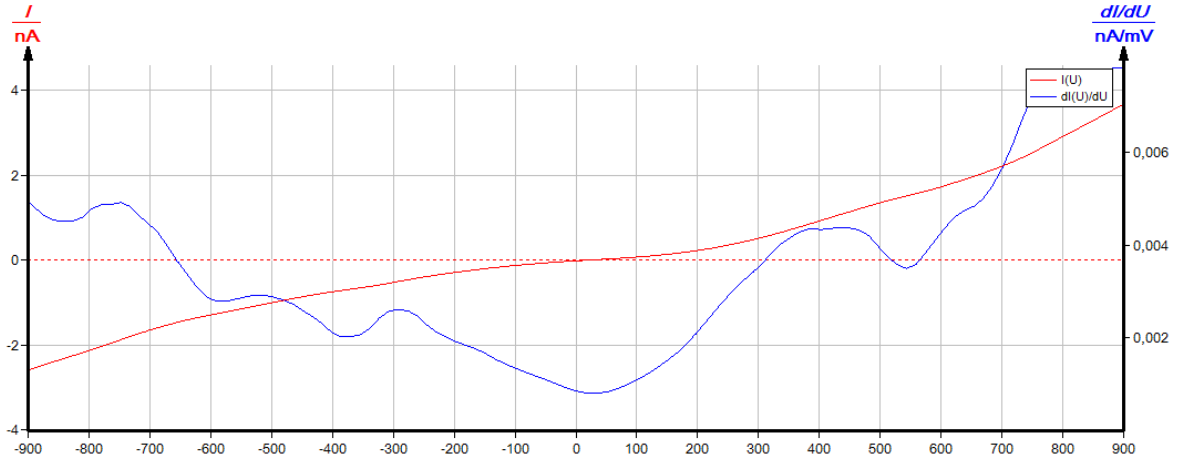


Fig. 15.8-16 Tunneling current and specific resistivity of HOPG over the tunneling voltage

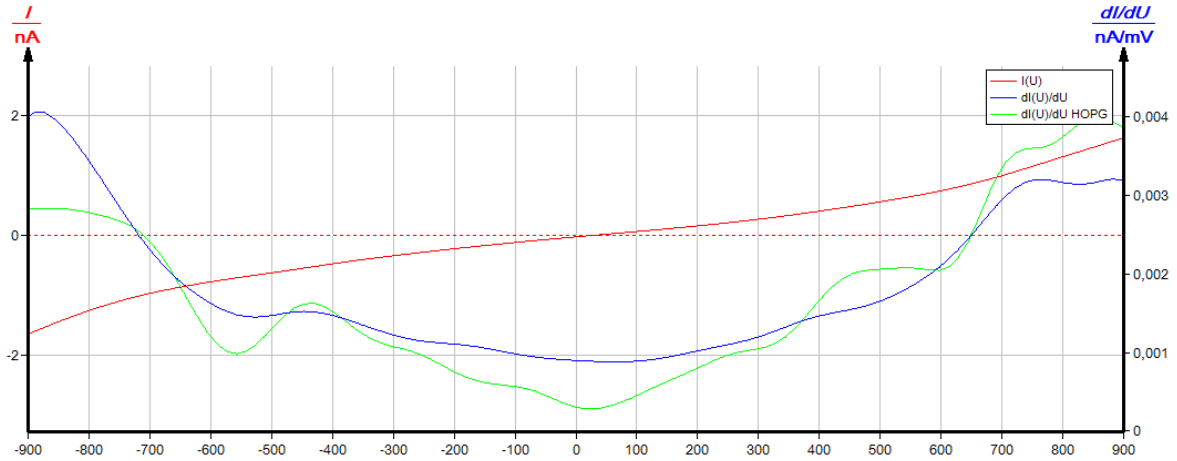


Fig. 15.8-17 Tunneling current and specific resistivity of a metallic carbon nanotube over the tunneling voltage

Note

The x-axis are zoomed in a bit (use mouse-wheel on x-axis) because the smoothing process distorts the data at the edges. Furthermore the specific resistivity of HOPG is plotted in the each of the nanotube graphs for comparison by “Measurement” → “Adopt channel”.

Furthermore in figure 15.8-18 the van-Hove singularities are indicated.

In literature one finds a different representation of the specific resistivity called normalized tunneling spectrum. This is the specific resistivity dI/dU divided by the conductivity I/U for each value of U. This is to compesate the decreasing tunneling barrier as the tunnelvoltage increases. The problem with this method is that the tunneling current needs to vanish at low tunneling voltages which is not the case in our measurements, creating a pole of I/U at U=0V.

Task 4: Interpret the results regarding to the band structure.

The first peculiarity in the spectroscopies is that the tunneling current is not vanishing for zero tunneling voltage. Obviously this must be a measurement error and needs to be taken account of when interpreting the results.

The expected tunneling curves from figure 15.8-5 are confirmed very good by our measurements as seen in Image 20. The difference in the slopes for the semiconducting and metallic nanotube around U=0V becomes even more obvious when adapted (Fig. 15.8-4) and corresponds to the expectations. Especially one sees the conductivity for the semiconducting nanotube increases by more than a magnitude in the applied voltage interval whereas for the metallic nanotube it increases only by a small factor.

For the spectroscopy on HOPG (Fig. 15.8-16) one sees the LDOS looks as expected for a semimetal. Because of HOPG having no band gap low energies i.e. temperatures are viable to excite electrons into the conduction band resulting in a finite DOS at U=0V. Hence, this result is in approval with figure 15.8-5.

For the metallic carbon nanotube (Fig. 15.8-17) one sees a practically constant DOS for low tunneling voltages. The slight fluctuations can be explained by the substrate or ambient conditions influencing the

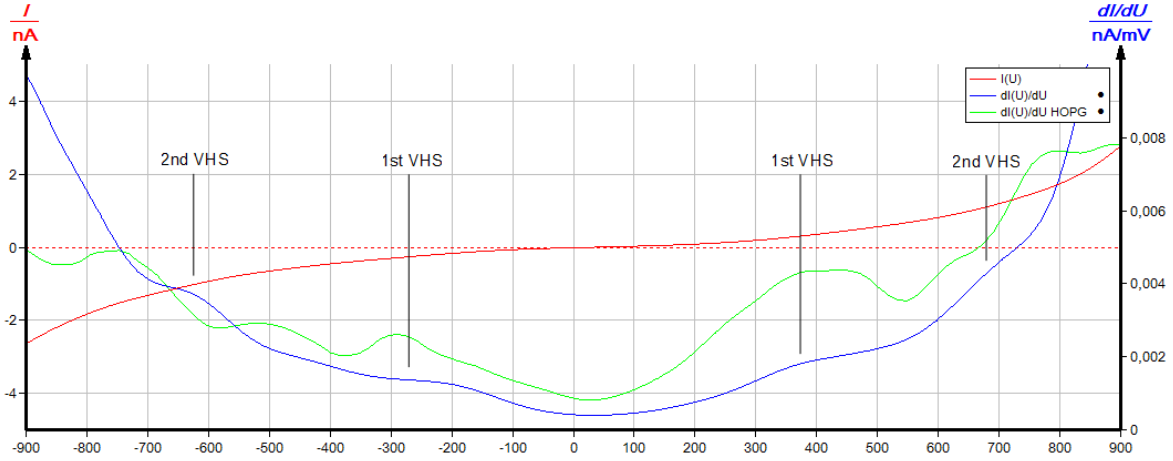


Fig. 15.8-18 Tunneling current and specific resistivity of a semi conducting carbon nanotube over the tunneling voltage with indicated van-Hove singularities

measurement. The constant DOS is the result of the metallic CNTs being a conductor. This means it has no band gap due to the valence and conduction band overlap, meaning electrons can move freely along the metal. The I(U)-spectroscopy-curve therefore images Ohm's law U=RI. Furthermore the DOS for low tunneling voltages is above that of HOPG which corresponds to the expectations (Fig. 15.8-5).

The DOS of the semi conducting carbon nanotube (Fig. 15.8-18) looks good, too. One can see an interval of the tunneling voltage at which the DOS is nearly zero indicating the band gap. As before the high temperatures are viable to excite electrons into the conduction band explaining this result deviating from

figure 15.8-5 a bit. Furthermore the DOS is lower than that of HOPG which corresponds to the expectations. The van Hove singularities from figure 15.8-6 can be identified and are at the expected tunneling voltages for a zigzag nanotube. Especially they are at different tunneling voltages than those of the pure HOPG substrate confirming the assumption that the DOS of a nanotube is imaged.

The supported nanotubes are a mixture of different kinds of nanotubes. One can calibrate the STS measurements by measuring samples of pure nanotubes of one kind one might have to clearly identify nanotubes in the measurements of the mixture.

This experiment demonstrates the potential of the scanning tunneling microscope to investigate not only the electrical characteristics in general but also at the nanoscopic scale. Especially for artificial nanostructures STS can be used to investigate the electrical characteristics.

For further readings see e.g.

- Carbon nanotubes: synthesis, structure, properties and applications. *M. S. Dresselhaus, G. Dresselhaus, Phaedon Avouris. Springer Science+Business Media*
- Carbon Nanotubes: Basic Concepts and Physical Properties. *Stéphanie Reich, Christian Thomsen, Janina Maultzsch. Wiley-VCH*

measure nano PHYWE

nano compact STM PHYWE